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# Vacuum Technology

# and

# Vacuum Design Handbook

# for

# **Accelerator Technicians**

Prepared by David Garton November 2011 Revision 0 Vacuum Technology and Vacuum Design Handbook for Accelerator Technicians

This handbook is a compilation of information gathered from over 50 years of direct hands-on experience to applicable information widely available from the vacuum technology industry. It seeks to address common and specific vacuum technology problems whilst clarifying the design standards and philosophies adopted for use in the ANSTO accelerator facilities. The author wishes to thank the reviewers and the many technicians from ANSTO that have contributed directly and indirectly to this booklet.

Revision 0 Author: David Garton, 18-11-11 Reviewer: Michael Mann, 9-12-11 Reviewer: David Button, 19-12-11

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# Chapter 1

# Vacuum design standards and good practises for the Accelerator Area

- \* This chapter must be read in conjunction with the detailed vacuum information in this document.
- \* Based on best practises developed at ANSTO and other local Australian accelerator laboratories in conjunction with Australian Standards and industry technical guides, referenced throughout this document.
- \* Where a standard is unavailable for a specific design, best practices are used from industry's best practises.
- \* *Refers to all vacuum chambers, beam lines and other enclosures used on the accelerators or stand alone.*

#### 1. Basic design

- All vacuum vessel designs must be consistent with the design standards in Australian Standard AS 1210-2010 and maintain a Hazard Level of E as per the design conditions tabled in "Hazard Levels of Pressure Equipment" in Section 2, Hazard Levels of Pressure Equipment in Australian Standard AS 4343-2005.
  - The vacuum equipment designed by/for the accelerator area maintains a Hazard level of E. All pressure equipment with a pV  $\leq$  30, pressure (p in MPa) x volume (litres)  $\leq$  30 is classed as Hazard level of E. For vacuum equipment in Hazard Level E the derating value of 0.1 is applied, ie 0.1pV  $\leq$ 30. Note the amount of stored energy at this level is very small. For example a Ø 1000 mm chamber, 1000 mm high will have a pV for vacuum equal to 7.85.
  - AS 4343-2005, Section 2.1.2 Typical hazard levels, part (d), states for Hazard Level E, "This equipment is usually exempt from special regulatory control but is <u>covered</u> by general plant safety regulations". See Appendix 7.
- All designs of chambers excluding general beam lines and fittings should be checked for structural integrity using finite elements analysis. Sound designs with acceptable structural integrity will be released for manufacture. A copy of the design acceptance tests must be filed with the project file. The standard finite element analysis can be performed utilising Solidworks. (Modelling for irregular shaped chambers, calculations Appendix 1 where appropriate for regular shapes)
- Stainless steel (304 or 316) will be used to construct chambers, beam lines and general fittings unless it is a *special* requirement to use other metals. Do not choose substances that have high outgassing rates or can contaminate vacuum systems.
- Measurement chambers and other large volume chambers should be cylindrical unless the chamber is an enclosure between pole faces (eg magnet box) or is an odd shape to accommodate steering plates or devices. When non-cylindrical chambers are required deflection of chamber walls should be modelled and taken into consideration.
- Other chambers including magnet boxes, ESA and deflector coffins to have suitable rib strengthened rectangular or curved sides where it is not practicable to increase the wall thicknesses.
- Minimum wall thickness calculation methods can be found in Appendix 1. As a general rule care must be taken when using formulas for vacuum calculations as historically many units other than SI units have been used to form some equations. For example length in cm, and pressure in torr is common in old notes.

- Top and bottom flanges to have integrated structural support rim which becomes the flange and seal for chamber base and top (user) flanges.
- Ports must be a suitable length to ensure unobstructed insertion for screws into flanges. Suitable weld preparation must allow for adequate weld penetration. See Welds below.
- All internal edges must have a smooth radius. No sharp edges to be left after welding or surface finishing.
- All machined finishes other than vacuum seals to be equal to or better than 1.6 microns
- No sections within the vacuum space will have metal to metal contact where gas can be trapped unless it is at the edge of a seal joint. When this is necessary, pump out grooves must be incorporated in the design.
- Vacuum windows shall be made from toughened glass or quartz. No plastic to be used for windows unless the window is a flange on a chamber that is designed to withstand more than 100 kPa. The large plastic flange/windows should be made from >18 mm thick up to Ø200 and >25 mm thick for up to Ø400 in clear Perspex or polycarbonate.
- For ANTARES beam lines the preferred flanges are Dependex, nominal diameter 100 mm. See section 8 for detail on Dependex flanges.
- For STAR there is no preferred flange type. It must be selected depending on the desired base vacuum and interface to other adjoining flanges.
- As a guide, the MDC catalogue from Vacuum Products Corporation 2003 onwards, Building Blocks for Vacuum Science and Technology, provides vacuum tubing dimensions for most common sizes. The dimensions must be assumed to be minimum dimensions. Other tubing sizes should be calculated using the formulas in Appendix 1.
- Vacuum pumping ports leading to the vacuum pumps must be designed to optimise conductance.
- Pressure relief valves to be incorporated where a vacuum system can be vented to >10 kPa above atmospheric pressure.
- ASME state that the design of an external pressure vessel must consider Material type, diameter of chamber, unstiffened length, temperature and wall thickness

### 2. Welds

- TIG is preferred for vacuum sealing welds as minimal scale and flux is produced. Electron beam welding is suitable for thin walled materials. TIG produces high quality welds with or without filler rods to produce flat, smooth well penetrated welds. TIG comes into its own where the weld preparation allows for melting together of the parent materials without filler rods. MIG has a higher deposition of filler material so care must be taken to ensure welds are kept smooth and flat.
- No standard exists for vacuum welding of compact scientific vacuum equipment where pressures approaching zero are necessary. Equipment designed in the accelerator area and welded in the ANSTO workshops since 1990 has had penetrations of ≥1mm but ≤2mm on a butt weld and ≥2 mm on a fillet weld. Experience has demonstrated the adequacy of these figures. No failure reported.
- For thin walled materials ie < 1 mm, such as bellows, manufacturers' will use their industry standard. ANSTO does not weld thin wall materials.
- Weld preparation must be provided where possible to ensure even heat penetration.

- Electric arc with rods is not recommended due to flux trapping. This can cause pits which have long lasting outgassing periods.
- All welds that form a seal against atmospheric or other positive pressures must be made to the internal side (vacuum side) of the joint.
- Tack (stitch) welding is recommended on the external side only with less than 50% of the diameter covered in small intervals. This minimises the chance of trapped voids and facilitates He leak testing post welding.
- All welds must be free from pitting and scale. Must be cleaned to bare metal. If chemicals are used to clean welds care must be taken to remove all traces of chemicals.
- Some chambers such as magnet boxes will need to have some joints welded externally due to access problems on thin chambers. In this case, good penetration must be achieved to ensure minimal gas trapping points.

#### 3. Surface finishes

- The inside of the chamber and ports must be 1.6 micron or better for mechanical finishes.
- Can be chemically cleaned (polished) but all traces of chemicals must be neutralised and removed prior to use in vacuum.
- All machining oil to be removed with an alcohol scrub then acetone rinse, minimum. The use of kerosene, turpentine or other oily solvents is not encouraged however where they are used the final clean must be with acetone followed by alcohol. Water based cutting fluids can be used as they are more readily removed therefore having less effect on outgassing.
- Nitrile gloves to be used when assembling cleaned components. Other un-powdered gloves may be used but only those that do not react with the solvents being used.
- Care must be given to cleaning and handling surfaces that will be exposed to vacuum. Gloves should be used to minimise the transfer of dirt and oils from the skin to vacuum surfaces.
- A suitably clean area should be used for the preparation of vacuum systems and all tolls used should also be cleaned (degreased).
- Stainless steel can be cleaned in nitric acid to remove scale that remains from rolling or wire cutting. It is may also be used to prepare stainless steel components for use in ultra-high vacuum systems.

### 4. Flanges

- Flanges must be standard flanges selected from the group referenced this document for compatibility reasons. Exceptions may be made but all 'o' ring seals must be to Australian Standard AS 2842-1986. External flange rings not in contact with the vacuum space may be aluminium alloy. Stainless flanges are not economical or necessary unless the seal requires forces to crush metal seals such as copper ConFlat, aluminium or indium wire.
- Minimum thicknesses for top and bottom flanges to be calculated using the standard in Appendix 1 Structural Calculations for Scientific Vacuum Vessel Design.
- All other flanges to have threads consistent with their design standard.
- ANTARES Primary flanges will be Dependex unless interface is necessary to accommodate other standard flanging.
- For Dependex, screws to be imperial sizes consistent with UNC series.

• STAR utilises many of the major vacuum flange/seal designs. The most appropriate design must be chosen that meets the specific application.

#### 5. Seals

- 'O' ring sizes and sealing groove dimensions must comply with the Australian Standard AS 2842-1986. Adjustments to groove sizes can be made where specific 'crush' is necessary for special applications.
- Viton rubber 'o' rings to be used unless a special requirement for an alternate elastomer is identified.
- Standard flange and seal sizes to be used unless an application prohibits their use.
- It is recommended that vacuum sealing surfaces are cleaned just before a seal is made to minimise the possibility of dust and hair settling on sealing surfaces.
- Metal seals may be used in specifically designed sealing surfaces including, aluminium and indium wires. They are very good as ultra-high vacuum seals.

#### 6. Screws

- All screws for flanges other than Conflat will be made from stainless steel in either 304 or 316 grades.
- Screws used in Conflat flanges will be made from A2-70 or A4-70.
- All screws to be socket head unless access necessitates hexagon or other special head. Slotted or Philips head must not be used.
- Stainless steel flat washers to be used under all screws where practicable. Do not use lock or star washers on standard flanges.
- In the vacuum space, no screws to be used in holes unless the holes are through holes and can be pumped from both sides. Screws can be used in blind holes if they have a minimum Ø1 mm hole drilled through the centre to allow trapped gas/air to escape.
- Unless a standard flange uses non metric thread, all screw threads must be within the metric series. Unless there is a specific need, avoid using intermediate metric sizes, that is, 0.5 mm steps such as M1.5 mm, M2.5 mm etc.
- Anti-seizing compounds can be used on threads but extreme care must be taken to ensure they do not enter the vacuum space. Graphite powder can be used as a lubricant inside the vacuum space. It can be applied using ethanol to wet the surface to be lubricated. Allow solvent to evaporate prior to pumping.

### 7. Feedthroughs

- Sliding seals should have dual 'o' ring seals with a gland formed seal at least at one end. The shaft finish must be 0.8 micron or better.
- All sliding shafts must have retainers to stop them being pulled through into the vacuum space.
- All coax feedthroughs to be glass or ceramic hermetically sealed. No plastic.

#### 8. Lubricants, vacuum greases and epoxies

- Only vacuum specific grease and oils to be used in vacuum systems.
- Silicone based or particular Apiezon family lubricants design for vacuum use, are recommended on sliding seals. Thin smears only.
- No vacuum greases should be used unless there is a small leak through a damaged sealing surface and all other options are not feasible. If grease must be used it must be designed for high vacuum use and only a very thin smear to be used.
- Do not use grease to fill suspected leaks in welds or fittings.
- Where a vacuum epoxy must be used the surface must be well prepared to ensure appropriate bonding. Care must be taken to ensure the epoxy seal doesn't create a trapped void of air which may become an outgassing problem. 'Torr-Seal' is commonly used due to its relatively low vapour pressure, and thermally stable to 120°C. (this is a Varian product)
- Vacuum oils are not recommended for general lubrication as they can creep on surfaces spreading contamination and increasing the outgassing surface.
- See Appendix 3 section 1.5.

#### 9. Mounting

- The load of the chamber assembly must rest on a standard accelerator beam line table ANSTO, HVEC, HVEE or NEC or a purpose built stand that minimises vibration.
- Vibration decoupling will be achieved by supporting the chamber separately from the vacuum pump and using an edge welded bellows between the chamber and pump.
- For ANTARES beam line vacuum equipment the beam axis alignment is nominally 1750 mm from floor height.
- For STAR beam line vacuum equipment the beam axis alignment is nominally 1193 mm from floor height.

#### 10. Access

- The working face of the chamber must face an area that is easily and comfortably accessible. The floor space immediately in front of the working face should be raised using steps to facilitate access.
- Heavy or awkward shaped vacuum equipment must be designed to be lifted with the aid of over cranes for example lifting lugs.

#### 11. Testing

- Vacuum equipment must be tested prior to routine use to minimise time spent chasing leaks in completed systems.
- Vacuum vessels must be helium leak tested to ensure all seals are better than  $1 \ge 10^{-10}$  Pa.m<sup>3</sup>/s ( $1 \ge 10^{-9}$  mbar.l/s). As a standard, during a leak test there should be no deviation from this baseline level for a leak-tight system. A record of the test is to be noted on the engineering drawing showing, date, maximum leak rate, leak location/s, leak detector used and operator name.

- For all other vacuum equipment in-service testing may be necessary to allow complete operation of various feedthroughs and positioners.
- Caution must be taken before positive pressure testing vacuum equipment as the equipment may be irreversibly damaged. Warning Do not use water testing on any vacuum equipment.
- A Residual Gas Analyser (RGA) can be used to give more detailed information about the gas composition inside a vacuum volume. An RGA can be used as a substitute for a helium leak detector with the benefit of providing more information about outgassing, or virtual leaks inside the vacuum volume. The pressure of the vacuum system must be  $< 1 \times 10^{-2}$  Pa to prevent damage to the filament inside the instrument. Instruction manuals must be checked before using the RGA head to ensure safe and effective use.

#### 12. Operation and Continued Monitoring

- All chambers should have a high vacuum gauge sensing within the chamber space. This includes all adjoining compartments that may be isolated by a valve.
- The chamber must have valves attached that allow complete vacuum isolation from the beam lines and other equipment.
- To protect high vacuum gauges against the effects of operating in low vacuum, high vacuum gauges should be automatically isolated if the vacuum pressure rises above  $1 \times 10^{-2}$  Pa.
- High vacuum gauges must be cleaned at minimum every 3 years or whenever a gauge develops instability as compared with adjacent gauge readings.
- Systems indicating higher than normally observed pressure must be isolated and repaired. A helium leak test is recommended if the leak location is not clear.
- To avoid undesired contamination, vacuum chambers should be vented with dry nitrogen or argon instead of air. This minimises water vapour and oil from depositing on the vessel walls.

### Chapter 2

# Design Criteria, Information and Philosophies for Vacuum Systems used in the Accelerator Area

#### Overview

In an accelerator system ion beams are transported from ion sources through to end stations (or detectors) through vacuum space maintained inside of beam tubes (lines). The relatively low number of gas molecules in the vacuum ensures a higher mean free path for the accelerated ions and hence less loss of ions through collision which is crucial especially for AMS measurements when counting a very small number of ions. A high vacuum also minimises the formation of plasmas in an electric field at high voltages which can avalanche into spark breakdown damaging sensitive equipment.

The vacuum systems used in the accelerator area are designed to be fail-safe to protect people and equipment. A very high proportion of problems with vacuum systems relate to, seals leaking on first use, being incorrectly mounted or failing due to aging 'o' rings. Other problems, however less frequent, include mechanical damage to sealing surfaces or fatigued parts such as bellows. The problems are all characterised by a poor vacuum or a previously steady vacuum that begins degrading slowly as observed during the operation of the vacuum system over its life time. The remaining problems are due to poor welds or vacuum fittings breaking. Poor welds are usually found during the initial helium leak test. They are remedied by either removing and re-welding the section or using a commercial vacuum epoxy. The choice is dependent on whether the weld is on a structural component or just a seal. Broken vacuum fittings are replaced or repaired.

Within the accelerator area. physicists work directly with technicians who apply broad knowledge and experience in vacuum technology to develop their concepts for use in the accelerator environment. This mix has been successful for 50 years. However not all designs are completed in the accelerator area. There are cases where commercial projects call for a more production based design or designs of chambers with complex noncylindrical shapes. These may go to the ANSTO drawing office for completion or a specialist vacuum design company.



Fig 1. ANSTO designed and developed beam lines on the STAR accelerator in building 22

It is not possible and often not necessary to standardise the design requirements for all components used in the accelerator area so the following information is intended to help designers of vacuum equipment to comply with accelerator specific and Australian standards. As of 2011, there are no known commercial workshops in Australia who specialise in the production of vacuum vessels. Local representatives from vacuum suppliers can provide access to overseas specialist workshops.

# 1. Terminology

**ANTARES** – Australian National Tandem for Applied RESearch. The HVEC FN Tandem accelerator in B53.

Backing – To pump on the exhaust (back) of a high vacuum pump

Beam line – The evacuated tubes that carry the ion beams from the ion sources to the end stations

**Cryopump** – Cryogenic vacuum pump. Uses a helium cooling interface to the vacuum space to freeze gases.

**Dependex** – A type of flange which is the standard flange used on ANTARES and ANSTO built beam lines.

**Fore line** – The vacuum line, tube, pipe, whatever between the high vacuum pump and the backing pump

High vacuum – See details in Chapter 3, para 1.3

ISO -- International Standards Organisation

KF – Klein (small) Flange

LF – Large Flange

Low Vacuum – See details in Chapter 3, para 1.3

Outgassing - the liberation of gaseous vapours from the surfaces within a vacuum space

Pa – Unit of pressure, Pascal. 1 Pa = 1 N/m<sup>2</sup> = 0.01 mBar = 7.5 mTorr = 1.45 x 10<sup>-4</sup> PSI

**Pump down = Pump out** – evacuating gas from a vacuum space

**Roughing or Roughing-out** – Usually used to describe the initial transfer of gas from atmospheric pressure to low vacuum.

STAR – Small Tandem for Accelerator Research. The HVEE Tandetron accelerator in B22.

Turbo – an abbreviation for turbomolecular vacuum pump

**Ultra-high vacuum –** See details in Chapter 3, para 1.3

Venting – the equalisation of negative pressure to atmospheric pressure in vacuum equipment

Vessel – generic for a vacuum chamber where an instrument is mounted or interfaced into the vacuum space.

### 2. Standard Units

#### 2.1 General

Although metric standard is adopted across all ANSTO accelerator facilities it does not mean that all equipment is metric as the US built FN Tandem is mostly imperial, conversely, the Dutch built HVEE Tandetron is all metric. There is a combination of metric and imperial devices used throughout the facilities but designers must ensure that where practicable all new designs are metric.

#### 2.2 Threads

New equipment for use in the accelerator area, where practicable will be designed with metric standard threads.

• Contrary to the standard, on ANTARES there is a combination of threads in use. The accelerator high voltage generator and parts that have originated from the United States are

mostly imperial. Typically, UNC and some UNF. Most beam lines and endstations are metric but all Dependex and most Conflat flanges are imperial.

• ON STAR all threads are metric. ANSTO designed and built beam lines are metric except for the Dependex flanges which are UNC.

Care must be taken to identify threads and use the correct screws. Where practicable, metric threads are to be used other than on Dependex flanging.

#### 2.3 Pressure

All pressure indicating devices shall be in the SI unit, Pascal. Historically, Torr was used but this was replaced by Pa in the late 1980's. Most international accelerator labs use mbar. The conversion is as simple as multiplying mbar by 100 to get Pa. Pascal refers to a physical force of newtons per metre squared.

# **3.** General Design Philosophies

There are many varieties of vacuum chambers and equipment in use in the accelerator area. Most have been designed and made at ANSTO. Examples include vacuum equipment measurement chambers where samples are measured, beam line transport components, ion source components or sensitive detectors, to name a few. Each has its own unique characteristics and vacuum design considerations.

In order to maintain a suitable mean free path for ion beams whether being transmitted from a source or scattered from a sample into a detection system, high to ultra-high vacuums are necessary. In a high vacuum the density of gas is low enough to minimise electron flow from surfaces at high voltages to surfaces at lesser potentials. It is therefore prudent to ensure designs of vacuum equipment that directly couple to the accelerator are designed for a minimum of high vacuum operation.

In every case the designers must ask the following questions:

- What shape? How big? Where will it fit? How will it be mounted? What level of vacuum?
- Will it be ridged enough and have low enough distortion to mount ion optical components?
- Is the design flexible enough to enable small scope changes in the future? Is it designed with consideration for reuse in another application when the project finishes?
- Does the design use vacuum technology best practises?
- Does it meet Australian and local standards where applicable?
- Is it a safe design?
- Is it achievable with resources available?
- Is it cost effective. Can we modify existing equipment to achieve a similar result?
- Does it have low environmental impact?

#### 3.1 The Shape

The first consideration is to define the actual use of the vacuum equipment relative to the experiment. Where possible cylindrical shapes should be considered first as they are stronger under vacuum and consequently being round, ports can be welded into the chamber aligned radially to the centre which ideally aligns instruments including detectors and cameras with the intersection point of the ion beam and the sample being measured.

Non-cylindrical chambers must be carefully designed to included adequate strengthen ribs to minimise distortion and hence misalignment of instruments.



Fig 2. A few examples of vacuum vessels used in the accelerator area

### 3.2 The size

The size is dictated by the space needed inside of the vessel or equipment in order to meet the operational demands. In the case of measurement chambers too big may mean problems with the distance between samples and detection systems and too small may mean manual handling of samples may be inhibited. Large chambers may have to be certified as per AS 4343-2005, Hazard Levels of Pressure Equipment in Australian Standard.

#### 3.3 Location

It will be an agreement between the accelerator user groups as to where equipment and beam lines will be located. This agreement will endeavour to minimise conflicts in beam line space, usage, accessibility and resourcing.

#### 3.4 Mounting

In general equipment must be secured onto the floor unless it is designed as mobile. An accelerator compliant beam line table should be used with standard beam line supports. Vibration must be identified during the design stage and decoupling stages provided.

Insertion bellows must be provided in long sections of beam lines to enable compression of the beam lines to facilitate dismantling and to ensure minimal strain on vacuum joints.

Designs must consider trip, bump and fall hazards. Structures that support platforms or heavy equipment that may otherwise fall must not be reliant on the structure supporting the vacuum chambers and beam lines. For example, ladders, walkways, etc.

- For ANTARES beam line vacuum equipment the beam axis alignment is nominally 1750 mm from floor height.
- For STAR beam line vacuum equipment the beam axis alignment is nominally 1193 mm from floor height.

#### 3.5 Flexible design

It is prudent to ensure where practicable designs ensure some redundancy for feedthroughs, access ports and internal space. A major problem with early chambers was the lack of space allowed for inserting samples by hand or manually manipulating internal equipment and wiring. It is easier to design flexibility into the chamber or equipment at the design stage rather than having to dismantle it and have it reworked in the workshops later.

#### 3.6 Can it be made?

When designing any equipment seek advice from a qualified machinist. Often designs include machining that may be very expensive or not possible. Early in the design phase ensure that the resources are available to service the vacuum equipment. It is recommended to consider the following early in the design stage:

- Power
- Cooling water
- Air cooling
- Signal lines
- Control lines
- Add-on electronic and mechanical instrumentation

#### 3.7 Safe design

There are no cases that the author has been able to find of scientific vacuum chambers or equipment, like those used in accelerator facilities, failing and causing injury. Vacuum pressure is relatively low, -100 kPa, or about half the equivalent magnitude in a car tyre.

For safe designs the structure must meet the minimum dimensions as calculated in Appendix 1 - Structural Calculations for Scientific Vacuum Vessel Design.

Care must be taken with the design of the following:

- Glass view ports (most glass breakages are in the form of a crack which leak to atmospheric pressure quickly without the spread of shards)
- No internal sharp edges; in the case where a person must insert their hands blindly to access components.
- Venting limits pressure relief for positive pressure?
- Evacuation rate regulation; especially in the case where very thin material windows separate different pressure systems within a single vacuum space for example beryllium windows on x-ray detectors.
- Equipment mounts/stands; where an operator must climb up high to operate devices on the system
- If oil type vacuum pumps are used then mist filters must be fitted to the exhausts.

### **3.8** Cost effective design

All *bells and whistles* may not be necessary even after considering some design redundancy for future changes. Where possible, designs should consider integrating components that are available off the shelf. Designing what has already been designed by someone else is wasting time and effort.

It may be cheaper to modify an existing chamber for use rather than start from the beginning. Many chambers used around the accelerator area have successfully had one or more lives.

#### **3.9** Design by numbers

This is a matter of ensuring structural integrity and being able to prove it. Likewise, designing the vacuum pumping system requires calculating the most appropriate size pump rather than going for the "biggest"!

There are formulas in Chapter 3 that allow must crucial calculations to be made from wall thicknesses to conductance. Good enough is not acceptable. Replication of previously designed equipment is acceptable.

#### **3.10** Australian Standards

Compliance is mandatory however there are few standards that are relevant to scientific vacuum vessels and equipment. The industry has developed its own standards for flange and seal designs which can be seen in the catalogues of most leading manufacturers of vacuum equipment. It is the responsibility of the designer to ensure compliance with all relevant Australian Standards. Chapter 1 discusses what standards are included.

#### 3.11 Environmental impact

Most vacuum systems used in the accelerator area today are designed to be oil free. That is, they do not use oils for lubrication of vacuum seals. There is however some oil vane and diffusion pumps still in use but the oils are responsibly disposed of through waste management at ANSTO. Some old diffusion pumps had been used with mercury as the pumping agent. Personnel must be cautious of using old diffusion pumps and their associated backing pumps as there may be residual mercury on surfaces and in the backing pump oil.

Solvents used for cleaning are used in small amounts and usually for wiping rather than immersing. Where baths are needed the solvents are reused when possible. The solvent bath used for degreasing, self recycles the solvent by design however there is a small loss of solvent over time into the atmosphere.

Where possible solvents used are selected with the environmental impact in mind.

#### 3.12 Manufacture

If ANSTO cannot provide the level of manufacture quality in-house then scientific vacuum equipment is best manufactured by companies that specialise in high tolerance machining and welding. A general metal fabricator will not suffice.

Attention must be paid to considering standard sized tooling during manufacture. Some companies will charge for the purchase of tooling that they consider to be non-standard.

It is imperative that the drawings for manufacture state all tolerances and qualities for the finished product.

On all design drawings, where the angle of the welded ports is crucial, ensure angular tolerances are clear and achievable. If necessary, specify the use of mandrels to hold ports in place during welding.

Some success has been made outsourcing the manufacture through vacuum companies to overseas manufacturers who specialise in high quality small runs.

#### 3.13 Testing

Testing the vacuum equipment or chamber is essential to establish compliance with the design and the all important ultimate base vacuum. Helium leak testing is the standard method used for chambers and most equipment however on some equipment in-service testing may be the only option. This is where add on vacuum equipment doesn't change the original base vacuum of the system it is connected to.

#### 3.14 Inspection

All parts should be cleaned prior to inspection. This doesn't need to be the final clean but enough to ensure all welds and surfaces can be clearly inspected. Welds must be checked for pits and alignment along the weld lines and seals must be checked for surface finishes. All crucial dimensional must be checked. It is recommended that any non-complying findings are photographed and documented in the project file. All non-complying parts should be reworked rather than *patched up*.

#### 3.15 Certification

As most vacuum vessels and equipment in the accelerator area fall within Hazard Level E of AS 4343-2005, certification is a local process. For large measurement vacuum vessels, results from the inspection and leak testing will be recorded on the engineering drawing and registered in the local accelerator register and/or ANSTO drawing register as appropriate.

#### 3.16 Documentation

For all large measurement chambers requiring any level of design calculations, design documents must be kept with project files.

#### 3.17 Maintaining

Vacuum chambers and beam lines do not require high levels of maintenance other than a general periodic clean and occasional seal replacement. Vacuum fittings and devices will have their own level of maintenance so access to these components must not be difficult.

The design must be easy to maintain especially moving parts, sliding or rotating seal and other parts in the vacuum space that may wear. Also vacuum pumping systems that may be mounted under or near the vacuum chambers. Generally any part that will require any level of routine maintenance must be accessible without the need for a major disassembly.

Vacuum pumping systems other than oil free systems, connected onto beam lines and chambers must include suitable oil traps between the backing and high vacuum pumps and where mist filters on all exhausts.

Some high vacuum gauges will require periodic cleaning. On ANTARES high vacuum cold cathode gauges are scheduled for cleaning every 12-18 months or max 3 years for difficult to service or less crucial instrument locations. Otherwise gauges will be cleaned on demand within the periods.

### 4. Performance and Operations

#### 4.1 Ultimate Vacuum

The ultimate vacuum is the maximum vacuum or lowest pressure the vacuum equipment typically reached after 3-5 days of uninterrupted pumping. This is a benchmark value which is used to assess the long term quality of the vacuum system. It is sometimes referred to as the *base vacuum or static vacuum*.

Generally, vacuums in the beam lines and chambers are well within the high vacuum range. Vacuums lower than this range will indicate a higher than normal gas load, a failing seal or a pump malfunction. On the accelerators the two main types of high vacuum pumps are turbomolecular and cryogenic pumps. For a system designed for high vacuum or better with no leaks and low outgassing then vacuums better than  $4 \times 10^{-6}$  Pa should be readily achieved.

The ultimate vacuum is improved by the correct choice of materials, seal types, high vacuum pumps used cleanliness and correct operation. All fittings must be designed or selected deliberately for high to ultrahigh vacuum use. Surface preparation and cleanliness will contribute to improved vacuums. A finger print can outgas for long periods of time. A bigger (higher speed, higher ultimate vacuum) vacuum pump does not mean a better vacuum!

#### 4.2 Working Vacuum

The working vacuum may be lower than the ultimate vacuum due to sample cycling (changing), introduced gas loads from adjoining systems or outgassing samples. A system must be designed to ensure the high vacuum pumping system has the optimum pumping speed and ultimate vacuum. Once a vacuum system reaches the "working vacuum" little operator intervention is necessary to maintain it but there are some important points regarding ongoing monitoring to ensure a failure is not immanent.

For sections of beam lines that are holding a static vacuum a cursory glance at the local vacuum gauge will indicate the stability of the vacuum. Generally, the (vacuum) pressure will change slightly throughout a day for example due to diurnal changes in ambient temperature and perceived changes due to ionisation gauges "wandering" as they become dirty.

In sections of beam lines that are in the vicinity of operating beam lines or near end stations the vacuum will be continually changing. Factors that influence these changes include increased gas loads from samples outgassing or surfaces that have just be exposed to high humidity, outgassing due to temperature changes within the vacuum space (including bake outs), outgassing from ion beams striking plastics, etc. Gas molecules don't have a preference to whether they travel towards or away from a pump during molecular flow so the source of gas can come from just about anywhere in the open system. These factors must be considered when monitoring vacuums throughout an entire system.

If a vacuum system's pressure rises slowly over many days or weeks if it is not related to the cryopump loading then it may be a seal failing. In this case a helium leak test is recommended.

A fundamental flaw in many vacuum system designs is the neglect for monitoring the backing pressure between the backing pumps and turbo pumps. Pirani gauges which are best suited for this role are relatively cheap and are the best diagnostic tool for determining overall vacuum system performance. The backing pressure should be consistent with changes in the high vacuum pressure. By monitoring the backing pressure over time, the base vacuum will be realised relative to the high vacuum so a pending failure of the backing pump can be determined, hopefully, well before it happens.

Care must be taken to maintain systems that have the potential to degrade over time for example an oil vane roughing pump will back stream oil mist that may accumulate in the high vacuum side of the system over time. This will contribute to lower vacuums due to higher gas loads from the partial pressures of contaminants.

#### 4.3 Sustainable Operation

Now that the vacuum system is designed, manufactured and commissioned for routine service, operating procedures have to be defined and adhered to, to maintain reliable trouble-free service. There are 3 processes that summarise the continuous cycle of operating a vacuum system:

- Pumping down from atmospheric pressure
- Using the working vacuum
- Venting to atmospheric pressure

The accelerator beam lines are divided into stand alone vacuum systems but in essence function as one. Each system can impact on a neighbouring system if things go wrong. In the case of end stations which cycle up and down in pressure more than any other devices an operator must be 100% sure of the pump down sequence in order to avoid damaging, pumps, samples, high voltage devices, detectors, etc. It is possible to shut down the accelerators as well. Most sections of the accelerator vacuum systems are interlocked to isolate a pressure surge to small area. Beam line vacuum systems are design so that both the vacuum pump and the beam line are isolated via gate valves. This is a typical arrangement for endstations as well.

The following must be read remembering that all 3 processes work in a cyclic fashion in a continuous loop.

#### 4.4 **Operational Protocols**

Key vacuum systems operate continuously, 24 hours per day, seven days per week. There are several vacuum systems that are cycled on and off as usage demands. All vacuum systems have one or more high vacuum gauges, some of which are interlocked to make the systems fail-safe. Any slow leaks or major catastrophic failures will close valves and isolate the system. In the few cases where no interlocks are used, a catastrophic failure will result in the high vacuum pump shutting down.

- For a <u>turbo pump</u>, as the pressure rises the gas load on the pump will cause the pump to draw more current to maintain it's speed. It will eventually fail when the current trip level is reached in the controller.
- For a <u>cryopump</u>, as the pressure rises the inflowing gas will condense or freeze onto the in the pump until it begins to warm up. Eventually there will be a thermal trip activated o the compressor which will shut is down. In both cases it is fail-safe.

As part of the accelerator's maintenance system, daily monitoring of all vacuum pressures is carried out. Where a pressure is seen to rise by a few percent without a particular reason, further monitoring will occur and corrective action started. Instantaneous failures are dealt with immediately. All observations and whether any maintenance carried out is noted on the maintenance log.

Where a vacuum system is isolated for service or where it has failed the appropriate tags are placed on the power leads.

Unattended out of hour's operation notification is not normally used as vacuum systems run continuously.

#### 4.5 Pump down

#### General

Pump down time is when most mistakes are made and significant time lost. Before a pump down begins the operator must ensure that all seals that were disturbed had been cleaned and replaced if damaged. The system must then be sealed ready for pump down and the roughing-out valve on the rough-out port, closed.

If a dry pump is to be used, such as a scroll pump, for rough-out then the tube is connected to the pumping port. If an oil vane rotary pump is used then ensure the cold trap has been filled beforehand.

#### Rough-out

Start the roughing pump and allow it to reach base vacuum. This is your first indicator of whether you have a leak in the system being pumped and when you have reached the pumping limit of the

pump. Slowly open the pump out valve on the system. This is where mistakes are made and damaged caused. Relatively speaking the volume in most sections of beam lines and endstations is small and the bulk of the gas can be removed very quickly. The problem is that a reduction in pressure too quickly can cause mechanical shock to fragile parts that must adjust slowly in order to minimise stress such as thin windows on detectors and detectors or devices that contain carbon foils.

Operators must always open roughing out valves slowly then gradually increasing as the gas load reduces. The change in the sound of the loaded pump is a very good indicator. A good design for more sensitive pump outs is to use a metering valve in line with the rough-out port to act as a regulating orifice.

#### Cryogenic Vacuum Pumps

For systems using cryopumps as the primary high vacuum pump it is essential to bring the base pressure down to the limit of the roughing pump before changing over pumping to the high vacuum pump. This reduces the gas load to be trapped (captured) on the cryo pump and extends the service life of the cryopump. Cryopumps are widely used on ANTARES as the primary high vacuum pumps but their limitation is that the warm up as they trap more gas causing a rise in base pressure. That is, when a cryopump is reaching saturation the temperature on the surface increases. More energy is required to hold the molecules on the surface and since it is almost fixed by design the temperature increases. A cryopump's effective pumping speed depends on the freezing and boiling points of the gases being pump relative to the cryopump's temperature. As the temperature increases more molecules leave the surface (boil away) raising the pressure. Some molecules are re-trapped as they hit the surface of the arrays and loose kinetic energy but they have a short residency time and soon add to the rising pressure in the system. Eventually the base pressure in the system becomes unusable so the pump must be regenerated.

#### Turbomolecular Vacuum Pumps

The other type of high vacuum pump widely used in the accelerator area is a turbo pump. Unlike a cryo pump that traps or captures gas molecules a turbo pump transfers the gas by conducting it almost one-way out of the vacuum space. The lowest roughing pressure before changing to a high vacuum pump is not as crucial when a turbo pump is being used. In saying this, the bulk of the gas must be removed before changing from roughing out to high vacuum pumping otherwise the vanes on the turbo pump may be overly stressed due to the pressure surge. A typical changeover pressure range is 5 - 50 Pa.

The optimum changeover time to minimise pump down times can be estimated with the assistance of the formulas found within the document. Letting the roughing pressure reduce to the limit of the roughing pump before changing over to the turbo pump is an inefficient method of system pumping. A turbo pump is more efficient at removing gas molecules as the flow moves towards molecular flow. Changing pumping to a turbo pump just before molecular flow is reached will increase the pumping speed and base vacuum will be reached sooner.

#### Pumping Speed

Each type of gas will pump away at a different speed depending on the mean velocity of the molecule which is a function of its mass. A hydrogen molecule of 0.02 kg/mol and mean velocity of 1762 m/s will reach a pump much quicker than say a Xenon atom of 0.13 kg/mol with a mean velocity of 217 m/s.

#### **Limitations**

In rare circumstances roughing pumps may be left pumping high vacuum systems by mistake. It is unlikely to damage either pump but the ultimate vacuum will not be reached as the roughing pumps are ineffective at conducting away molecules in molecular flow. Water vapour and residual solvents are difficult to remove from a vacuum space with *normal* vacuum pumping. Most low vacuum backing pumps provide gas ballast which can speed up the removal of water vapour and solvents that may otherwise condense inside of the pump during the compression stage. By introducing a small amount of air near the outlet of the compression stage the small increase in pressure helps carry water vapour and solvents to the exhaust before they can condense.

Some vacuum systems have been designed to allow chambers and beam lines to be heated. Heating causes contaminants to be removed from surfaces at lower than normal vacuums. The higher the temperature the faster the removal will be. Care must be taken to ensure heating doesn't inadvertently cause plastics and elastomers used within the vacuum space to outgas. Most bakeable systems use ceramics and metals only.

It is important that where vacuum systems are permanently connected to high voltage devices for roughing out, the insulated (plastic) pump out lines between the roughing pump and high voltage device must be brought up to atmospheric pressure before high voltages are reapplied. Otherwise the low molecular density of gas in the tube may ionise causing burning of the tube or even spark damage that can puncture the tube causing a leak.

### 4.6 Venting

#### General

Venting can cause damage to vacuum components just like pumping out a system from atmospheric pressure too fast.

#### Venting Speed

As discussed earlier the volume of vacuum systems on the accelerators is measured in litres to a several tens of litres. A catastrophic loss of vacuum will be more of a brief audible event rather than a dangerous mechanical failure. Little energy is required to change these systems by 100 kPa. There are a few devices in the vacuum space that may be damaged due to pumping out too fast but none that are considered a danger to personnel.

Other than venting too fast the second point to consider is the introduction of water vapour if venting with air. Key vacuum systems on the accelerators are vented with either dry nitrogen or argon both of which are delivered through a closed reticulation system from gas bottles. The choice of gas is dependent on molecular contamination within the system for example nitrogen is not preferred to vent the 846 ion source as nitrogen is an element that is known to bond with other elements causing molecular ion beams.

#### Regulation of Flow

There are many low flow regulators and rotameters (tubular flow meters) available that provide adequate control over venting. A simple in-house gas regulating system which works well is a tee piece connected between the venting port and the venting gas supply line. The centre port of the tee piece faces upwards and a ping pong ball sits over the hole with a cage over it so that it cant be blown away. Under the ball is an 'o' ring seal. Before the venting valve is opened the venting gas is allowed to flow. The pressure of the gas lifts the ping pong ball of its seal. The gas flow is adjusted depending on the height of the ball above the seal. The ball can only travel a centimetre or so. Once the flow is set the venting valve is opened and the now regulated gas flows into the vacuum space and the balls drops onto the seal stopping air from entering the vacuum space. As the pressure reaches equilibrium the ball begins to rise off the seal until eventually it rises to the original set height indicating the vacuum space is now at atmospheric pressure.

#### Precautions

- Before venting a vacuum system it is wise to isolate all ports and beam lines that must remain under vacuum. To save stressing a beryllium window on an x-ray detector due to the changes in pressure the system should be designed so that the x-ray detector can be isolated with a gate valve. The same logic can be applied to all equipment that may be effected. It is also important to ensure all power supplies and high voltages that may be hazardous to personnel or equipment are isolated prior to venting. This can be done either by administrative protocols or using a vacuum switch such as programmable relays in a vacuum gauge controller.
- Some vacuum gauge controllers use the signal from a Pirani gauge measuring at the same location as a high vacuum gauge to switch off the high vacuum gauge when the pressure becomes too high. This saves the gauge from ionising air when the system is at atmospheric pressure which will significantly reduce its life time.
- Another problem with venting too quickly is the disturbance of dust. Accelerator tubes must be vented slowly to minimise equalisation stresses and also the disturbance of dust. In this case a metering valve is used to limit the vent flow rate.
- Venting can cause loud high frequency noise so care must be taken to dampen the noise to a comfortable level by controlling the air admittance rate, or use hearing protection.
- Most beam line vacuum pumping systems are interlocked to isolate either the beam line or pump or both. In the event where an accidental venting occurs the gate valves will close minimising the effect on surrounding systems.

# 5. Safety

#### 5.1 General

Catastrophic failure causing injuries are not common and no reference is known from overseas accelerator facilities. This is almost certainly due to the low pressures involved in vacuums and the size and nature of the scientific apparatus. There are of course many failures reported for industrial sized tanks and equipment.

The vacuum systems are essentially fail-safe from absolute vacuum to atmospheric pressure. The worst case has been a number of beam lines and measurement chambers developing leaks and thin windows that have ruptured. In each case there has been a gradual rise in pressure to atmospheric pressure.

However, a few rules do apply.

- All vacuum systems must have one or more vacuum gauges attached with the exception to some static chambers used for storing moisture sensitive items or radiation detectors.
- High vacuum gauges must be switched off before vacuum systems are vented to atmospheric pressure.
- Only nitrogen, argon or other inert gas to be used for venting. No flammable gases to be used such as hydrogen.
- Turbomolecular pumps must not be vented from full speed. See manufacturer's recommendations.
- All designs must be consistent or better than the best practises outlined in this document. Inferior equipment must be isolated and reported to the Leader of Accelerator Operations.

- Cryopumps must not be operated without a serviceable pop off valve.
- Cold traps must be warmed and blown dry at least every 6 months to minimise  $O_2$  concentrating (and for de-icing as necessary).

### 5.2 Interlocks

Nearly all vacuum systems on the accelerators are interlocked via a set of integrated relays in a vacuum gauge controller, to protect equipment. Generally, if a rise in pressure is detected above a particular set level then the interlock relays will activate to close corresponding gate and line valves. This is designed to minimise the number of vacuums that must be regenerated in a fault. The interlock system also protects accelerator tubes from exposure to moisture and in the event of a catastrophic failure of the accelerator tubes; the vacuum system will close the main accelerator gate valves to trap SF6 gas.

If an interlocked failed causing a gate valve to stay open in the event of a pressure rise then the next interlock should activate the next gate valve. If the next interlock fails it is possible that the two interlocks share the same vacuum gauge controller which is at fault. In that case the next interlock should activate. If no interlocks activated there would be no increased risk of injury to personnel but all vacuum systems will require regeneration or a restart after the interlocks were repaired and tested.

#### 5.3 Fumes from exhausts

There are a number of oil vane pumps in use on the accelerators. In the past, the exhaust on the pumps has released oil mist into the atmosphere inside the buildings. Now, on nearly all oil vane pumps in continual service there are mist filters attached or the exhaust is plumbed into an extraction system that vents the exhaust fumes outside of the building.

#### 5.4 Earthing

Some vacuum gauge controllers are prone to high voltage transient surge damage. These sometimes cause unnecessary isolation of systems requiring resetting. It is very important that all vacuum gauge controllers are well earthed.

### 6. Serviceability

#### 6.1 Access

Most beam lines with the exception to some measurement chambers are static volumes of vacuum requiring little operator intervention other than during preventative maintenance. Vacuum pumping systems do require a higher level of intervention during maintenance and operation. With these points in mind the designer must ensure that all controls and monitoring devices are localised at the working face of the beam line or chamber.

Due to beam lines being so high above floor level, working on them usually requires some form of ladder or steps. When designing beam lines, be sure to have pump out ports facing the passageway beside the beam line. The same applies to vacuum chambers and other large volume vessels.

Measurement chamber will need to have the access port accessible from a location near the control side of the chamber. A platform is recommended to allow easy reach and eay viewing into the ports.

#### 6.2 Maintenance

A well designed vacuum system will require routine maintenance including:

- Changing faulty seals cleaning seals that are regularly opened
- Cleaning vacuum gauges especially cold cathode type high vacuum gauges
- General cleaning inside of measurement chambers and equipment that is; regularly opened and touched, whenever it looks dirty, when the base vacuum degrades or when the surfaces are exposed to the atmosphere for long periods at a time (weeks).
- Servicing of vacuum pumps the group of the most routine maintenance tasks includes; cryopump regeneration, turbo pump bearings, scroll pump tip seals and oil vane pump oil changes to name a few
- Actuator service sliding shafts that penetrate into the vacuum space

#### 6.3 Cleaning

Cleaning is by far the most essential part of general maintenance of a vacuum system. Poor cleanliness will lead to poor vacuums and possibly sample contamination. It is a general philosophy that cleaning is done with solvents, wiped over and/or scrubbed and thoroughly dried.

#### **Solvents**

Typically ethanol, methanol and acetone are used. These solvents remove most greases and oils that are encountered. What ever solvent is chosen the most crucial point to remember is that it must not leave any residue.

For surface finishing see section on 'Surface Finishes'.

Chemical polishing is not commonly used in the accelerator area due to the size of the baths that are needed for large parts, the problem of cleaning or neutralising residual chemicals and not having a suitable facility for handling the types of chemicals used. Although once used electro-polishing is no longer encourages for the same reasons. If suitable facilities were available both chemical and electro-polishing are real options for very good cleans.

A general clean will consist of a number of different levels of cleaning. A single clean with the same solvent will not remove some oils and greases therefore a progressive approached is used using a variety of solvents and wiping materials. The following is a guide only of some such techniques.

Using a dry gas or filtered compressed air, most loose dust can be blown away. This is sometimes the best starting point for cleaning. A final blow down is also useful to ensure complete removal of lint.

The first clean will take away coarse particulates or chemicals. Ethanol dampened tissues if good for this level of cleaning. The ethanol helps remove some oils and greases and mixes with water to reduce the evaporation time. It is also good for mopping up dirt and dust. Scour pads (or 1200 wet or dry paper) moistened with ethanol is good for removing stubborn dirt. This must be continued until better than 95% of dirt, dust, oil and grease is removed. For stubborn dirt it may be necessary to use a petroleum based solvent followed by an ethanol rinse and wipe. Wiping is key to cleaning. The light mechanical brushing dislodges most dirt.

The second clean starts with ethanol but finishes with acetone. Only lint free wipes are used such as Kimwipes. At the end of this level of cleaning the equipment must look ready for use in the vacuum. That doesn't mean it is. It will more than likely have residues remaining on the surface.

Final clean. It is recommended that methanol is used for the final clean or another low residue solvent. The final clean must use lint free wipes dampened with methanol and lightly rubbed over the entire surface. An alternative final clean is to use the solvent bath and suspend the equipment in the hot vapour for a few minutes. Care must be taken with the bath as the hot vapour will degrease skin very quickly.

If a shiny (healthy looking) clean is desired then a metal polish such as Brasso can be used. Whenever Brasso is used the residue must be cleaned off with ethanol. Care must be taken not to get metal polish pastes into gaps that cannot be thoroughly cleaned out. Brasso is an enemy of vacuum, it traps water.

#### 6.4 leak detection

In the accelerator area, a leak is characterised by a flow of gas molecules into a space that is below atmospheric pressure. The pressure of the gas entering the space will restrict the vacuum system from reaching its design pressure which is typically in the high vacuum range. As a benchmark, if a vacuum is  $1 \times 10^{-5}$  Pa or more then disregarding the effects from outgassing, there may be a leak. In system designed for ultrahigh vacuum the benchmark will be in the order of  $1 \times 10^{-7}$  Pa.

Leak detection is usually only necessary for new chambers or equipment. It can be done offline or after the part has been mounted into a beam line. The most common method of leak detection is a helium leak test. Helium atoms being so small will find their way through openings that are too small for most other molecules. Generally, the smaller the leak, the less helium that can make its way along a leak path. The leak detector is usually a mass spectrometer tuned to have highest sensitivity for helium. The detector is connected directly to the device being tested. Any helium that makes its way through the leak will eventually be measured in the mass spectrometer. Residual Gas Analysers (RGA), although not specifically leak detectors, can be used for just that. Working on the same principle, mass spectrometry, the RGA can detect helium as well as other gases.

In some cases leaks can be too large for a helium leak test, that is, the amount of gas in the device being tested is too high diluting the helium to an ineffective percentage. Also, if the pressure is too high the leak detector cannot be opened to the device as the pressure gradient will be too high. Leak detectors work best from the higher end of the low vacuum range.

Often there are multiple leaks in systems. It may mean repairing large leaks in order to find small leaks. Where a leak is too large for the helium leak detector a few other methods can be used to locate leaks.

- Solvents have a very low viscosity and vaporise very quickly in air. Wetting surfaces with small amounts of acetone or ethanol can show small changes on Pirani and Penning gauges.
- Isolation of seals can, by process of elimination, restrict the testing area allowing large leaks to be isolated. For example, if a chamber with many ports and devices attached has a leak. It may be prudent to close all valves leading away from the chamber except for the leak detector valve then sequentially blank off the ports one at a time (using a sound flange and new seal) and test for a leak.

### 7. Soldering wires vs mechanical joints

It is preferable that wires in a vacuum space are joined together with a mechanical clamping type joint. The clamp must allow for gas to escape. Where a direct joint is necessary then soldering is permitted. Standard lead/zinc with resin core is okay provided that ALL flux is removed with ethanol. This may require some soaking in solvent with gentle agitation. Solder without resin core

can be used on stainless steel with a zinc chloride based eutectic flux, used sparingly. Ensure all flux is removed especially between the wire strands.

Be aware that in Faraday cups the continual flexing of wires can work harden the wire causing it to break. These are typically soldered joints. If soldering, be sure not to melt the insulation. It is highly recommended to replace normal plastic insulation with loose fitting PTFE (Teflon) spaghetti.

# 8. Flanges and seals

#### 8.1 Common vacuum seals used in the accelerator area

A variety of standard vacuum seals are used on all vessels depending on the degree of vacuum to be achieved and the type of fitting that will be used. The types are widely used in the vacuum industry. ANTARES uses the Dependex style seals and flanges. This was developed for use with HVEC accelerators over 50 years ago. Other types in use include Conflat, ISO, and KF are used as well.

Туре	Vacuum level	Flange type	Seal type
KF	Low to High	Clamp (typically)	Viton 'o' ring, Al, PTFE
Edwards	Low	Threaded retaining nut	Viton 'o' ring
Dependex	High	Ring retained by spring clip	Viton 'o' ring supported by a ring
LF or ISO	High	Ring or clamps	Viton 'o' ring supported by a ring or in a groove
Conflat	Ultra high	Ring fixed or rotating	Copper ring, Square section 'o' ring
Specials	Various	Various	Aluminium or indium wire

Table 1. Common types of vacuum flanges and seals. Each type is available in multiple sizes

### 8.2 KF – Klein Flange

A common flange and seal used for general low vacuum applications due its ease of assembly and vacuum performance. It is common to have KF flanges and seals within the high vacuum side of a system. Vacuum performance can be enhanced by the use of aluminium or PTFE seals which directly replace the Viton 'o' ring.

Many general and specialised vacuum equipment is fitted with KF fittings as the range of adaptors from KF to other major types is readily available.





Fig 3. Assembly drawing for a typical KF flanges seal and a photo of the flange, seal and clamp

#### 8.3 Edwards

This is no longer a preferred type that is used in the accelerator area however some equipment still uses this type.

One end of the fitting is shaped to retain an 'o' ring while the other end has an angled sealing surface that mates and crushes the 'o' ring to make a seal. They are connected by a threaded nut which when tightened squeezes the two halves together. It is normally for low vacuum applications.

#### 8.4 Dependex

This was designed for use on HVEC Van der Graaff accelerators and beam lines. It was introduced to ANSTO in the early 1960's when the 3MV Van de Graaff operated in B22. Here, 1", 2" and 4" Dependex was used. When ANSTO purchased the FN Tandem nearly all fittings used were 4" Dependex (100 mm nominal). It is suitable for high vacuum use and often mated with Conflat flanges in systems that operate near ultra high vacuum.

The Dependex seal consists of a supported 'o' ring sealing between two opposing  $5^{\circ}$  metal faces. The 'o' ring is supported around its inner diameter by a ring to ensure the 'o' ring aligns with the centre of the 2 faces. The inner ring also sets the maximum crush on the 'o' ring that is the ring fits into a step on both halves of the Dependex seal keeping them separated at a fixed distance. Another ring which fits on the outer diameter of the 'o' ring does little more than keep the 'o' ring clean. The rings are often called *egg rings* due to their resemblance.

The Dependex flanges are held in place on a tube, or other fitting that is designed to accept Dependex, by a large circlip that fits into an outer groove a few mm from the end of the sealing face.

The flanges are normally drilled such that the holes alternate between being tapped or clearance. 1" and 2" Dependex use <sup>1</sup>/<sub>4</sub>" UNC, cap (socket or Allen) head screws and 4" (100 mm) Dependex use 5/16" UNC, cap (socket or Allen) head screws. Only use stainless steel screws (of one length) and washers. Discard plated steel screws as they are identified during maintenance. Washers are recommended as most flanges are aluminium alloy.

To complete the Dependex assembly, the flanges must be placed over the tube ends and then retained in place with a circlip. Secondly, the 'o' ring is stretched onto an inner ring and then an outer ring is placed around the assembly. The two Dependex ends are brought close together then the seal assembly is placed between them. The inner ring is aligned with the step on one side then the other tube is aligned and than pushed onto the ring. At this point it is important to ensure the 'o' ring assembly stays in place until the bolts are tightened. Screws should be finger-tightened and the flange faces must be parallel. Tighten screws in an opposite pattern. Some old flanges will have worn threads so screws may only be turned with the aid of an Allen key.

#### Problems

There are a number of things that can go wrong with assembling Dependex fittings.

- There have been cases where inner egg rings have been made too wide so that when fitted between the tubes the 'o' ring is prevented from crushing adequately to form a vacuum seal.
- Another problem with the inner rings is where some Dependex seals have been designed with too shallow steps so that a standard inner ring becomes too wide. In this case special inner rings have been made which are narrower. Personnel servicing non-standard joints must take care to reuse the right sized inner ring. A label should be placed on all non-standard joints identifying them.

- Some steps for inner rings have also been found to be out of tolerance preventing standard size inner rings from locating in the step. In this case some operators have cut a small section from the inner rings so that they can be squeezed together. Cutting inner rings like this is only recommended to solve this type of problem.
- Some flanges have been design for special applications with slightly different PCD's. These have unfortunately bee mixed up with standard stocks. As personnel who identify them (sometimes after a frustrating encounter) should put them aside and label them accordingly.
- Old flanges that have been over-tightened may have stretched threads. They are identifiable by the difficulty in starting screws. Either re-tap the threads or discard the flange. There have been case were tight threads have mislead personnel into thinking they have tightened the Dependex joint only to find leaks.
- Circlips can stretch especially those in over or under sized slots. They are easily identified by there distorted shape. Only use circlips that are flat when not under tension. A discerning technician should discard rusty circlips that cannot be successfully cleaned.
- Care must be taken to make sure the two faces of the Dependex joint are parallel before the joint is closed and bolted together. Failing to do this may cause a leak in the joint. It is acceptable if a bellows is used between sections that have misalignments.
- Twisted 'o' rings can cause leaks although small. There have been cases where a twist has caused the 'o' ring to bulge towards the outer ring making a less than adequate seal.

### 8.5 Special Flanges

These consist of flanges that have been designed to fit application where "off-the-shelf" flanges and seals cannot be used. They are typically based on common designs but modified to suit the application.

#### Example

The Alphatross ion source uses two stainless steel flanges to hold the heater reservoir in place. Here the vacuum sealing surface is a flat surface about 8mm wide on each flange. The fixed flange is threaded in an imperial thread and the reservoir flange is open hole. The gasket used is aluminium wire which has been fused together to make a ring. The joint is similar in size to the wire. The wire is supported in place using about 3 to 4 strips of aluminium foil looped around the wire and sticky taped to the edges of the flange. The flange is tightened in a circular pattern to ensure maximum crush on the wire.

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- The wire is essentially unsupported so vacuum must not be applied until the wire is completely crushed.
- Bulbous joints in the aluminium wire dent the sealing faces on the flanges as they are tightened so they must not be used.

### 8.6 ISO or LF (Large Flange)

This is a European range of fittings designed for high vacuums and to suit most tube sizes used in the vacuum industry. There are two types of design which meet most light and heavy duty applications.

ISO-K flanges are a robust joint which are suitable for supporting larger and heavier items such as vacuum pumps and large gate valves. They are characterised by the outer clamping system which holds the two halves of the joint together.

ISO-F flanges are more of a traditional bolted flange type joint like Dependex. ISO-F and ISO-K can be joined together using a half clamp which fits onto a standard ISO-F flange. The half clamps then attach to the ISO-K flange.

Available in sizes from 63 mm nominal bore to 500 mm NB.



Fig 4. ISO family of Flanges

#### 8.7 Conflat CF

Conflat is designed as an all metal vacuum seal for ultra high vacuum applications. A copper gasket is sandwiched between two thick stainless steel flanges that have a knife edge machined into the sealing face. As the flanges are tightened together the knife edges "bite" into the copper to produce an all metal seal with very low outgassing.

The term "ConFlat" is a registered trademark of Varian, Inc., so "CF" is commonly used by other flange manufacturers. Conflat sealed systems can reach vacuums as high as  $1 \times 10^{-11}$  Pa.

The copper gasket locates into a partially recessed in a groove in each flange to provide alignment prior to tightening. It also stops the gasket from moving during bake out. Conflat can be baked up to  $450^{\circ}$ C.

Most Conflat flanges have imperial sized bolts. The original style of bolt had a multi-face head that a ring spanner would fit over. This is to allow more purchase on the head from many angles. It is not uncommon now to find hexagon and socket head screws in use. Be aware that the steel must be a high tensile grade to ensure optimum force can be used to crush the gasket. It is essential that the flange faces are parallel during and on final tightening. A torque wrench may be used to facilitate uniform tightening. It is highly recommended that a molybdenum grease or graphite loaded grease is used on the threads.

Conflat flanges are available in sizes from 10 mm nominal bore to 250 mm NB. Sometimes nominal bore is written DN in front of the size, eg DN63.

Square section Viton 'o' rings may be used with Conflat flanges however this compromises the ultra high vacuum design and ultra high vacuums may not be reached.

#### Problems

- Copper gasket not aligned in groove and edge is partially clamped on the bolt hole face of the flange. This lead to partial seal and most likely, a leak. Most Conflat flanges have two small grooves a little more than a millimetre wide machined into the bolt face. These are for locating special clips that hold the gasket in place while the flanges are brought into place. Just prior to tightening they clips are removed. They are sometimes mistakenly called pressure relieving grooves.
- Copper gaskets must only be used once. Unless the copper hasn't been fully compressed after the first use there will not be enough material for the knife edge to reform a seal.
- Due to the forces required for a knife edge to indent a copper gasket the flanges are made of stainless steel and quite thick. Large flanges are quite heavy and care must be taken not to drop them.
- Where Conflat flange bolts have been used more than once the threads in the flange and screws may have stretched. In this case, it is recommended that the same screws are reused in the stretched threads (with a little molybdenum grease) as they will have the same profile. Sometimes using new screws in a stretched thread will cause binding. Re-tap the flange if necessary.
- Stretched threads are possible where nuts and bolts are used through open holes so ensure the nuts go onto the bolt the same way they were removed. Alternatively replace the nuts and bolts outright.
- For ultra high vacuum ensure the gaskets are handled with gloves. Copper reacts well with the sweat in a fingerprint leading to a long outgassing process.

# 9. Bellows

Bellows consist of two styles, edge welded for ease of extension or compression and corrugated style which is has minimal movement. Each style is made from a thin stainless steel that has been electron beam welded. The ends are usually finished in a standard flange to suit the application.

#### 9.1 Corrugated style

These are typically used for two purposes. Firstly, to allow sections of beam lines or equipment to be compressed to allow easy removal of sections without dragging the sealing surfaces against each other. Secondly, as bellows can distort and still maintain their structural integrity they are ideal to

join sections of beam line or equipment that have a deliberate mis-alignment. They can be purchased with almost any style of flange including specials. See example pictures.



Large bellows. ~300mm diameter. Stainless steel, wall thickness ~ 0.8 mm. This bellows is used to correct alignment offsets in the magnet box

Medium bellows. ~ 100 mm diameter. Stainless steel. wall thickness <0.5 mm. Used to allow sections of the beam line to be compressed to facilitate beam line component removal.



Small bellows. Electron beam edge welded stainless steel. Material thickness <0.25 mm. Used to isolate vibrations in the beam line.

Fig 5. Examples of bellows

#### 9.2 **Edge Welded Bellows**

These bellows are made up from very thin stainless steel discs stacked together and welded along their edges. The advantage of the design is the concertina style bellows that is very flexible allowing designs that have long extensions and short compressions. They have two main applications. Firstly, applications that requires a device to extend or contract into the vacuum space where an elastomeric seal on a sliding shaft is not adequate. Such as a sample manipulator or Faraday cup. Secondly, where a device such as a detector or sample positioner is sensitive to vibration and must be decoupled from the vibration source.

### 10. Valves

Along the axis of the beam lines gate valves are used to enable the isolation of sections for maintenance and also to minimise loss of vacuum in the whole accelerator if one section develops a leak. Most gate valves are electro-pneumatically operated and many are interlocked to local high vacuum gauges. The few that are manually operated are for maintenance isolations.

On ANTARES the electro-pneumatically operated valves are interlocked to close when the pressure, as measured near the gate valve, rises above 5 x  $10^{-5}$  Pa. This figure can vary depending on the location of the valve. See the Accelerator WIKI for values around the ANTARES. Once an interlock has tripped the switch that operates the gate valve is bypassed. To reset the valve:

- The vacuum must be within the high vacuum range as read on the corresponding vacuum gauge controller with a pressure better than the trip point.
- Once this level is reached the bypass button is held down momentarily.
- At this point the pressure will rise then fall as the gas load is pumped away.
- When the system pressure is less than the trip level the bypass switch can be let go.
- An indicator light near the switch will show its status.

#### Notes:

- Operators and technicians must be aware that gate valves must be orientated so that the gate always seals towards the vacuum pump. That is, they seal best in one direction only. Failure to orientate the gate the correct way may cause a vacuum leak when atmospheric pressure forces the gate off its seal.
- In some cases gate valves may remain sealed after they have been switched to open. This can be due to the withholding (sometimes called back pressure) pressure not being adequate enough to lift the gate off the seal.
- Gate valves, while mostly metal can become hot if a gate is closed in front of an ion beam. If left for long periods with high current beams the elastomer seals may warm up and begin to outgas.
- For roughing ports typically 25KF bellows sealed valves are used. On STAR these same valves are used to isolate vacuum gauges for cleaning.
- Where fine flow control is required for either roughing out or venting, metering valves are used. These are basically multi-turn needle valves for fine adjustment.
- Valves do require maintenance to keep the seals clean and in working order. After long periods of time the 'o' ring seals will deform and often will not recover their original shape.

There have been attempts to build fast acting gate valves for the purpose of isolating the accelerator tank's load of  $SF_6$  in the event of a breach in a tube. While it seemed like a good idea the force of the valve closing at high speed could damage the gate seal causing the valve to leak. Designers must use care to ensure that a standard high differential pressure rated gate valve isn't adequate before launching into these expense type valves.

# **11. External Interfaces**

#### **11.1 Electrical Feedthroughs**

a) Low Voltage, Low Current

There are few applications where voltages greater than 24Vac are used within the vacuum space. For these voltages at a few amps coaxial feedthroughs such as BNC, SHV, MHV, etc, are often used in place of more expensive dedicated commercial types. In one application 200Vdc is used for faraday Cup suppressors but current is only a few micro-amps at most.

Notes:

• Do not use multi-pin signal feedthroughs to carry voltages unless the pins used for voltage can be a few unused pins away from used signal pins.

- Feedthroughs can be purchased is almost any flange style but are usually restricted to small size flanges.
- Insulation must be used on all wires carrying voltage. It is highly recommended to use single or stranded wires (preferably nickel coated) with loose fitting PTFE (Teflon) spaghetti.

#### b) Signal

Signal feedthroughs other than coaxial such as BNC, SMC, Microdot, etc are from one to multiple pins. It is recommended that connections to multi-pin feedthroughs are by the use of slide on pins. Many are either sliver of gold plated for good electrical contact. Avoid soldering unless care is used to remove all traces of flux and no pin-holes in the solder have been created

On coaxial feedthroughs the coaxial connector will be either both sides of the feedthrough and hermetically sealed or with the coaxial connector on the atmospheric side and a plain pin on the vacuum side, also hermetically sealed. For either type, where coaxial cable must continue into the vacuum space it is recommended to remove the outer plastic insulation and replace it with loose fitting PTFE spaghetti. This is also recommended for all single or stranded wires carrying signals.

#### c) High Voltage, High Current

There is a wide range of high voltage and high current feedthroughs available in most flange types. High voltage feedthroughs usually have a larger insulator in ceramic and sometimes glass. These must be kept clean on the atmospheric side. Long term breakdown on high voltage feedthroughs can damage the insulator causing vacuum leaks.

For high current applications such as heaters, dedicated high current feedthroughs must be used. These are characterised by larger cross section wires to carry the higher currents. Like HV feedthroughs they must be kept clean. It is recommended to clean the insulators on signal feedthroughs periodically to remove any accumulation of dust.

#### d) Power

There are few feedthroughs designed for powering devices at mains voltages. It is highly recommended that experiments are designed not to have mains voltages in vacuum vessels or equipment due to safety concerns.

### **11.2** Mechanical Feedthroughs

#### a) Rotary

Rotary feedthroughs allow the transition of rotary movement into the vacuum space to, for example, operate shutters or position samples or detectors. They are widely available in many of the common flange styles. There are three common types both of which are very good in high vacuums.

The first type is a continuous shaft between the atmospheric side and vacuum side using rubber vacuum seals in the form of either supported 'o' rings, square or odd shaped rings (like common shaft seals), and flat rubber sheet seals. These are best for general rotary actuation rather than continually rotating. Okay in high vacuum but care must be taken to keep the seals clean and periodically replaced or whenever wear is noticed.

The second type is a ferro-fluidic type which also uses a continuous shaft between the atmospheric side and vacuum but the seal is made by encapsulating a dense fluid around the shaft. These types usually have bearings to keep the shaft centred and free moving. They are best for high revolutions in high vacuum applications.

The third type is magnetically coupled rotary feedthroughs. These are expensive. They provide a seal by magnetically coupling the shaft on the atmospheric side to the shaft on the vacuum side across a continuous sealing plate. If either shaft is removed the vacuum is not compromised. These are excellent for ultrahigh vacuums and lower. They are for moderate speed revolving applications and general actuations.

#### **b**) Sliding

Usually these feedthroughs are a round shaft that can slide in and out of the vacuum as well as rotate. Sliding feedthroughs are used for positioning devices or where a linear actuation is necessary. The length can vary from a few millimetres to hundreds. In either case there is a shaft which penetrates from the atmospheric side to the vacuum side. The type of seals used varies like in the rotary seal. Sometimes dual seals are used to minimise leakage especially in high actuation rate applications. In some applications the void between the two seals is pumped increasing the reliance of the seal. These types are good in high vacuums. Notes:

- Care must be taken to ensure the shaft is lubricated with a suitable vacuum oil or grease. It is imperative that the oil or grease on the shaft is changed regularly to minimise dust build.
- There are bellows sealed sliding seals available. These types do not rotate and can only move in and out a few tens of centimetres at most. Very good in high to ultrahigh vacuums.
- Some sliding seals are able to move radially which will affect alignment. Where alignment is crucial a linear bearing type is recommended.

#### c) Wobbler

A wobbler feedthrough allows users to use a shaft as a lever or pick up. They are designed around a flexible rubber or bellows seal they allows a greater range of circular movement.

#### 11.3 Manipulators

These are a class of feedthroughs design for high to extremely high precision positioning of samples, detectors or other instruments. They can be purchased having all or either X, Y, Z and rotary movement. The X and Y axes can have various travel lengths however they are typically less than 100 mm each. The Z axis (vertical) can have greater lengths but is limited as with the X and Y axes by the physical limitations of the vacuum bellows.

#### 11.4 Actuators

'Actuators', combine sliding and rotary feedthroughs with pneumatic, electro-pneumatic or mechanical actuators to do the work.

In the accelerator area, actuators can be found on Faraday Cups to insert the cup into the beam line on a bellows sealed sliding feedthrough and inside of the ANTARES pressure vessel to move the stripper tube in and out of the beam axis.

#### 11.5 Load locks

Load locks are not common in the accelerator area but are ideal for moving samples into a high vacuum space without significant loss of vacuum pressure. A sample or device is mounted onto the end of the load lock shaft. It is inserted into a tube connected to a chamber but isolated from the chamber vacuum by an in-line gate valve. The tube is sealed and then evacuated to a vacuum pressure similar to the chamber. Once the pressures equalise the gate valve is opened and the shaft pushed into the vacuum space. From this point the sample or device can be removed from the shaft by other remote means.

# **12. Internal Devices**

There are many devices that are commonly used inside of vacuum systems. Not all are represented in this section however similar reasons are use to assess new and unusually devices for use in vacuum.

#### 12.1 Lighting

Lighting is necessary in most sample measurement chambers where the operator must be able to see the sample in order to position it in front of the ion beam. In most cases a standard bare 12Vac halogen bulb is used with good results. Care must be taken to ensure the bulb can be dimmed as when running at maximum power the heat load can only be dissipated through the connecting wires. That is, there is little gas inside a vacuum to conduct head away from the bulb. Bright white LED's are also used with mixed success. They have less radial light output as compared with a bare halogen bulb. If the LED is setup correctly the light can be focussed towards the necessary target.

Preferably, wires must be stranded or solid wire with a loose Teflon sleeve but normal plastic coated hook up wire works in high vacuums. In some cases where the wires as not likely to come into contact with other components then they are used uninsulated. All wiring must be restrained so that it cannot move into the path of the ion beam. As a rule-of-thumb wires should be at least 20 mm from the beam.

A standard double BNC feedthrough can be used, that is, only the centre core connections are used. There are many off-the-shelf electrical feedthroughs available that will be more suited for this specific use.

#### 12.2 Heating

Providing power to a heating stage on a sample (target) stick requires a high current feedthrough. If a feedthrough isn't provided from the supplier of the heating stage then many types can be found from larger vacuum suppliers. Ensure the feedthrough selected is designed to carry the maximum current that can be supplied and not the maximum current likely to be used. Like in the lighting section above, care must be taken to ensure wires are insulated and away from the beam path.

Heating stages rely on heat dissipation through the support for the stage which is usually made from stainless steel, a relatively poor conductor. When opening devices for handling be sure they are cool to touch first.

#### 12.3 Cooling

Cooling stages come in 2 distinctly different types. The first is the Peltier cooled stage which is all electric. Powering the stage can be managed as for lighting and heating. The second type is a liquid nitrogen cooled system. It uses an insulated tube to carry LN2 through a feedthrough into the vacuum space. A typical design is where LN2 is pumped through a coil attached to a heavy copper braid. The braid is then connected to the cooling stage where heat is conducted away from the mounting stage. Thermal isolation of the cooling stage from the holder can be Teflon or sapphire standoffs or similar materials.

These cooling devices must only be switched on when the vacuum is at the working level otherwise condensation and possibly ice can form on their surface becoming a large outgassing problem. Likewise ensure the stage is close to room temperature before opening up the vacuum system to atmospheric pressure. Always vent with a dry gas.
# **13. Pumping**

### 13.1 General

It is not the intention of this document to describe the fundamental operation of particular vacuum pumps. Adequate information is available from manufacturer's catalogues. The following is information on what type of systems are used in the accelerator area and the reasons why.

Vacuum pumps don't suck. They act essentially as one way valves in a system where gas is flowing towards them. This is discussed in Chapter 3 of this document. There effectiveness is there ability to stop recoiling gas molecules returning into the vacuum space such as a beam line or vacuum chamber.

When considering what type of pump to use for a vacuum system the following points must be considered.

- What is the application?
- Will it have a relatively high gas load, that is;
- Will it be pumping a static gas load or changing gas load?
- Will there be a high outgassing rate?
- Are there known hidden/trapped voids to be pumped?
- It is a clean or dirty device? Is it contaminated with grease and oil?
- What pumping speed will be needed to maintain a high vacuum?
- What is the volume of the system? What is the conductance of the system? Will a small pump be just as effective as a large pump?
- What gases will be pumped?
- What type of pumps and how many are being used in the vicinity?
- Will the pumping speed need to be regulated such as for pumping near foils or delicate samples, detectors, etc?
- Cycling time whether it will be required to regularly pump from low vacuums
- Will the pumps operate reliably in their chosen application?
- Are they compatible with other pumps used on the accelerators? Are they cost effective?

Another consideration is vibration. Pump vibration can be significant leading to micrphonic interference in detectors and vibration of samples being measured. Vibration can be reduced by selecting pumps with less inherent vibration like turbomolecular pumps or the more fickle ion pumps. If a pump is a source of vibration it can be decoupled from the detector or measurement chamber by the use of a flexible bellows. An fine edge welded bellows is recommended.

### **13.2 High Vacuum Pumps**

See section 4.5, Pump Down characteristics for cryogenic and turbomolecular vacuum pumps.

#### <u>Cryopumps</u>

For ANTARES the main beam line pumps in use are cryopumps. The types of pumps trap (or immobilise) gas molecules. They are used because they have a long lifetime between major

services, can be regenerated without removing them from service, have a high pumping speed for most gases and can maintain high vacuums for long periods in a static vacuum application.

#### Turbomolecular pumps

Where a vacuum system is cycled such as at a measurement chamber, then turbo pumps are used. Turbo pumps are transfer type pumps meaning, gas is transferred from the vacuum space into the atmosphere. A turbo pump can routinely recover a vacuum from low vacuum after a measurement chamber has been roughed out without needing regeneration as for a cryopump.

Turbo pumps are also more effective at pumping lighter molecules such as helium and hydrogen as compared with cryopumps. Using turbo pumps near systems that use these gases will reduce the effect of loading on cryopumps.

Most new turbo pumps can be serviced in-house. Some models purchased require special balancing of the bearings after they had been changed. In this case they are sent back to the manufacturer at great cost. It is important that models chosen can be maintained in-house.

#### Other high vacuum pumps

There have been few applications where titanium sublimation or ion pumps have been used in the accelerator area. Most have been stand alone UHV systems or cryostats for specialised applications. The practicality of cleaning and regenerating these gas trapping type vacuum pumps on systems requiring relatively frequent cycling is prohibitive.

Getter materials once used widely in electronics in tubes are becoming more used within larger static volumes such as beam lines and associated equipment. They can be coated onto surfaces to assist in maintaining uniform high vacuums in spaces that have poor pumping speeds.

#### **13.3** Low Vacuum, Roughing or backing pumps

#### Oil free pumps

These pumps are categorised by either being an oil type pump or dry (oil free) pump. Oil free vacuum systems are of choice for accelerator systems now that the technology has been proven to be reliable and cost effective. The majority of roughing and backing pumps now in use are scroll pumps as well. Piston pumps have been trialled with little success due to the short life of the seals. Some applications have used diaphragm pumps and multistage roots pumps but scroll pumps remain the most cost effective and easiest to maintain option. The Edwards type scroll was chosen over other brands as this type of scroll pump is designed with the bearings mounted outside of the vacuum which in other brands often caused the bearings to fail from lubricant loss.

#### Oil vane pumps

Low Vacuum, Roughing or backing pumps are all the same type of pump. Oil vane type pumps were widely used in the accelerator area until 2006 when a systematic change began on the STAR accelerator to replace oil vane pumps with oil free scroll pumps. Some oil vane pumps remain in use where some back streaming oil vapour is not considered a problem. If an oil vane pump is to be used for roughing out purposes, it is high recommended that a liquid nitrogen cold trap is used to trap vapours. It is the ultimate aim to have oil free pumping on all accelerators in the future.

# 14. Monitoring

#### 14.1 Vacuum Gauges

The standard type of high vacuum gauge in use is the cold cathode penning type gauge.

- On ANTARES the Pfeiffer brand is in use in two versions; the standard cold cathode type and full range type which has an integrated Pirani gauge.
- On STAR the most widely used brand is Leybold.

Generally, vacuums are measured as an indication only for process controlling and establishing basic quality to ensure pressures are within levels that are accepted for "normal" operation. This varies depending on location and use of the vacuum system for example a measurement chamber is cycled often and affects surrounding vacuum systems. It will more than likely be a much high pressure than surrounding static systems.

In most cases, absolute vacuums are not required to be measured. However on some AMS beam lines where thin windows are used it is important to have accurate measurement of pressures when cycling pressures in the gas detectors. If accuracy is required it must be remembered that different gases ionise at different pressures and so pressures readings from the same volume will be different depending on the dominant background gas. Most gauges have characteristics related to Nitrogen (air).

#### 14.2 Gauge Controllers

On ANTARES the most widely used gauge controller is the Pfeiffer TPG300. This is an aging model and is being replaced in some key areas with the Pfeiffer Maxi-gauge which allows 6 heads to be connected at once.

The TPG300 has inputs for up to 4 heads whether Penning or Pirani. The limitation with the TPG300 is the electrical robustness when operated near the accelerator high voltage generator. Many failures have been attributed to high voltage transient surges. The TPG300 is now technologically aged and will ultimately be replaced outright. Various other models of TPG's are in use on ANTARES especially where single gauges are required. It is the TPG300 controllers on ANTARES that provides the interlocking capabilities.

The MAXI gauge is still being trialled as a replacement to the TPG300. A unit under test for many years at the HE end of the accelerator has already failed during an accelerator sparking event. Its real place may be away from the accelerator where the 6 heads allow consolidation of a whole beam line's vacuum monitoring system.

On STAR the common high vacuum gauge is the Leybold cold cathode type. It has been integrated into the STAR control system via cat 5 cable. They operate using a similar principle of measuring current flow across the plasma of the ionising gas. These gauges have been far more unreliable than the Pfeiffer equivalent. Unfortunately they are not interchangeable.

If venting with gas other than air or nitrogen be aware that the response on the Pirani gauges may show lower pressure than actual pressure.

### 14.3 Bourdon Gauges

Bourdon gauges are normally used for indicating that pressure is below atmospheric. They are not used on accelerator systems as they have poor accuracy in the range of interest i.e., below  $1 \times 10^{-3}$  Pa.

# Chapter 3



# **Overview of Basic Vacuum Technology**

The ANSTO accelerators consist of 140 metres of beam lines under vacuum. Supporting these beam lines are 60 individual high vacuum pumping stations. There are over 70 isolation zones along the beam lines to allow isolation of a zone without interrupting the adjacent zones. This facilitates isolations to repair leaking sections, routine maintenance and cycling of pressure in zones to change samples, etc.

The following information are the general tools used by technicians and engineers to design reliable and high performance vacuum systems for the accelerator facilities.

# 1. Vacuum Overview

### 1.1 What is vacuum?

A vacuum is defined as a diluted gas, or the corresponding state at which its pressure or density is lower than that of the ambient surrounding atmosphere.

Due to the Earth's gravity, atmospheric pressure varies with altitude. As a standard atmospheric pressure at sea level  $p_o = 101.325$  kPa. A Pascal is unit of vacuum relating to a force of newtons per metre squared.

An absolute vacuum of 0 kPa is not practically achievable so in real terms we can only approach zero for example on the ANSTO accelerators we have "operating" vacuums better than  $1 \times 10^{-4}$  Pa. In outer space the vacuum is less than  $10^{-12}$  Pa but not 0 Pa as there are some molecules of gas present which exert a very small partial pressure.

The pressure exerted from a vacuum lies between 0 to 100 kPa (see table below). In an ideal pumped vacuum system the pressure will continue to decease approaching 0 kPa. In accelerator systems the working ranges are medium to ultra high vacuums. In some special cases ion sources may operate in the low vacuum range but generally vacuum systems for the majority of accelerator systems are in the high vacuum range with some vacuum end stations close to ultra high vacuum.

#### 1.2 Units used

The SI unit  $Pa = N/m^2$  or kg·m<sup>-1</sup>·s<sup>-2</sup>, will be used where any vacuum quantity is referenced. In the accelerator area the vacuum units used are in Pascals (Pa) and although the vacuum pressures are below atmospheric pressure "negative" pressures are not represented as -kPa but of a magnitude which approaches absolute zero using scientific notation, e.g. 1 x 10<sup>-6</sup> Pa.

#### **1.3** Vacuum categories

Vacuums used in accelerator applications vary in magnitude from low vacuum to ultra high vacuum. These categories describe the pressure ranges which in the vacuum world become references for vacuum quality in a given system. Low vacuum is managed differently from high and ultra high vacuums due to the physical limits of the system designs. Generally, a vacuum system must be designed with the desired ultimate vacuum in mind to ensure the most appropriate materials, seals, pumps and vacuum management are used.

The table below shows each of the ranges based on the European system. This is the closest to that used for the ANSTO accelerators. The shaded section represents the working ranges used throughout the accelerator for example, ion sources may have low to high vacuums, beam lines may have very high vacuums and some endstation vacuum chambers may use high to very high vacuums. This document describes the design factors and particular management controls for achieving vacuums in the various ranges.

Range	Pressure (Pa)
Atmospheric pressure	1.013 x 10 <sup>5</sup>
Low vacuum	$1 \times 10^5$ to $3 \times 10^3$
Medium vacuum	$3 \times 10^3$ to $1 \times 10^{-1}$
High vacuum	$1 \times 10^{-1}$ to 1 x $10^{-4}$
Very high vacuum	$1 \ge 10^{-4}$ to $1 \ge 10^{-7}$
Ultra high vacuum (UHV)	$1 \ge 10^{-7}$ to $1 \ge 10^{-10}$
Extremely high vacuum	$< 1 \times 10^{-10}$
Outer Space	$< 1 \times 10^{-12}$
Perfect vacuum	0 Pa

Table 2. Reference pressure ranges (European scale).

A. Berman, Vacuum Engineering Calculations, Formulas, and Solved Exercises, Academic Press, Inc. (1992).

In the accelerator area:

Low Vacuum = atmospheric pressure to medium vacuum

High Vacuum = high vacuum to very high vacuum

Ultra high vacuum = all those equal to and higher than ultra high vacuum

# 2. Pressure Fundamentals

#### 2.1 General

To understand the reason why vacuum systems must be carefully engineered to achieve high vacuums consideration must be given to how gases act in a closed chamber.

Any gas enclosed within a volume (a chamber or pipe work) is uniformly distributed or moving towards uniform distribution as in the case of accelerator tubes connected to stripper gas enclosures. The individual gas particles are constantly moving back and forth at high-speed within the volume; upon striking the vessel wall, they exert a force F on surface A due to pulse transmission. The pressure p that is exerted on the wall is defined as:

$$p = F/A$$

If the gas is made up of different types of gases, each of these gases will exert a pressure that corresponds to its concentration; this is called <u>partial pressure</u>. The sum of all partial pressures equals the total pressure. Air is a good example of this: In addition to its main constituents of nitrogen, oxygen and water vapour, air also contains many trace gases, the sum of which equal  $\sim 101.3$  kPa, total pressure (atmospheric pressure). See table below.

In an accelerator system it would not be uncommon to have various carbon based compounds, sulphur hexafluoride and specific stripper gases present in trace amounts.

Gas	% by volume	Partial Pressure (Pa)			
Ar	0.92	934			
CO <sub>2</sub>	3.26 x 10 <sup>-2</sup>	33			
H <sub>2</sub>	4.9 x 10 <sup>-5</sup>	4.94 x 10-1			
He	5.16 x 10 <sup>-4</sup>	5.23 x 10-1			
Kr	1.1 x 10 <sup>-4</sup>	1.15 x 10-1			
$N_2$	77.17	78180			
Ne	1.8 x 10 <sup>-3</sup>	1.82			
O <sub>2</sub>	20.7	20970			
Xe	8.59 x 10 <sup>-6</sup>	8.7 x 10-3			
H <sub>2</sub> O	1.18	1200			
Other	remainder	remainder			
Total	100 %	101325 Pa			
Notes: H <sub>2</sub> O may vary depending on environment					
"Other" may include: $CH_4$ , $O_3$ , $N_2O$					

Table 3. Partial Pressures in AirThe Vacuum Technology Book – Pfeiffer Vacuum September 2008

#### 2.2 Ideal (General) gas equation

1 mole of any gas at STP occupies 22.414 litres. At a temperature 273.15 K (0 °C) and a pressure of 101,325 Pa (standard pressure) 1 mole of any gas contains 6.02 x  $10^{23}$  particles. This is referred to

as Avogadro's number. The mass of the gas thus enclosed is its molecular weight in grams. The ideal gas equation describes the state of a gas as a function of pressure, temperature and volume.

$$pV = nRT$$

Example:

For a chamber, Ø300 mm x 500 mm long, a volume of 0.035 m<sup>3</sup>, calculate the mass of gas in the chamber at a vacuum of 1 x  $10^{-6}$  Pa.

pV = nRT

n = pV/RT  $n = 1 \times 10^{-6} \times 0.035 / 8.31451 \times 293 = 1.43 \times 10^{-11} \text{ moles}$ Mass of remaining volume = moles x mass = 1.43 x 10<sup>-11</sup> x 29 (air) = 0.415 ng No. of molecules = moles x Avogadro's Number = 1.43 x 10<sup>-11</sup> x 6.02 x 10<sup>23</sup> = 8.6 x 10<sup>12</sup> Per cm<sup>3</sup> = 8.6 x 10<sup>12</sup> / 35000 cm<sup>3</sup> = 246 x 10<sup>6</sup> molecules per cm<sup>3</sup>

Where:

 $p = \text{pressure (Pa)}, V = \text{volume (m}^3), n = moles, R = \text{general gas constant} = 8.314510 \text{ kJ/(kmol K)}, T = \text{thermodynamic temperature (K)}, Avogadro's number = 6.02 x 10^{23}$ 

### **3.** Flow Fundamentals

#### **3.1** Mean free path $(\lambda)$

The mean free path is the mean path length that a molecule traverses between two successive impacts with other molecules. High vacuums provide a longer mean free path than low vacuums. In accelerator systems high vacuums are crucial as ions that collide with gas molecules in beam lines loose energy and are not transported to the point of measurement. In the accelerator tubes high mean free paths are crucial to ensure low energy loss in the ion beam and optimised ion beam transmission.

Gas (at 0°C and 1 atm)	$\lambda$ (nm)
Air	68
Ar	62.6
Cl <sub>2</sub>	27.4
СО	58.6
CO <sub>2</sub>	39
$H_2$	110.6
Не	173.6
Kr	36
$N_2$	58.8
Ne	124
<b>O</b> <sub>2</sub>	63.3
Xe	26

Table 4. Mean free Paths for various gases

Hirschfelder, Curtiss and Bird (1954) Molecular Theory of Gases and Liquids, Wiley, New York

Pressure (Pa)	Molecules / cm <sup>3</sup>	Molecules / m <sup>3</sup>	Mean free path
101325	$2.7 \times 10^{19}$	$2.7 \times 10^{25}$	68 nm
		1	

Table 5. Other characteristics of Air

#### 3.2 Mean Velocity (u) m/s

The residency time of various gases in a system (or the time taken to remove various gases) relates to their individual mean velocity. The following table shows mean velocities for some gases. It can be seen that very light gases travel significant faster than heavy gases. It should be noted that Hydrogen and Helium do not pump efficiently in turbo pumps or cryopumps but heavy gases generally do.

Gas	Molar Mass kg/mol	Mean Velocity m/s	Mach Number
Air	0.028966	463	1.4
Ar	0.039948	394	1.2
Cl <sub>2</sub>	0.0709	296	
CO	0.028011	471	
$CO_2$	0.04401	376	1.1
$H_2$	0.002016	1762	5.3
He	0.00402	1246	3.7
Kr	0.0838	272	
$N_2$	0.02801	471	1.4
Ne	0.020179	555	
O <sub>2</sub>	0.03199	441	
Xe	0.1313	217	
H <sub>2</sub> O	0.01802	587	1.8

Table 6. Mean Velocity for gases at 20°C

The Vacuum Technology Book – Pfeiffer Vacuum September 2008

$$u = \sqrt{\frac{8.R.T}{\pi.M}}$$

Where: *M* = Molar mass (kg/mol)

For example: Mean velocity of Argon @  $20^{\circ}$ C  $u = SQRT (8 \times 8.31451 \times 293 / 0.039948 \times 3.1416)$ u = 394 m/s

#### 3.3 Types of flow

The different flow phases need to be understood in order to select the appropriate pumping system for a particular application. It may be that in most cases a chamber or other piece of equipment is

pumped at the highest flow rate achievable with a given pump but it could be the case for example where a chamber has delicate films inside and gas movement past the foils has the potential to tear them apart. In this case a low steady flow rate is needed.

The flow phases in a vacuum system characterise the gas molecule movement related to pumping speed in a vacuum system. Factors that determine flow include pressure differentials, mean free paths, gas type, geometry, dimensions of the system and temperature. There are many different nomenclatures used to represent flow phases but 3 types in particular are used in the accelerator area, Turbulent, Laminar (both in Viscous phase), and Molecular. Less emphasis is placed on the intermediate or transitional flow as once this state is reached it is assumed that the system is pumping well. At atmospheric pressure up to about 100 Pa, the mean free path of the gas molecules is very small. Therefore, the gas flow is limited by the viscosity of the gas being pumped so the type of flow is called Viscous.

In an *ideal* system, if a roughing pump has a pumping speed of say 22 m<sup>3</sup>/hr and there is negligible flow resistance between the pump and the chamber then a chamber of  $\emptyset$ 300 mm ID x 500 long (a volume of 0.35 m<sup>3</sup>) at atmospheric pressure, will take approximately 1 minute to remove the bulk of the gas. During this time the flow remains in the [Viscous] Laminar and/or Turbulent phase.

Where a system has a leak, depending on the magnitude of the leak it is possible that the pumping will plateau at either the Turbulent, Laminar or Molecular flow phase. With outgassing systems the less referred to transitional state may be reached and with systems designed to reach ultra high vacuums outgassing may be occurring well into the molecular flow state.

Pumping	Flow	Phase	Description	Type of vacuum	Flow rate Pa-l/sec
Pumping begins		Turbulent Flow	Initial flow experienced at start of pumping or in a low vacuum environment where the flow rate is high. Chaotic movement of molecules. Eddies and other non-parallel flows occur.	Low Vacuum	>1.01
	Viscous	Laminar Flow	Initial flow experienced at start of pumping or in a low vacuum environment where the flow rate is low. Gas molecules bouncing off other gas molecules (gas-gas) in the vacuum system. Short mean free path. Molecules streaming from system.	Medium Vacuum	10.1 to 1.01 x 10 <sup>-4</sup>
	Transiti (Kn	ional Flow udsen)	Intermediate flow between laminar and molecular. Flow is dominated by both gas-gas and gas-wall collisions.	Medium to High Vacuum	1.01 x 10 <sup>-2</sup> to 1.01 x 10 <sup>-5</sup>
Base vacuum reached	Molec	ular Flow	Remaining gas molecules bounce off the walls (gas-wall) of the vacuum system and not each other. Long mean free paths. Flow rate very small.	High to Ultra-High Vacuum	<1.01 x 10 <sup>-5</sup>

Table 7. Vacuum flow phases

By evaluating the Knudsen (Kn) and Reynolds (Re) number, one can predict the various flow phases. These phases can be seen in the picture below which also shows how the gas molecules behave in the different flow phases moving through the opening d.

#### 3.4 Knudsen Number (Kn)

The Knudsen number is a dimensionless number defined as the ratio of the molecular mean free path length to a representative physical length scale. The Knudsen number in a viscous flow phase < 0.01 and molecule-molecule collisions dominate gas behaviour which behaves as a fluid. In molecular flow Kn > 1 and molecule-surface collisions dominate.

$$Kn = \frac{\lambda}{d}$$

 $\lambda$  = Mean free path

d = diameter of the opening that gas will pass through

#### 3.5 Reynolds Number (Re)

Reynolds number indicates whether the flow of a gas is absolutely steady (laminar flow) or on average steady but with small, unsteady changes (turbulent flow). The Reynolds number, Re, has no dimensions and is defined as the size of the flow.

Osborne Reynolds demonstrated in 1883 that the change from laminar to turbulent flow in a pipe occurs when the value of the Reynolds number exceeds 2,100. The exact value of Re for which the flow changes from laminar to turbulent depends on the geometry of the component, its surface roughness and other experimental factors. During evacuation of a vessel, turbulent flow normally occurs only for a short period of time at the beginning. It has been found to be approximately proportional to the root of the pressure gradient.

$$\operatorname{Re} = \left(\frac{\rho}{\eta}\right) d.v$$

 $\rho$  = gas density (kg/m<sup>3</sup>)  $\eta$  = viscosity (Pa.s) (Pascal-second) v = flow velocity (m/s) d = tube diameter (m)

Gas	Formula	Molecular weight	Gas Density [2] kg/m <sup>3</sup>	Viscosity η Pa.s
Air [1]	$N_2 + O_2$	29	1.1839	1.79 x 10 <sup>-5</sup>
Ammonia	NH <sub>3</sub>	17.031	0.7449	1.01 x 10 <sup>-5</sup>
Argon	Ar	39.948	1.723	2.24 x 10 <sup>-5</sup>
Carbon Dioxide	$CO_2$	44.01	1.9105	1.51 x 10 <sup>-5</sup>
Carbon Monoxide	CO	28.01	1.2082	1.74 x 10 <sup>-5</sup>
Chlorine	Cl <sub>2</sub>	70.906	3.1124	1.34 x 10 <sup>-5</sup>
Helium	He	4.02	0.171	1.99 x 10 <sup>-5</sup>
Hydrogen	$H_2$	2.016	0.0868	8.80 x 10 <sup>-5</sup>
Hydrochloric Acid	HCl	36.5	1.5844	1.46 x 10 <sup>-5</sup>
Hydrogen Sulphide	$H_2S$	34.076	1.4876	1.26 x 10 <sup>-5</sup>
iso-Butane	$C_4H_{10}$	58.12	2.3758	7.49 x 10 <sup>-5</sup>
Methane	$CH_4$	16.043	0.6556	1.11 x 10 <sup>-5</sup>
Nitrogen	$N_2$	28.02	1.2088	1.76 x 10 <sup>-5</sup>
Oxygen	$O_2$	32	1.381	2.02 x 10 <sup>-5</sup>
Propane	C <sub>3</sub> H <sub>8</sub>	44.09	1.8024	8.26 x 10 <sup>-5</sup>
Sulphur Hexafluoride	SF <sub>6</sub>	146.5	6.27	$1.53 \times 10^{-5}$

Table 8. Gas density and viscosities

Unless otherwise indicated gases referenced from McGraw Hill Chemical Properties Handbook 1 atm 25°C. [1] Air STD Atmosphere at Sea Level: 1 atm 15°C [2] Gas Density Values Interpolated From 15°C Data using Charles Law

http://pipeng.com/index.php/gsts/itdmodflup002a/itddaflup00201

#### 3.6 Viscous Flow

### Low vacuum, p = 100000 - 100 Pa, where $\lambda \ll d$

What characterises viscous flows, is frequent contact between gas molecules, yet less frequent contact with the walls of the vessel. The mean free path of the gas molecules is therefore very small and the gas flow is limited by the viscosity of the gas being pumped (Viscous Flow). In this case, the mean free path of the gas molecules is significantly shorter than the dimensions *d* of the vacuum equipment. For both Laminar and Turbulent Flow, Kn < 0.01. In addition, the term viscous flow is used if the product of pressure *p* and diameter *d* of the components through which gas is flowing is p.d  $\geq$  60 Pa.cm for air. Whether the flow is in the Laminar or Turbulent flows phases can be calculated using the Reynolds Number formula above.

#### 3.7 Knudsen flow

#### Medium vacuum, p = 100 - 0.1 Pa with $\lambda \le d$

If the Knudsen number is between 0.01 and 0.5, this is termed Knudsen flow. Because many process pressures are in the medium vacuum range, this type of flow occurs with corresponding frequency. Since this is a transitional flow, this range is transited relatively quickly when pumping down vacuum chambers and hence not usually considered in accelerator systems. This means that the influence of this conductivity on pump-down times is correspondingly low. A simple approximation for the Knudsen range can be obtained by adding the laminar and molecular conductivities. The

table in "Conductivities" below shows the conductivities of round, one meter long tubes of differing diameters in all three flow ranges.

#### 3.8 Molecular flow High vacuum, $(p = 0.1 - 10^{-5} \text{ kPa})$ , where $\lambda > d$ and in UHV $(p < 10^{-5} \text{ kPa})$ , with $\lambda >> d$

At Knudsen numbers of Kn > 0.5 molecule-molecule collisions virtually no longer occur. What prevails is molecular flow. In this case, the product of pressure p and component diameter d is p x d  $\leq$  1.3 Pa.cm.



Fig 5. Molecular paths during different flow phases Re reference: http://www.engineersedge.com/fluid\_flow/pressure\_drop/pressure\_drop.htm

#### **3.9 Pump throughput qpV**

The concept of pump throughput is of major significance in practice and should not be confused with the pumping speed! The pump throughput is the quantity of gas moved by the pump over a unit of time, expressed in Pa.1.s<sup>-1</sup>. Conversely, the pumping speed is the capacity of the pump to remove a volume of gas within a specific unit of time, measured in  $m^3/h$  or 1/s.

The throughput value is important in determining the size of the backing pump in relationship to the size of a high vacuum pump with which it is connected in series in order to ensure that the backing pump will be able to take away the gas moved by the high vacuum pump.

The pumping capacity (throughput) for a pump is equal either to the mass (m) flow through the pump intake port in a specified time:

$$q_m = \frac{m}{t}$$

Or to the pV (quantity of gas) flow through the pump's intake port:

$$q_{pV} = \frac{pV}{t}$$

It is normally specified in Pa.l.s<sup>-1</sup>. Here p is the pressure on the intake side of the pump. If p and V are constant at the intake side of the pump, the throughput of this pump can be expressed with the simple equation:

 $q_{pV} = p.S$ 

Where:

S = pumping speed of the pump at intake pressure of p.

#### 3.10 Speed of vacuum pump S

The speed of a vacuum pump is defined as

$$S = \frac{q_{pV}}{p}$$

p = Pressure at the pump inlet

However, pumps are usually connected to vacuum chambers via hoses resulting in a lower effective pumping speed  $S_{eff}$  at the chamber. If  $p_1$  is the pressure at the pump and  $p_2$  is the pressure in the vacuum chamber then:

$$q_{pV} = S.p_1 = S_{eff}.p_2$$

for continuous flow. Combining this with  $q_{pV} = C(p_1 - p_2)$  (see Conductance below) results in an expression for S<sub>eff</sub>

$$S_{eff} = \frac{S.C}{S+C}$$

#### **3.11** Conductance C

In a vacuum system, the volume that is pumped by a vacuum pump exhibits a level of flow resistance (Z) due to chamber and pipe sizes and their geometries, and the various sized orifices and other in-stream devices. The terminology used to describe this effect is the reciprocal of flow resistance which is *conductance* (C) or the systems ability to conduct gas.

Conductance is expressed in either l/s or sometimes  $m^3/h$  and is usually for steady, continuous flow through one or more components in a vacuum system. It is affected by the geometry of the piping element and relative to the flow phase and capacity of the pump used. In the high and ultrahigh vacuum ranges (molecular flows), C is a constant which is independent of pressure. In the rough and medium-high vacuums (turbulent and laminar) it is, by contrast, dependent on pressure. As a consequence, the calculation of C for the piping (beam line) elements must be carried out separately for the individual pressure ranges.

Flow resistance (sec/litre) 
$$Z = \frac{P_1 - P_2}{q_{pV}}$$

Conductance (litre/sec) therefore

 $C = \frac{1}{Z} = \frac{q_{PV}}{P_1 - P_2} \qquad (continued over)$ 

$$q_{pV} = C(p_1 - p_2)$$

Where:  $q_{pV}$  = Throughput flow rate p = Pressure (Pa)

At the molecular level, the conductivity of a system is reduced due to the friction of both the walls of the system and other molecules. As discussed above in "Types of Flow", these two affects occur at different flow phases. The total effect of conductance is the sum of the various conductance elements. In the case where a valve, a trap and an elbow in series each having a different conductance, the sum is:

$$\frac{1}{C_{total}} = \frac{1}{C_{valve}} + \frac{1}{C_{trap}} + \frac{1}{C_{elbow}}$$

If the components with different conductances are connected in the flow path in parallel, for example, 2 different diameter tubes connecting a chamber to a pump then the equation will be:

$$C_{Total} = C_{tube1} + C_{tube2}$$

For the design of vacuum systems used on the accelerators conductance is mostly calculated for systems in molecular flow.



*Fig 6. Diagram for estimating pipe conductance Pupp/Hartmann, Vakuumtechnik, Grundlagen und Anwendugen, Hanser Verlag* 

# 4. Limitations

The most fundamental problems with vacuum systems are leaks and outgassing. Leaks can be categorised as either real leaks where gas enters the vacuum system from outside of the vessel or leaks form trapped voids which is in effect an internal leak. Trapped voids can contain gas that will continue to outgas for long periods reducing the ultimate vacuum that can be reached in a vessel. Real leaks can be found with the aid of a helium leak detector whereas internal leaks from trapped voids may never be found.

Outgassing can be minimised through careful selection of materials used within the vacuum space. The appendices have helpful tables to estimate outgassing rates. Outgassing may be in the form of desorption, diffusion or vaporisation. Permeation is not outgassing as the source of gas originates from outside of the vacuum space.

Examples commonly used substances in the accelerator systems that outgas,:

- Teflon, PVC, Ertalyte
- Viton, neoprene
- Copper, aluminium, stainless steel, brass, tantalum, rubidium, caesium, lithium, zinc
- Vacuum pump oil, vacuum greases, vacuum epoxies

These are just a few but it demonstrates that all materials will outgas at some point when the temperature and vacuum pressure reach their individual vapour pressure.

Neither of the problems is resolved by increasing the pumping speed (capacity) of the vacuum pump. There will be a point where the rate of gas entering the system will equalise with the pumping speed of the pump. The rate may vary depending on the vapour pressure of the substance outgassing and the temperature of that substance.



Fig 7. Limitation of Pumping From: http://www.mse.ncsu.edu/WideBandgaps/classes/MSE%20751/Handouts/HO02\_Leakage.pdf



Fig 8. Unwanted gas source wheel

### 4.1 Contamination

Vacuum chambers must be clean in order to reach the desired pressure as quickly as possible when they are pumped down. Typical contaminants include oil and grease on screws and seals, process reaction products or condensed vapours, particularly water that is adsorbed on the walls of the vessel. Consequently, it is necessary to ensure that the components are clean when installing vacuum equipment. All components attached in the vacuum chamber must be clean and grease-free. All seals must also be installed dry. If high or ultra high vacuum is to be generated, clean gloves must be worn during the assembly process.

Examples of common contaminants in accelerator vacuum systems

- Rotary pump oil
- Water
- Plasticisers from various plastics
- General airborne dust
- Machining oil
- Residual gases from stripper gases, venting gases, ion source gases
- Sample breakdown

### 4.2 Condensation and vaporisation

All substances can occur in a liquid, solid or gaseous state. Their aggregate status is a function of pressure and temperature. Liquids are transformed into their gaseous state through vaporisation, solids through sublimation. The separation of liquids or solids out of the gaseous phase is termed condensation. Since normal room air contains approximately 10g of water vapour per m<sup>3</sup>, condensed water vapour is always present on all surfaces.

Adsorption on surfaces is especially pronounced due to the strong polarity of the water molecules. Natural fibres, in particular, such as paper, contain large quantities of water that escape during drying processes under vacuum. Cooled condensers are used to separate the water vapour in this connection. Even some metals (Cd, Zn, Mg) can vaporise in noticeable quantity at temperatures of several 100 °C. Consequently, use of these metals is avoided in plant construction.

#### 4.3 Desorption

In addition to water, other substances (oil) can be adsorbed on surfaces. Substances can also diffuse out of the metal walls, which can be evidenced in the residual gas. In the case of particularly rigorous requirements, stainless steel vessels can be baked out under vacuum, thus driving the majority of the volatile components out of the metal walls.

Gas molecules, (primarily water) are bound to the interior surfaces of the vacuum chamber through adsorption and absorption, and gradually desorb again under vacuum. The desorption rate of the metal and glass surfaces in the vacuum system produces a gas yield that is a function of time, however. A good approximation can be obtained by assuming that after a given point in time  $t > t_0$  to the reduction will occur on a linear basis over time.  $t_0$  is typically assumed to be one hour.



Fig 9. Desorption curves



Fig 10. Adsorption Curves From: http://www.mse.ncsu.edu/WideBandgaps/classes/MSE%20751/Handouts/HO02\_Leakage.pdf

#### 4.4 Diffusion with desorption

At operation below  $10^{-4}$  kPa, desorption of plastic surfaces, particularly the seals, assumes greater significance. Plastics mainly give off the gases that are dissolved in these plastics, which first must diffuse on the surface.

Following extended pump downtimes, desorption from plastics can therefore dominate the metal surfaces. Although the surface areas of the seals are relatively small; the decrease in desorption rate over time occurs more slowly in the case of metal surfaces. As an approximation it can be assumed that the reduction over time will occur at the square root of the time.

The gas produced from plastic surfaces can thus be described as:

Desorption from plastic material  $Q_{diff} = q_{diff} \cdot A \sqrt{t_o/t}$ 

Where Ad denotes the surface area of the plastics in the vacuum chamber and  $q_{diff}$  denotes the surface area-specific desorption rate for the respective plastic. At even lower pressures, similar effects also occur with metals, from which hydrogen and carbon escape in the form of CO and CO<sub>2</sub> and can be seen in the residual gas spectrum.

#### 4.5 **Permeation and leaks**

For a gas passing through small holes in a thin wall in the Knudsen Flow regime, the number of molecules that pass through a hole is proportional to the pressure of the gas and inversely proportional to its molecular weight.

To Convert to Leakage	Multiply Helium Leak Rate by:		
Rate of:	Laminar Flow	<b>Molecular Flow</b>	
Argon	0.88	0.316	
Air	1.08	0.374	
Nitrogen	1.12	0.374	
Water vapour	2.09	0.469	
Hydrogen	2.23	1.410	

Table 9. Conversation table for leak rates

Seals, and even metal walls, can be penetrated by small gas molecules, such as helium, through diffusion. Since this process is not a function of time, it results in a sustained increase in the desired ultimate pressure. The permeation gas flow is proportional to the pressure gradient  $p_0 / d$  (d = wall thickness,  $p_0 =$  atmospheric pressure = ambient pressure) and to the permeation constants for the various materials  $k_{perm}$ .

Permeation

 $k_{perm}$ .  $Q_{perm} = k_{perm}$ .  $A \cdot p_0/d$ 

Permeation first manifests itself at pressures below  $10^{-6}$  kPa.  $Q_1$ , denotes the leakage rate, i.e. a gas flow that enters the vacuum system through leaks at a volume of V. The leakage rate is defined as the pressure rise  $\Delta p$  over time  $\Delta t$ :

Leakage rate  $Q1 = (\Delta p \cdot V)/\Delta t$ 

If a vessel is continuously pumped out at a volume flow rate S, an equilibrium pressure  $p_{gl}$  will be produced. Throughput is equal to the leakage rate  $Q_l = S \cdot p_{gl}$ . A system is considered to be

adequately tight if the equilibrium pressure  $p_{gl}$  is approximately 10 % of the working pressure. If, for example, a working pressure of  $10^{-4}$  kPa is attained and the vacuum pump that is being used has a pumping speed of 100 I/s, the leakage rate should not be more than  $10^{-3}$  kPa I/s. This corresponds to a leak of approximately 20.20  $\mu$ m<sup>2</sup> in size. Leakage rates  $Q_l$  of less than  $10^{-6}$  kPa I/s can usually be easily attained in clean stainless steel vessels. The ultimate pressure achievable after a given period of time *t* primarily depends upon all of the effects described above and upon the pumping speed of the vacuum pump. The prerequisite is naturally that the ultimate pressure will be high relative to the base pressure of the vacuum pump.



Fig 11. Permeation of Different Gases From: http://www.mse.ncsu.edu/WideBandgaps/classes/MSE%20751/Handouts/HO02\_Leakage.pdf

#### 4.6 Leaks

The source of leaks is as wide and varied as can be imagined. There is no such thing as a common leak however there are several types generally seen. The following lists types in rough order of prevalence:

- Dirty seals elastomer type seals with traces of dust, dirt and fibres on the surface
- Jarred seals ill fitted flanges where the seal lays across the sealing surface
- Vacuum pumping tubes where a fitting has been fitted without care and the pump cannot reach its full capability
- Distortion over tightened or misaligned flanges and fittings that "lift" seals off the sealing surface
- Stress cracks usually in places around flanges and fittings that have been incorrectly tightened
- Pressure dependant mostly related to accelerator tubes that develop leaky seals allowing insulation gas to enter when the pressure is greater than atmospheric pressure.

#### 4.7 Bake-out

The following prerequisites must be satisfied in order to achieve lower pressures ( $<10^{-6}$  kPa):

- The base pressure of the vacuum pump should be a factor of 10 lower than the required ultimate pressure
- Stainless steel vacuum recipients and components must be used
- Metallic seals (CF flange connections) are required
- Leaks must be avoided and eliminated prior to activating the heater (use helium leak detectors!)
- Clean work is a must, i.e. all parts must be thoroughly cleaned and must be installed with grease-free gloves
- Pump and equipment must be baked out

Bake-out significantly increases desorption and diffusion rates, and this produces significantly shorter pumping times. Bake-out temperatures of up to 300°C are used. The instructions of the pump manufacturers relating to maximum bake-out temperatures and maximum permissible radiation levels in the pump flange must be observed.

Following installation the equipment is switched on, and after reaching a pressure of  $P < 10^{-3}$  kPa the heater is then switched on. During the heating process, all gauge heads must be operated and degassed at intervals of 10 hours. In the case of stainless steel vessels and the use of metallic seals, bake-out temperatures of 120°C and heating times of approximately 48 hours are sufficient for advancing into the pressure range of  $10^{-8}$  kPa. Bake-out should be continued until 100 times the expected ultimate pressure is attained. The heaters for the pump and vacuum chamber are then switched off. After cool-down, the desired ultimate pressure will probably be achieved. In connection with pressures  $P < 5 \times 10^{-8}$  kPa and large interior surface areas, it will be advantageous to use a titanium sublimation pump that pumps the hydrogen escaping from the metals at a high volume flow rate.

#### 4.8 Residual gas spectrum

When leaks have been solved in a vacuum system and poor vacuum persists than a residual gas analysis (RGA) measurement should be made to determine the composition of the gas load. This will give vital information on where the vacuum problem may be. Interpreting the information will be trial and error as users will have to think about all possible sources of gases (and outgassing) that may be present. Also relative ratios of gases should be considered to make sense of the measurement. Care should be taken especially if making a measurement near a source such as a cryopump. Some gases that have been trapped on the cryopump may be liberating from the cold surface and will make up a part of the gas spectrum. Other places to take care are near the accelerator tubes and ion sources.

If developing an ultra high vacuum system it is important to know all sources of gas otherwise moving lower than  $1 \times 10^{-6}$  Pa will be an unnecessary challenge. For all other systems it is nice to know but not essential unless residual gases affect ion beam measurements. The dominate gases (and their masses) that may be seen include:

Gas	Mass	Gas	Mass
Water	18	Carbon Monoxide	28
fragment HO	17	Carbon Dioxide	44
Nitrogen	28	Argon	40
Oxygen	32	Sulphur Hexafluoride	146
Hydrogen	2	Helium	4

Table 10. Table of masses for common "in vacuum" gases

# **Appendix 1 – Structural Calculations for Scientific Vacuum Vessel Design**

Calculations for Vacuum Vessel wall and end plate thickness

The following formulae are from the Vacuum Society of Australia (VSA) training course notes which have been in circulation since the early 1980's. VSA continues to support these calculations for training vacuum technologists around Australia. The calculations provide conservative estimates of vacuum vessel wall thicknesses as compared with formulas used in ASME Section VIII – Division 1 UG-28 (Thickness of Shells and Tubes under External Pressure) and associated documents, which have been developed for larger industrial type vacuum vessels.

#### Cylindrical Vessel – Stainless Steel @ 20°C

Thickness of plate in a given diameter (h):

$$\frac{D}{h} \le 105, \ \frac{h}{D} \ge \frac{1}{105}$$

Where: D = diameterh = thickness

Maximum length of vessel for a given diameter (L<sub>c</sub>):

$$\frac{L_c}{D} \le 11.5$$

Where: D = diameter $L_c = \text{length}$ 

#### **End Plates – Stainless Steel**

Thickness of plate in a given diameter (h<sub>1</sub>):

$$\frac{D_1}{h_1} \le 89$$
 ,  $\frac{h_1}{D_1} \ge \frac{1}{89}$ 

Where:  $D_1$  = diameter  $h_1$  = thickness

Minimum thickness for given deflection at centre:

 $\frac{h_1}{\delta} \ge 3$ 

Where:  $\delta$  = deflection  $h_1$  = thickness For Hemispherical End (h<sub>2</sub>):

$$\frac{R}{h_2} \le 830 \text{ requires } \frac{h_2}{R} \ge \frac{1}{830}$$

Where: R =radius  $h_2 =$  thickness

**Example 1:** To construct Stainless Steel vacuum chamber at  $20^{\circ}$  C as shown – Length L = 50 cm = 500 mm, D = 40 cm = 400 mm, Maximum allowable deflection at centre  $\delta = 1$  mm. Find  $h, h_1, h_2$  to satisfy vacuum chamber strength requirements.

#### For Cylinder

1. 
$$\frac{D}{h} \le 105$$
,  $\frac{h}{D} \ge \frac{1}{105}$   $h \ge 400/105$ ,  $h \ge 3.8 \text{ mm}$   
2.  $\frac{L_c}{D} \le 11.5$ ,  $L_c \le 11.5 \times 400$   $L_c \le 4600 \text{ mm}$  with  $L = 500 \text{ mm} \checkmark$ 

#### **For End Plates**

1. 
$$\frac{D_1}{h_1} \le 89$$
,  $\frac{h_1}{D_1} \ge \frac{1}{89}$   $h_1 \ge 396.2/89$ ,  $h_1 \ge 4.45 \text{ mm}$   
2.  $\frac{h_1}{\delta} \ge 3$ , with  $\delta = 1 \text{ mm}$  then require  $h_1 \ge 3 \text{ mm}$ , however  $h_1 = 4.45 \text{ mm} \checkmark$ 

#### For Hemispherical End

1. 
$$\frac{R}{h_2} \le 830$$
 requires  $\frac{h_2}{R} \ge \frac{1}{830}$   $h_2 \ge 198.1/830 \ge 0.24$ mm

Dimension of cylindrical, planar and hemispherical parts of vacuum enclosures

	At	Cylin	nders	End I	Plates	Hemispherical
Material	Temp (°C)	D/h	$L_c/D$	$D_1/h_1$	$h_l/\delta$	<i>R/h</i> <sub>2</sub>
Copper	20	84	10	52	15	600
Copper	500	58	8.5	-	-	-
Nickel	20	100	11	73	8	780
Nickel	500	90	10.5	-	-	-
Aluminium Alloy	20	70	9	37	57	470
Aluminium Alloy	500	62	8.7	-	-	-
Stainless Steel (304)	20	105	11.5	89	3	830
Stainless Steel (304)	500	89	10.5	-	-	-
Glass (hard)	20	70	9	16	117	470
Neoprene	20	2.5	1.7	10	0.2	30
Teflon	20	12	3.8	14	9	-
PVC (Tygon)	-	3.7	2.1	-	-	-
Perspex	-	_	-	30	-	-
Mica	-	_	-	58	15	-

Factor tables for thickness calculations of common materials used in high vacuum systems

 $\delta$  = maximum permissible deflection at centre of plate L<sub>c</sub> = critical length for cylinder = 1.11D(D/h)1/2

**Example 2:** Using the ASME Boiler and Pressure Vessel Code (the Code) Section VIII, Division 1 for the safe design pressures for a vacuum vessel is used to calculate the maximum external pressure acceptable for a given wall thickness. The following example uses the same vessel dimensions as in Example 1, with a wall thickness of 3.8 mm as calculated using the VSA method.

The calculations reference the Vacuum Vessel Cylindrical Shell Thickness using paragraphs UG-28, UG-27 of ASME Boiler and Pressure Vessel Code.

Vessel shell thickness t = 3.8 mm  $D_o = 400$  mm (outer diameter of shell)  $D_o/t = 105.26$  Since this ratio is greater than 10, follow UG-28(c)(1) L = 500 mm (length of stiffened shell)  $L/D_o = 1.25$ E = 28 x 10<sup>6</sup> (modulus of elasticity for 304 stainless steel)

From Table G in Sec II, Subpart 3 to determine Factor A

A = 1 x 10<sup>-3</sup> (Factor A, from Table G of Sec. II, Subpart 3)  $P_{ext} = \frac{2AE}{3(\frac{D_o}{t})}$ 

 $P_ext = 177.33$  psi (max allowable working external pressure for given *t*) = 1222 kPa

A wall thickness of t = 3.8 mm results in a maximum allowable working external pressure of P\_ext = 325 kPa, which is greater than the external pressure that the vacuum vessel will see.

**Example 3:** For comparison, calculate the maximum external pressure as in Example 2 but with a wall thickness of 2.5 mm.

 $D_{o} = 400 \text{ mm}$ t = 2.5  $D_{o}/t = 160$ L = 500 L/D\_{o} = 1.25 E = 28 x 10<sup>6</sup> (modulus of elasticity for 304 stainless steel)

From Table G in Sec II, Subpart 3 to determine Factor A

A =  $5.3 \times 10^{-4}$  (Factor A, from Table G of Sec. II, Subpart 3)

$$P\_ext = \frac{2AE}{3(\frac{D_o}{t})}$$

P\_ext = 61 psi (max allowable working external pressure for given t of 2.5 mm) = 420 kPa

Therefore a wall thickness of 2.5 mm has a P\_ext of 420 kPa.

**Example 4:** This is a worked example for a large vacuum vessel from ASME Boiler and Pressure Vessel Code (the Code) Section VIII, Division 1, paragraph UG-28. It is in imperial units.

Calculate the maximum allowed external working pressure following UG-28. The minimum required thickness takes into account the support of the stiffening rings. Note that the actual vessel shell thickness t = 0.375-inch

 $D_o = 38.0$  inch (965 mm) (outer diameter of shell) t = 0.261 inch (6.63 mm) (minimum required thickness of shell for external pressure of 14.5-psia)  $D_o/t = 145.594$  Since this ratio is greater than 10, follow UG-28(c)(1) L 235.7 inch (6057.5 mm) (length of stiffened shell)  $L/D_o = 6.203$ E =29x10<sup>6</sup> psi (modulus of elasticity for SA-516 Gr 60 carbon steel)

From Table G in Sec II, Subpart 3 to determine Factor A A =  $1.108 \times 10^{-4}$  (Factor A, from Table G of Sec. II, Subpart 3)

$$P\_ext = \frac{2AE}{3(\frac{D_o}{t})}$$

P\_ext = 14.709 psi (maximum allowable working external pressure for given t) = 101.35 kPa

A wall thickness of t = 0.261 inch results in a maximum allowable working external pressure of P\_ext = 14.7 psia, which is greater than the external pressure that the vacuum vessel will see. Since the actual wall thickness is 0.375 inch (9.5 mm), the vessel design is adequate for the working external pressure.



Factor A table for Stainless Steels

# **Appendix 2 – Low Outgassing Specialist Non Metal Materials**

#### • Celazole® PBI (PolyBenzImidazole) http://www.boedeker.com/celazo\_p.htm

Celazole® is the highest temperature-capable plastic available. However, it is very brittle (almost ceramic-like) and quite difficult to machine. That said, is it frequently used for bushings, bearings, rollers, and spacers in extreme environments. Its outgassing values are listed as 2.50% TML, 0.00% CVCM, 0.40% WVR.

#### • Vespel® (Polyimide)

http://www2.dupont.com/Vespel/en\_US/assets/downloads/vespel\_gen/E61500.pdf

DuPont Vespel® SP-1 is one of the most-used high-temperature plastic materials used in applications where high-purity and electrical properties are needed. Vespel is frequently used in ultra-clean semiconductor and chemical applications. It is also one of the most expensive materials sold, but is flight-approved for NASA, USAF and other aerospace agencies. Its NASA outgassing values are listed as 1.09% TML, 0.00% CVCM, 0.40% WVR.

#### • Duratron® XP (Polyimide)

http://www.portplastics.com/download/pdf/plastics/highPerformance/highPerformance26.pdf

Duratron® XP is the first real alternative to Vespel ... it was developed specifically to replace Vespel in extreme applications at a slightly lower price. It contains less than 1% metallic impurities as measured using the ICP-MS test standard. Duratron XP is ideal for use in high-energy gas plasma etch and strip processes. Outgassing values for Duratron XP are 0.75% TML, 0.00% CVCM, 0.49% WVR.

#### • Torlon® 4203 unfilled PAI (PolyAmide-Imide) http://www.boedeker.com/torlon-4203.htm

Unfilled Torlon® 4203 has high dielectric properties and low thermal expansion, and is much less expensive than some advanced polymers. Torlon 4203 is typically used for insulators, spacers, and mechanical parts up to 520°F. Its outgassing values are listed as 1.85% TML, 0.00% CVCM and 0.49% WVR.

#### • Torlon® 5530 glass-filled PAI (PolyAmide-Imide) http://www.boedeker.com/torlon-5530.htm

Torlon 5530 (30% glass-filled) is typically used for applications where dimensional stability over a wide temperature range is needed, as with temperature test sockets, nests, and fixtures. Its outgassing values are listed as 0.58% TML, 0.00% CVCM (% WVR is not shown). NOTE: Torlon's moisture absorption is a bit high, so critical dimensional stability can be an issue.

#### • Semitron® ESd 500HR (filled PTFE)

http://www.portplastics.com/download/pdf/plastics/staticControl/staticControl18.pdf

Semitron® ESd 500HR is antistatic/conductive PTFE. This material is relatively clean, readily machinable, dissipates static electricity reliably ... as a result it is used in test handling equipment, fixtures, and other applications where static generation may cause failures and/or errors in production environments. This material has low outgassing values of 0.04% TML, 0.00% CVCM and 0.01 % WVR. PTFE has good mechanical properties up to approximately 500°F.

### • Neoflon® PCTFE (PolyChloroTetraFluoroEthylene)

http://www.aftonplastics.com/materials/pdfs/neoflan\_pctfe.pdf

PCTFE exhibits high chemical resistance, low and high temperature capability, resistance to most chemicals (including strong acids and bases), low friction, electrical and thermal insulation, and "slipperiness". PCTFE has the lowest outgassing values of any thermoplastic material we sell ... 0.01% TML, 0.00% CVCM, 0.00% WVR.

• **PEEK (PolyEtherEtherKetone)** http://www.dotmar.com.au/ketron-peek-1000/ketron-peek-1000-polyetheretherketone.html

PEEK is pure, easily machinable, chemically resistant, stable, and also has relatively low outgassing values (0.31% TML, 0.00% CVCM, 0.06% WVR). PEEK has good mechanical properties, but will not take temperatures over  $350^{\circ}$ F, so it may not have the mechanical or thermal performance needed.

• **Techtron® PPS (PolyPhenylene Sulfide)** http://www.dotmar.com.au/techtron-hpv-pps/techtron-hpv-pps.html

Techtron® PPS is easily machined to close tolerance, has excellent mechanical, thermal and chemical stability and has one of the lowest outgassing values of any thermoplastic material we offer (0.04% TML, 0.00% CVCM ... % WVR is not shown). Techtron PPS is generally a bit less expensive than PEEK or Torlon, but again, will not take as high temperatures.

• Ultem® PEI (PolyEtherImide) http://www.boedeker.com/ultem\_p.htm

Ultem<sup>®</sup> has good dielectric properties and low thermal expansion, and is considerably less expensive than some other polymers. PEI is also clean and stable, but is not particularly resistant to chemicals or solvents ... it has outgassing values of 0.40% TML, 0.00% CVCM and 0.06 % WVR. PEI has good mechanical properties up to approximately 410°F.

• Semitron® ESd 410C (filled PEI) http://www.boedeker.com/sem410\_p.htm

Semitron® ESd 410C is antistatic/conductive PEI. This material is relatively clean, readily machinable, dissipates static electricity reliably ... as a result it is used in test handling equipment, fixtures, and other applications where static generation may cause failures and/or errors in production environments. This material has outgassing values of 0.46% TML, 0.00% CVCM and 0.17 % WVR. PEI has good mechanical properties up to approximately 340°F.

#### • Ertalyte® PET-P (Polyethylene Terephthalate) http://www.boedeker.com/ertaly\_p.htm

Ertalyte® offers the dimensional stability of acetal with the wear resistance of nylon. Ertalyte® PET-Polyester is clean, chemically resistant, stable, and also has relatively low outgassing values (0.13% TML, 0.00% CVCM ... % WVR is not shown). PET-P is considerably less expensive than most of the other materials listed above, but may not have the mechanical or thermal performance needed for all applications.

#### • Semitron® ESd 225 (filled acetal) http://www.boedeker.com/sem225\_p.htm

Semitron® ESd 225 is antistatic/conductive acetal. This material is relatively clean, readily machinable, dissipates static electricity reliably ... as a result it is used in test handling equipment, fixtures, and other applications where static generation may cause failures and/or errors in production environments. This material has low outgassing values of 1.00% TML, 0.05% CVCM and 0.60 % WVR. Acetal has good mechanical properties up to approximately 180°F.

# Appendix 3 – Materials for use in Vacuum

The choice of materials is limited for vacuum systems to a range that has little impact on vacuum production and ultimate base vacuums.

#### 1.1 Metals

- <u>Stainless Steel</u> is used for the majority of vacuum system designs due to its:
  - o Durability
  - Surface finishing
  - Ability to withstand moderate temperatures for system bake out
  - Low magnetic susceptibility
  - Low thermal conductivity
  - Resistance to oxidisation
  - Low vapour pressure

Type 304 or 316 stainless steel is ideally suited for vacuum vessel construction because of its machining/welding characteristics, excellent corrosion resistance and overall cost effectiveness.

Not all stainless alloys are acceptable. Free-machining 303 steel contains sulphur, which tends to outgas. Alloys with good weldability using TIG or MIG welding are usually chosen.

- 304 or 316 stainless steel is a common choice of a stainless steel.
- $\circ~$  304L stainless steel, a low-carbon variant of 304 steel, is used for ultra-high vacuum systems.
- 347 stainless steel does not accept high polish.
- $\circ$  321 stainless steel is chosen when low magnetic permeability is needed.
- <u>Mild steel</u> is okay for low to moderate vacuums above 10<sup>-4</sup> Pa. Outgassing can be reduced with suitable plating such as nickel. It has high permeability to hydrogen and tendency to rust. Mild steel must not be used for any components or chambers used near the bending magnets
- <u>Aluminium alloys</u> are easily machined and have a low vapour pressure, unless the alloys contain high proportion of zinc. 6061 grade is good for general use. Components used in high vacuums or better must not be anodized, as the oxide layer can trap water vapour and outgas. Aluminium and its alloys have low strength at high temperatures, distort when being welded, and the copper-containing ones are poorly weldable. Aluminium wire rings can be used as cheap gaskets in demountable seals. Soft alloys must be used to ensure wire gaskets do not damage the seal faces. Aluminium has high thermal conductivity, good corrosion resistance, and low solubility of hydrogen. Loss of strength at high temperatures limits its use in bakeable applications, but aluminium is advantageous for large-size systems due to its lower weight and lower cost than stainless steel. Aluminium is not recommended for vacuum chambers.
- <u>Brass</u> is not acceptable for vacuum chambers however it may be suitable for some small beam line inserts for specific applications. Once used widely but copper can cause problems with neutron production if struck by proton beams of particular energies. Brass is for high thermal conduction applications in cooling baffles or sample mounts that may be heated. Although bare

brass has good corrosion resistance the zinc content may cause outgassing problems. This can be reduced by plating with nickel.

- <u>Nickel</u> is widely used in vacuum technology, e.g. as mechanical parts in vacuum tubes. It is relatively low-cost, can be spot welded, can be easily machined, has high melting point and is resistant to many corrosive fluids and atmospheres. Its potential drawback is its ferromagnetism, which restricts applications that would be influenced by magnetic fields.
- <u>Beryllium</u> is used primarily for x-ray windows.
- <u>Oxygen-free copper</u> is okay for high vacuum but it is difficult to outgas completely. Copper is insensitive to hydrogen and impermeable to hydrogen and helium, has low sensitivity to water vapour, but is attacked by mercury. Although, oxygen-free copper is widely used as it is easily machined and has good corrosion resistance. It is unsuitable for bakeable vacuum envelopes due to its tendency to oxidize and create scales. Conflat flange seals are made from copper. Its strength falls sharply above 200 °C. Its vapour pressure becomes significant at above 500 °C.
- <u>Indium</u> wire is used as a gasket in demountable seals. Not suitable for high temperature above  $\sim 100^{\circ}$ C applications.
- <u>Gold</u> wire is used as a gasket in demountable seals for ultra-high vacuum.
- <u>Tantalum</u> is acceptable in vacuum systems but it is difficult to work with due to its hardness. It is expensive so it is usually purchased in sheet form. Typically mounted as a shield where ion beams contact a surface as it has a low production of secondary electrons. Good for apertures and defining slit faces and anywhere that a metal edge is used to intersect the ion beam.
- <u>Zirconium</u> is corrosion-resistant. Like tantalum, it has low production of secondary electrons, so it is used as a shield of areas where reducing their production is important. It is used for neutron windows. It is expensive and rarekly used. Zirconium and zirconium hydride are used for gettering.

#### **1.2 Plastics**

Plastics or ceramics are not to be used as the primary vacuum chamber structure without engineering advice from ANSTO or a specialist scientific vacuum instrument company. Plastic is very good for electrical insulators, bushes or light weight components are necessary in vacuums.

- <u>Polytetrafluoroethylene</u>, PTFE or Teflon as it is commonly known is suitable for use inside of vacuum systems from low to ultra high vacuums. Being soft PTFE can flow (creep) so for applications such as sample positioning systems or other systems requiring high stability then other plastics may need to be considered. PTFE has be far the highest dielectric strength, for extruded PTFE, 19.7 kV/mm 60 173 kV/mm for an insulating film. This compares with air which is around 1 kV/mm. It is self-lubricating, tolerant to fairly high temperatures, and has low outgassing. It is not suitable for barrier between vacuum and atmosphere, due to its permeability.
- <u>Ertalyte</u> offers the dimensional stability of acetal with the wear resistance of nylon. It is stable with relatively low outgassing values and has very low water absorption. It is very good for small mechanical loads requiring electrical isolation in vacuum systems.

- <u>Polyvinyl Chloride (PVC)</u> is acceptable in vacuum systems but not where heat is above 50°C. It is usually an amorphous thermoplastic material with excellent chemical resistance and dielectric properties, good tensile, flexural and mechanical strength, low moisture absorption, exceptional dimensional stability and good flammability characteristics.
- Other plastics can be used in vacuum systems but must have low vapour pressure. Care must be taken to ensure they are not used in applications that may require temperatures that will cause high outgassing rates.
- <u>Perspex</u> (acrylic plastic) is another plastic which has good dielectric properties but a high vapour pressure especially at raised temperatures making it unsuitable in vacuums however it can be used in small amounts in a well pumped high vacuum system.
- <u>Vespel</u>, a polyimide, is very expensive, but machines well, has good electrical insulator properties and is compatible with ultra-high vacuum. It does however absorb moisture and requires a longer pump down time. It also performs well in extremely low cryogenic temperatures. Good for bushes or where a mechanically stable plastic is required.
- <u>Nylon</u> is self-lubricating but has high outgassing rate and relatively high water absorption.
- <u>Polycarbonates and polystyrene</u> are good electrical insulators with moderate outgassing.
- <u>Mylar</u> is used to make thin windows that allow ion beams to penetrate from vacuum to positive pressures with little energy loss. For example gas detectors mounted onto beam lines for AMS have gas inside of the detector separated from the beam line which is under vacuum. Ion beams pass through the window as they move from the vacuum side to the positive pressure side. Other materials in this category include Kapton which is rated for higher temperatures.

#### **1.3 Elastomers**

Some elastomers have sufficient vacuum properties and are used widely as vacuum seals in the form of 'o' rings.

- <u>Viton</u> is the standard seal used throughout the accelerator area as it is long lasting, low vapour pressure as compared with other elastomers and is bakeable to 200 °C.
- <u>Nitrile</u> rubber is used for vacuum seals. Does tend to break down over time. First signs of breakdown are small splits. If used for vacuum seals then they must be inspected regularly.
- <u>Natural</u> rubber is not typically used in vacuum systems other than inside of vacuum pumps usually on the low vacuum side of the system.
- <u>Silicone</u> rubber is not generally used as a seal in high vacuum environments. Silicone is soft when compared with other elastomers. It has a relatively low vapour pressure.

Further reading: Materials for high vacuum technology: an overview, S. Sgobba, CERN, Geneva, Switzerland. http://cdsweb.cern.ch/record/983744/files/p117.pdf

#### 1.4 Ceramics and glass

• <u>Alumina</u> ceramic based ceramics in vacuum systems perform well provided the ceramic is not porous which can trap gas bubbles. Ceramics are readily available bonded onto metal flanges or mounting plates. Ceramic can also be baked if necessary to speed up the outgassing of surfaces.

Glass is similar to ceramics in this application. It is very low vapour pressure but difficult to work with. Glass viewports can be purchased already mounted in a metal flange.

- <u>Borosilicate glass</u> is often used for smaller assemblies and for viewports.
- <u>Porcelain</u> ceramics, when fully vitrified and therefore non-porous, are excellent insulators usable to 1500 °C. it is generally commercially available bonded to metal in electrical insulators and other feedthroughs.
- <u>Mica</u>, although it is neither ceramic or glass it best fits in this category. Mica has been used in vacuum systems for both electrical and thermal insulation but because Mica is a series of laminations it contains trapped air which is not suitable in high to ultra-high vacuums.
- <u>Macor</u> is a machinable glass made by Corning Incorporated with similar properties to ceramics. It has a low vapour pressure and thermally stable up to 1000°C making it bakeable. It is made from mica and borosilicate glass. It is by far the most flexible of the glass-ceramic options.

 Leakage Testing Handbook, Prepared for Liquid Propulsion Section, Jet Propulsion Laboratory, National Aeronautics and Space Administration, Pasadena, California
 Nondestructive Testing Handbook, Volume One, Leaktesting, American Society for Nondestructive Testing.
 Leakage Testing Handbook, Revised Edition, July 1969, General Electric.
 Fluid Flow in Small Passages, Mars Hablanian, J.W.Marr, Varian

#### 1.5 Greases and oils

There are few acceptable greases and oils for use in vacuum systems. Users must ensure they purchase greases and oils specifically designed for use in vacuums. Typically they will have low vapour pressures, extremely low water absorption and easy to remove for cleaning.

- <u>Apiezon family</u> For further information http://www.apiezon.com/ . These have been used throughout the history of accelerators at ANSTO with excellent results. Wherever greases are used remember to use the minimalist amount.
- <u>Greases</u>
  - Apiezon H High Temperature Vacuum Grease, silicone and halogen free
  - Apiezon L Ultra high vacuum grease, silicone and halogen free
  - Apiezon M High vacuum grease, silicone and halogen free
  - Apiezon N Cryogenic high vacuum grease, silicone and halogen free
  - Apiezon T Medium temperature grease, silicone and halogen free
  - o Apiezon AP100 Ultra High Vacuum Lubricating Grease, silicone free
  - o Apiezon AP101 Anti seize Vacuum Grease, silicone free
- <u>Waxes</u>
  - Apiezon® Waxes and Compounds
  - Apiezon Wax W (Hard Vacuum Sealing)
  - Apiezon Wax W40 (Softer Vacuum Sealing)
  - Apiezon Wax W100 (Softest Vacuum Sealing)

- Apiezon Sealing Compound Q
- <u>Vacuum Oil</u> is used in few applications around the accelerator area. With a change to oil free pumps most oil consuming pumps are redundant. However these types of pumps may still in use in other applications. Oil vane roughing pumps and diffusion pumps were the largest consumers of oil. More recent brands of vane pumps were specifically tuned to oils of particular specifications so generic oils may not be compatible. Diff pumps are more forgiving and high quality generic synthetic oils may be used. The list below is a small sample of what is available and previously used.
  - $\circ\,$  Shell Vitrea 68 oil for roughing pumps. Check specification on pump to ensure compatibility with this mineral oil
  - Fomblin PFPE (Perfluorpolyether) oils designed for rotary pumps. Check specification on pump to ensure compatibility with this mineral oil
  - Apiezon® Diffusion Pump Fluids Apiezon AP 201 The Apiezon diffusion pump fluid is known for its exceptional higher degree of oxidation resistance.

# **Appendix 4 – Surface finishing**

In most cases a clean polished surface will yield the least outgassing as there are minimal surface traps for gas. Also, a polished surface is easier to clean and keep clean. Achieving a polished surface may not be practicable due to accessibility into tubes, chambers and small components but a combination of other methods may provide an adequate surface finish that will allow the desired vacuum level to be reached. The best method for a given surface will depend on the material, geometry and accessibility to its surfaces.

- <u>Polishing</u> A polished surface may be either smooth or uneven but the metal will lustre in either case. Polishing is a method of removing a layer of metal to expose clean metal underneath. This can be done with a mechanical buff or by hand with the aid of a polishing compound. Care must be taken to ensure all traces of the polishing compound are removed during final cleaning. All residues must be removed.
- <u>Grinding, sanding and other coarse to fine abrasive methods</u> These methods tear the surface dragging metal over metal. Some metal breaks away exposing clean metal underneath. The problem with this type of surface preparation is that the metal can fold over and not break away leading to voids of trapped gases. It is important that a fine grit is the last grit used to ensure larger metal drifts on the surface are torn away.
- <u>Grit blasting with grit or beads</u> This method propels grit or beads onto the metal surface causing fragments of metal to be removed with the impact. Like the abrasive techniques metal can be layered on metal (peening) if the grit is too coarse. Best to finish with a fine grit then a mechanical polish. Grit blasting can provide a more uniform surface finish and is very good for removing scale that may be present from rolling or extrusions.
- <u>Pickling</u> by far a very good way to clean welds and other heat induced decolourisations in the metal. It will remove a thin layer of metal, scale, oils, etc. Care must be taken to ensure any chemicals used are neutralised and thoroughly cleaned away. Residues cannot be tolerated. Makes general maintenance of the surface easier.
- <u>Passivating</u> a form of chemical polishing and surface protection. Can be stimulated with an electric current. Passivating is the spontaneous formation of a hard non-reactive surface film that inhibits further corrosion. It isn't a preferred method of surface preparation in vacuum systems as damaged surfaces can form micro cracks trapping water and other gases. For example, damage to anodised aluminium may allow aluminium oxide to form which can readily trap water. Passivation is not typically used for stainless steel as the surfaces are self healing, provided sufficient oxygen is available.

Further reading on surface passivation can be found at: http://www.euro-inox.org/pdf/map/Passivating\_Pickling\_EN.pdfGetter surfaces

• Nickel Plating – has been used on vacuum components to provide stability to the surface of normally oxidising metals such as steel, copper and brass. It is important to ensure that the nickel has uniform bonding to the parent metal. Cases have been observed where the plating has lifted creating voids in the vacuum space. Although nickel is resistant to corrosion it will form a thin layer if exposed to moist atmospheres. A periodic clean is recommended to minimise problems with corrosion.

Element	Melting Point °C	Critical Temp °C	Phase at Critical T
Aluminium	660	1124	Liquid
Americium	1176	896	Solid
Antimony	631	455	Solid
Arsenic	84	270	Liquid
Barium	729	574	Solid
Beryllium	1289	1102	Solid
Cadmium	321	226	Solid
Caesium	29	114	Liquid
Calcium	847	538	Solid
Californium	900	1402	Liquid
Cerium	798	1602	Liquid
Chromium	1863	1220	Solid
Cobalt	1492	1418	Solid
Copper	1084	1146	Liquid
Dysprosium	1412	1025	Solid
Erbium	1529	1139	Solid
Europium	822	547	Solid
Gadolinium	1312	1450	Liquid
Gallium	30	954	Liquid
Germanium	938	1260	Liquid
Gold	1064	1291	Liquid
Hafnium	2231	2255	Liquid
Holmium	1474	1146	Solid
indium	157	836	Liquid
Iridium	2447	1199	Solid
Iron	1538	1253	Solid
Lanthanum	918	1586	Liquid
Lead	328	622	Liquid
Lithium	181	465	Liquid
Lutetium	1663	1517	Solid
Magnesium	651	386	Solid
Manganese	1246	887	Solid
Mercury	-39	22	Liquid
Molybdenum	2623	2319	Solid
Neodymium	1021	1219	Liquid
Neptunium	639	1781	Liquid
Nickel	1455	1409	Solid
Niobium	2468	2501	Liquid
Osmium	3033	2721	Solid
Palladium	1555	1348	Solid
Platinum	1769	1800	Liquid
Plutonium	640	1506	Liquid
Potassium	64	165	Liquid
Praseodymium	931	1381	Liquid
Protactinium	1572	2636	Liquid
Rhenium	3186	2817	Solid
Rhodium	1963	1874	Solid
Rubidium	39	129	Liquid

# **Appendix 5 – Critical Vapour Pressures**

Substance	Vapour Pressure (Pa)	Temperature (°C)
Acetaldehyde	98.7	20
Acetone	24640	20
Butane	22	20
Carbonyl sulphide	1255	25
Ethanol	5.83	20
Ethylene glycol	0.5	20
Formaldehyde	435.7	20
Freon 113	37.9	20
Methanol	12800	20
Methyl isobutyl ketone	26.48	25
Nitrogen (N <sub>2</sub> )	63200	20
Oxygen (O <sub>2</sub> )	54200	20
Propane	2200	55
Propanol	2.4	20
Tungsten	0.1	3203
Water (H <sub>2</sub> O)	2.3	20
Xenon difluoride	0.6	25

# **Appendix 5 – Vapour Pressures continued**

Substance	Vapour Pressure (Pa)	Melting point (°C)	
Apiezon Oil J	0.13 at 200°C 1.3 x 10 <sup>-4</sup> at 20°C	-	
Apiezon Oil K	0.13 at 300°C 1.3 x 10 <sup>-7</sup> to 10 <sup>-8</sup> at 20°C	-	
Apiezon Grease L	0.13 at 100°C 1.3 x 10 <sup>-7</sup> to 10 <sup>-9</sup> at 20°C	47	
Apiezon Grease M	0.13 at 200°C 1.3 x 10 <sup>-5</sup> to 10 <sup>-6</sup> at 20°C	44	
Apiezon Grease N	0.13 at 200°C 1.3 x 10 <sup>-6</sup> to 10 <sup>-7</sup> at 20°C	43	
Apiezon Grease T	About 1.3 x 10 <sup>-6</sup> at 20°C	125	
High Vacuum Grease Dow Corning	< 1 x 10 <sup>-4</sup> at 20°C	-	
Nylon	~ 1 x 10 <sup>-3</sup> at 20°C	-	
Teflon	< 1 x 10 <sup>-4</sup> at 20°C	-	
Viton	1 x 10 <sup>-6</sup> at 20°C	-	

# Appendix 6 – Outgassing tables for various materials Reference site: http://home.fnal.gov/~mlwong/outgas\_rev.htm

					i sampies.	
Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	(hours)	Test method	Reference	Year
Aluminium	None	1x10 <sup>-6</sup>	1h		Schamus (ref Markley, et al)	1999
Aluminium	Degassed	$1.7 \times 10^{-7}$	1h		Schmaus (ref Markley, et al)	1999
Aluminium	Degassed	2.7x10 <sup>-8</sup>	10h		Schmaus (ref Markley, et al)	1999
Aluminium 6061-T6	Baked 13.5h @ 300°C	1.4x10 <sup>-8</sup>	10h		Schmaus (ref Das)	1999
Aluminium	Cleaned	8x10 <sup>-9</sup>	10h		Schmaus (ref Blears, et al)	1999
Aluminium	Fresh, degreased w/ trichloroethylene & cleaned w/ ethyl alcohol	6.3x10 <sup>-9</sup>	1h	conductance	Elsey (ref Schram)	1975 (1963)
Aluminium	Degassed	$4.6 \times 10^{-9}$	100h		Schmaus (ref Markley, et al)	1999
Aluminium 6061-T6	Baked @ 200°C	4.5x10 <sup>-9</sup>	10h		Schmaus (ref Das)	1999
Aluminium	Degassed 24h, degreased w/ trichloroethylene & cleaned w/ ethyl alcohol	4.14x10 <sup>-9</sup>	1h	conductance	Elsey (ref Schram)	1975 (1963)
Aluminium 6061-T6	None	2.5x10 <sup>-9</sup>	10h		Schmaus (ref Das)	1999
Aluminium 5083-O	Bell jar, as received, room temp.	2.18x10 <sup>-9</sup>	10.3h	Rate-of-rise	Schrank, et al	1964
Aluminium 5083-O	Bell jar, as received, room temp.	1.27x10 <sup>-9</sup>	8h	Conductance	Schrank, et al	1964
Aluminium 5083-O	Bell jar, as received, room temp.	6.6x10 <sup>-10</sup>	24h	Conductance	Schrank, et al	1964
Aluminium 5083-O	Bell jar, as received, room temp. after baking @ 220°C	$4.6 \times 10^{-10}$	50h (baked @27-31h)	Conductance	Schrank, et al	1964
Aluminium 6061-T6	Baked 15h @ 300°C	$1.6 \times 10^{-10}$	10h		Schmaus (ref Das)	1999
Aluminium	Degassed 24h, degreased w/ trichloroethylene & cleaned w/ ethyl alcohol	3.06x10 <sup>-10</sup>	1h	conductance	Elsey (ref Schram)	1975 (1963)
Aluminium 5083-O	Bell jar, no. 4 finish (lapped), room temp.	$2.87 \times 10^{-10}$	6.0h	Rate-of-rise	Schrank, et al	1964
Aluminium	Fresh, degreased w/ trichloroethylene & cleaned w/ ethyl alcohol	6.0x10 <sup>-10</sup>	10h	conductance	Elsey (ref Schram)	1975 (1963)
Aluminium, type 1100	Cleaned w/ detergent, rinsed w/ acetone, pumped 24 hours	~10 <sup>-10</sup>	0	conductance	Young	1968
Aluminium	LEP vacuum chamber, chem clean, baked in situ @ 150°C	$2.3 \times 10^{-11}$	24h		Mathewson, et al	1988
Aluminium 6061-T4	Degreased in acetone w/ methanol rinse; baked 100°C	6x10 <sup>-12</sup>	24h	Rate-of-rise & conductance	Halama, Herrera	1976

Outgassing rates of aluminium Note the different methods of measurement and treatment of samples.
Aluminium 6061-T4	Degreased in acetone w/ methanol rinse, chem polished, baked 100°C	3x10 <sup>-12</sup>	24h	Rate-of-rise & conductance	Halama, Herrera	1976
Aluminium 6061-T4	Degreased in acetone w/ methanol rinse, chem polished, glow discharge in Ar, baked 100°C	5x10 <sup>-13</sup>	24h	Rate-of-rise & conductance	Halama, Herrera	1976
Aluminium, type 1100	Above plus baked 15h @ 250°C under vacuum	$4x10^{-13}$	24h @ room temp	conductance	Young	1968
Aluminium	PETRA vacuum chamber, glow discharge@145°C	$1 \times 10^{-13}$	Up to 200h	In situ glow discharge, conductance	Mathewson, et al	1977

# Outgassing rates of stainless steels, along with the surface treatment and pumping time.

Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	Time (hours)	Test method	Reference	Year
Stainless steel	None	6.4x10 <sup>-7</sup>	1h		Schamus (ref Markley, et al)	1999
Stainless steel	degreased	4x10 <sup>-7</sup>	1h		Schamus (ref Markley, et al)	1999
Stainless steel	None	2x10 <sup>-7</sup>	1h		Schamus (ref Blears, et al)	1999
Stainless steel	None	2x10 <sup>-8</sup>	10h		Schamus (ref Blears, et al)	1999
Stainless steel NS22S	Fresh	1.4x10 <sup>-8</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel ICN 472	fresh	1.3x10 <sup>-8</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel	None	1.2x10 <sup>-8</sup>	10h		Schamus (ref Das, et al)	1999
Stainless steel ICN 472	sanded	8.3x10 <sup>-9</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel NS22S	electro polished	4.3x10 <sup>-9</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel NS22S	mechanically polished	1.7x10 <sup>-9</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel	fresh	1.5x10 <sup>-9</sup>	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel ICN 472	Polished & vapor degreased	1.4x10 <sup>-9</sup>	10h		Schamus (ref Dayton, et al)	1999
Stainless steel	None	1.4x10 <sup>-9</sup>	10h		Schamus (ref Das, et al)	1999
Stainless steel NS22S	fresh	1.3x10 <sup>-9</sup>	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel ICN 472	sanded	1.0x10 <sup>-9</sup>	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel	None	7.6x10 <sup>-10</sup>	1h		Schamus (ref Das, et al)	1999
Stainless steel NS22S	mechanically polished	$4.6 \times 10^{-10}$	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel NS22S	electro polished	$4.3 \times 10^{-10}$	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel	Baked 24h @ 200°C	$1.5 \times 10^{-10}$	1h		Schamus (ref Das, et al)	1999

Stainless steel	None	$1.1 \mathrm{x} 10^{-10}$	100h		Schamus (ref Das, et al)	1999
Stainless steel 304	Degrease + water rinse	$4.0 \times 10^{-11}$	40h	Conductance	Strausser	1973
Stainless steel U15C	Baked 25h @ 300°C	$4.5 \times 10^{-12}$			Elsey (ref Calder, et al)	1975
Stainless steel 304	Degrease + water rinse, baked in vacuum 150°C for 12h	4.0x10 <sup>-12</sup>	5h after bakeout	Conductance	Strausser	1967
Stainless steel 304	Electropolished, baked 30h @ 250°C	$3.0 \times 10^{-12}$			Elsey (ref Young)	1975
Stainless steel U15C	Baked 45h @ 360°C	$2.6 \times 10^{-12}$			Elsey (ref Calder, et al)	1975
Stainless steel	Baked 24h @ 200°C	$9.3 \times 10^{-13}$	100h		Schamus (ref Das, et al)	1999
Stainless steel U15C	Baked 3h in vacuum @ 1000°C + 25h in situ @ 360°C	1.6x10 <sup>-14</sup>			Elsey (ref Calder, et al)	1975

Other cleaning methods although outgassing rates are not measured:

Material	Treatment	Results	Reference	Year
Aluminium	Quench a hot aluminium extrusion in an Ar- $O_2$ atmosphere	Dense, thin (~20Å thick) oxide layer	Sasaki, Y.T.	1990
Aluminium 6063	Clean with an alkaline detergent (Almeco 18)	Removes MgO, C, and Al <sub>2</sub> O <sub>3</sub>	Sasaki, Y.T.	1990
Aluminium	Vapour degreasing, pump for 120h before and after bake	Dominant residual gases: H <sub>2</sub> O, H <sub>2</sub> , CO, CO <sub>2</sub> , & CH <sub>4</sub> ; after baking @ 150°C and pumping, H <sub>2</sub> O & CH <sub>4</sub> gone, outgassing rates of other gases reduced 1 order of magnitude except H <sub>2</sub>	Mathewson, A.G., et al	1989
Aluminium	Vapour degreasing, then light alkaline etch (CERN basic procedure), pump for 120h before and after bake	Dominant residual gases: H <sub>2</sub> O, H <sub>2</sub> , CO, & CO <sub>2</sub> ; after baking @ 150°C and pumping, only H <sub>2</sub> exists	Mathewson A.G., et al	1989
Aluminium	Strong alkaline etching in NaOH, pump for 120h before and after bake	Dominant residual gases: H <sub>2</sub> O, H <sub>2</sub> , CO, CO <sub>2</sub> , & CH <sub>4</sub> ; after baking @ 150°C and pumping, only H <sub>2</sub> exists	Mathewson A.G., et al	1989
Aluminium	Vapour degreasing, Ar glow discharge @ 150°C	On initiation of discharge, large pressure increase (normal in Ar glow discharge) was too large for accurate measurement	Mathewson A.G., et al	1989
Aluminium	Vapour degreasing, then light alkaline etch (CERN basic procedure), Ar glow discharge @ 150°C	H <sub>2</sub> , CO dominant gases desorbing from surface during discharge cleaning	Mathewson A.G., et al	1989
Aluminium	Strong alkaline etching in NaOH, Ar glow discharge @ 150°C	H <sub>2</sub> , CO dominant gases desorbing from surface during discharge cleaning at order of magnitude less than light alkaline etch	Mathewson A.G., et al	1989
SS	H <sub>2</sub> glow discharge - 22°C	Dominant residual gases: $CH_4$ , $CO$ , & $C_2H_4$ ; water production insignificant; frequent arcing	Dylla, H.F.	1988

		on all exposed surface noted during initial discharge period		
SS	H <sub>2</sub> glow discharge – 150°C	Dominant residual gases: $CH_4$ , $CO$ , & $C_2H_4$ , and $H_2O$ ; frequent arcing on all exposed surface noted during initial discharge period	Dylla, H.F.	1988
SS	Ar glow discharge – ambient temp	Dominant residual gases that were significantly removed: $CO_2$ , $H_2$	Dylla, H.F.	1988
SS	Ar/O <sub>2</sub> glow discharge	Improved removal of CO and CO2; minor amts of implanted Ar that can be removed by baking @ 350°C	Dylla, H.F.	1988
	Ar/O <sub>2</sub> glow discharge 2h, exposed to air, then Ar/O <sub>2</sub> glow discharge 2h	Some degree of passivation: residual gases were removed with 1 <sup>st</sup> cleaning & did not show up during the 2 <sup>nd</sup> cleaning	Dylla, H.F.	1988
	O <sub>2</sub> glow discharge	Rapid removal of carbon, not of hydrocarbons (which can be baked out initially), increased surf. Oxidation, sputtering of the base metal, and possible enhanced outgassing of O <sub>2</sub> - containing molec.	Dylla, H.F.	1988

# Different outgassing measurements of epoxies

Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	Time (hours)	Test method	Reference	Year
NMA w/ solid alumina filler	Cured 300°F	$1.6 \times 10^{-7}$			Hanson, Patel	1970
Polyimide polymer	Thermosetting; measured @ 40°C	~1.1x10 <sup>-7</sup>	4h+9min	conductance	Kendall, Zabielski	1965
Polyimide polymer	Thermosetting; measured @ 155°C	~5x10 <sup>-8</sup>	4.5h increase temp + 6h	conductance	Kendall, Zabielski	1965
Silicone resin	Needs dicumyl peroxide catalyst; thermosetting, measured @ 35°C	~5x10 <sup>-8</sup>	4h	conductance	Kendall, Zabielski	1965
Araldite epoxies	Cured ~150°C for 15h	~10 <sup>-8</sup>	51h pumping	conductance	Barton, Govier	1965

# Kapton is a newer material and little data exists about its outgassing rate. Below are 3 references

Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	Time (hours)	Test method	Reference	Year
Kapton cable		1.0x10 <sup>-5</sup>	0h	conductance	Postma	1999
Kapton foil		$1.0 \times 10^{-7}$	40h		Ferro-Luzzi	1999
Polyimide	Bake @ 300°C	$4.0 \mathrm{x} 10^{-8}$	12h		Weston	1970
Kapton cable		$2.4 \times 10^{-8}$	168h	conductance	Postma	1999
Kapton cable		2.7x10 <sup>-9</sup>	336h	conductance	Postma	1999
Kapton cable		$6.5 \times 10^{-10}$	504h	conductance	Postma	1999

# Outgassing rates of several types of plastics

Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	Time (hours)	Test method	Reference	Year
Nylon		$1.2 \times 10^{-5}$	1h		Elsey (ref	1975
					Power, et al)	
Nylon		6.0x10 <sup>-6</sup>	1h		Elsey (ref	1975

					Power, et al)	
PVC	24h @ 95% relative	8.5x10 <sup>-7</sup>	1h	conductance	Elsey (ref	1975
	humidity				Santler)	
PTFE	Fresh	$1.7 \mathrm{x} 10^{-7}$	1h	conductance	Elsey (ref	1975
					Santler)	
Teflon		6.5x10 <sup>-8</sup>	1h	conductance	Elsey (ref	1975
					Santler)	
PTFE	Fresh	3.3x10 <sup>-8</sup>	10h	conductance	Elsey (ref	1975
					Santler)	
Teflon		$2.5 \times 10^{-8}$	10h	conductance	Elsey (ref	1975
					Santler)	
PVC	24h @ 95% relative	$2.0 \times 10^{-8}$	10h	conductance	Elsey (ref	1975
	humidity				Santler)	
G-10		~10 <sup>-8</sup>			Beams	2001
					Division -	
					FNAL	

Reference to the following has been lost but it is believed to have originated in an early Vacuum Catalogue from the 1990's

 $K_1$  is the outgassing rate (air equivalent) after 1 hour pumping  $\alpha_1$  is the slope of the (log K - log t) curve at 1 hour

Material	K <sub>1</sub> Torr litre/sec cm <sup>2</sup> (x 10 <sup>-10</sup> )	α1	K <sub>10</sub> Torr litre/sec cm <sup>2</sup> (x 10 <sup>-10</sup> )	a <sub>10</sub>
Aluminium Alloy (fresh)	63	1	6.0	1
Aluminium Alloy (degassed 24 hours)	41.4	3.2	3.06	0.9
Aluminium Alloy (3 hours in air)	65.5	1.9	4.75	0.9
Aluminium Alloy (anodised -2µ pores)	2760	0.9	322	0.9
Aluminium Alloy (bright rolled)	-	-	75	1
Duralumin	1700	0.75	350	0.75
Brass (wave guide)	4000	2.0	100	1.2
Copper (fresh)	400	1	41.5	1
Copper (mechanical polish)	35	1	3.56	1
OFHC Copper (fresh)	118	1.3	12.6	1.3
OFHC Copper (mechanical polish)	19	1.1	1.63	1.1
Gold (fresh wire)	1580	2.1	5.1	1
Mild steel	5400	1	500	1
Mild steel (slightly rusty)	6000	3.1	130	1
Cr plated steel (fresh)	70.5	1	6.8	1
Cr plated steel (polished)	91	1	8.0	1
Ni plated steel (fresh)	42.4	0.9	4.84	0.9
Ni plated steel	27.6	1.1	2.33	1.1
Chemically Ni plated steel (fresh)	83	1	7.05	1
Chemically Ni plated steel (polished)	52.2	1	4.6	1
• • • • •				

Araldite (moulded)	116	0.8	35.2	0.8
Araldite D	800	0.8	220	0.78
Araldite D	190	0.3	125	0.5
Araldite F	150	0.5	73	0.5
Celluloid	860	0.5	430	0.5
(PTFE) (fresh)	16.6	0.8	3.31	0.9
Kel-F Oak ridge	4	0.57	1.7	0.53
Methyl methacrylate	420	0.9	140	0.57
Mylar V-200 (24 hr at 95% RH)	230	0.75	40	-
Nylon	1200	0.5	600	0.5
Pertinax	620	0.18	290	0.5
Perspex	72	0.44	27	0.44
Perspex	310	0.4	180	0.4
Polyamid	460	0.5	230	0.5
Polyester - glass laminate	250	0.84	80	0.81
Polyethylene	23	0.5	11.5	0.5
Polystyrene	2000	1.6	200	1.6
Polystyrol	56	0.6	12	0.61
Polyvinylcarbazol	160	0.5	80	0.5
PTFE	30	0.45	15	0.56
P.V.C. (24 hr at 95% RH)	85	1.00	2	-
Teflon	6.5	0.6	2.5	0.2
Terephenil (fresh)	62.2	0.5	16.8	0.5
Neoprene	3000	0.4	-	-
Viton	114	0.8	-	-
	1	1		

# **Appendix 7 – Local Plant and Safety Regulations**

Regular maintenance inspections are carried out on ANTARES and STAR accelerator vacuum equipment to ensure operational status, vacuum quality, vacuum pump performance, and the mechanical and electrical integrity of the systems.

Generally, the integrity of the vacuum systems can be measured as a direct function of the known static vacuum pressures in a given vacuum system. That is, a lowering of vacuum from the known average vacuum pressure in a given system will indicate either/and, a leak in a seal, a failing vacuum pumping system or a structural failure.

The following is an overview of the application of the ANSTO OHSE Plant and Safety Standard, AS 2309, to vacuum systems in the accelerator facilities. *Reference: http://docushare.ansto.gov.au/Get/File-32340/Plant\_Safety\_Standard.pdf* 

Risk	Hazard	Risk rating	Controls	Risk rating (with controls in place)
Catastrophic failure vacuum vessels	Implosion – scattering of material fragments and loose fittings becoming missiles	Very low	<ul> <li>Use of vacuum standards to design equipment for safe usage.</li> <li>Protective barriers around equipment and fittings that may be susceptible to knocking</li> </ul>	Very low
Cotostrophia failura	• Implosion – scattering of material fragments and loose fittings becoming missiles	Very low	<ul> <li>Use of vacuum standards to design equipment for safe usage.</li> <li>Purchased equipment from reputable companies/designers only</li> </ul>	Very low
Catastrophic failure vacuum beam lines and associated equipment	• Electric shock from fault in high vacuum gauge	Very low	• Vacuum gauges using high voltages in key areas will automatically switch off at low vacuums/atmospheric pressure	Very low
	• Failure of welded joint	Very low	• System will leak to atmospheric pressure at a rate commensurate with the conductance of the failed joint. No control is deemed necessary.	Very low
Electric shock from a failed vacuum pump or powered fitting	Electric shock from frayed lead or failure of earth inside of equipment	Low	<ul> <li>All equipment is protected from short circuits by circuit breaker protection.</li> <li>Most circuits now have RCD protection.</li> <li>A program is in place to put all circuits onto RCD protection</li> </ul>	Very Low
Failure of support structures	Crush, fall, bump, trip hazards during and after failure	Low	<ul> <li>Use of vacuum standards to design equipment for safe usage</li> <li>Access to equipment at height via approved ladders or dedicated platforms</li> <li>Non acceptance of standing on beam line stands and mounts</li> </ul>	Very Low
breakages to	<ul> <li>Fragments of broken</li> </ul>	LOW	<ul> <li>Barriers around exposed equipment</li> </ul>	LOW

1) Risk assessment of plant – Accelerator Facilities Only

fittings and equipment protruding from vessels and beam lines	<ul> <li>parts become missiles.</li> <li>Cuts to personnel bumping into fittings</li> <li>Secondary injuries including electric shock from exposed wiring</li> </ul>		<ul> <li>that is deemed to be hazardous</li> <li>Access control into accelerator areas restricted to accelerator personnel and visitors who are escorted</li> <li>See electric shock hazard controls above</li> </ul>	
Over pressure of vacuum systems during venting	Positive pressure in beam lines when venting with gases from high pressure storage	Very Low	<ul> <li>Pressure relief valves in use at systems where regular vacuum cycling is necessary</li> <li>Use of portable pressure relief fittings where local venting is required.</li> </ul>	Very Low
Implosion of glass view ports	Shards of glass scattering towards operators	Low	<ul> <li>Use purpose designed commercial vacuum windows</li> <li>Use small diameter (&lt;50mm x 6 mm thick) quartz windows on vacuum systems where ion beams can strike. Quartz is not known to crack through but to develop small surface anomalies</li> <li>Use double rubber seals to support windows to minimise mechanical stresses</li> </ul>	Low
Broken film windows	Sudden venting of vacuum to atmospheric pressure	Very Low	<ul> <li>Damage to equipment – high vacuum gauges must have auto shutdown at low vacuums</li> <li>Vacuum system isolated with gate valve to minimise loss of vacuum in adjoining systems</li> </ul>	Very Low
Breach of accelerator tubes with SF6	Vacuum systems pressurised with SF6 gas	Low	<ul> <li>Large gate valves either end of the accelerator vacuum tubes will shut if pressure rises to maintain SF6 within the vacuum vessels</li> <li>Some gas will enter the vacuum systems outside of the isolated section. The pressure will be relieved through blow off venting ports at each end of the vessel.</li> <li>Oxygen depletion alarms will sound if dangerous levels of SF6 are released. Personnel will be ordered to leave building through PA announcement</li> </ul>	Low

- 2) Risk control measures for eliminating or reducing the assessed risks *See table above*
- 3) Registration or notification of design of plant *Not deemed necessary. See AS 4343-2005*
- 4) Licensing of plant Not deemed necessary. See AS 4343-2005
- 5) Maintenance of plant

*Regular maintenance applied via Accelerator Maintenance system. See ENV-I-076-004 Vacuum Systems Maintenance manual* 

- 6) Plant operations and risk controls *See table above*
- 7) Requirements in relation to specific types of plant *Only vacuum system plant*
- 8) Training and supervision Vacuum technology training is provided in-house to world's best practices. Additional training has been provided by the Vacuum Society of Australia through their training courses.
- 9) Monitoring and evaluation Regular maintenance applied via Accelerator Maintenance system. See ENV-I-076-004 Vacuum Systems Maintenance manual

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# Vacuum Technology

# and

# Vacuum Design Handbook

# for

# **Accelerator Technicians**

Prepared by David Garton November 2011 Revision 0 Vacuum Technology and Vacuum Design Handbook for Accelerator Technicians

This handbook is a compilation of information gathered from over 50 years of direct hands-on experience to applicable information widely available from the vacuum technology industry. It seeks to address common and specific vacuum technology problems whilst clarifying the design standards and philosophies adopted for use in the ANSTO accelerator facilities. The author wishes to thank the reviewers and the many technicians from ANSTO that have contributed directly and indirectly to this booklet.

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# Chapter 1

# Vacuum design standards and good practises for the Accelerator Area

- \* This chapter must be read in conjunction with the detailed vacuum information in this document.
- \* Based on best practises developed at ANSTO and other local Australian accelerator laboratories in conjunction with Australian Standards and industry technical guides, referenced throughout this document.
- \* Where a standard is unavailable for a specific design, best practices are used from industry's best practises.
- \* *Refers to all vacuum chambers, beam lines and other enclosures used on the accelerators or stand alone.*

## 1. Basic design

- All vacuum vessel designs must be consistent with the design standards in Australian Standard AS 1210-2010 and maintain a Hazard Level of E as per the design conditions tabled in "Hazard Levels of Pressure Equipment" in Section 2, Hazard Levels of Pressure Equipment in Australian Standard AS 4343-2005.
  - The vacuum equipment designed by/for the accelerator area maintains a Hazard level of E. All pressure equipment with a pV  $\leq$  30, pressure (p in MPa) x volume (litres)  $\leq$  30 is classed as Hazard level of E. For vacuum equipment in Hazard Level E the derating value of 0.1 is applied, ie 0.1pV  $\leq$ 30. Note the amount of stored energy at this level is very small. For example a Ø 1000 mm chamber, 1000 mm high will have a pV for vacuum equal to 7.85.
  - AS 4343-2005, Section 2.1.2 Typical hazard levels, part (d), states for Hazard Level E, "This equipment is usually exempt from special regulatory control but is <u>covered</u> by general plant safety regulations". See Appendix 7.
- All designs of chambers excluding general beam lines and fittings should be checked for structural integrity using finite elements analysis. Sound designs with acceptable structural integrity will be released for manufacture. A copy of the design acceptance tests must be filed with the project file. The standard finite element analysis can be performed utilising Solidworks. (Modelling for irregular shaped chambers, calculations Appendix 1 where appropriate for regular shapes)
- Stainless steel (304 or 316) will be used to construct chambers, beam lines and general fittings unless it is a *special* requirement to use other metals. Do not choose substances that have high outgassing rates or can contaminate vacuum systems.
- Measurement chambers and other large volume chambers should be cylindrical unless the chamber is an enclosure between pole faces (eg magnet box) or is an odd shape to accommodate steering plates or devices. When non-cylindrical chambers are required deflection of chamber walls should be modelled and taken into consideration.
- Other chambers including magnet boxes, ESA and deflector coffins to have suitable rib strengthened rectangular or curved sides where it is not practicable to increase the wall thicknesses.
- Minimum wall thickness calculation methods can be found in Appendix 1. As a general rule care must be taken when using formulas for vacuum calculations as historically many units other than SI units have been used to form some equations. For example length in cm, and pressure in torr is common in old notes.

- Top and bottom flanges to have integrated structural support rim which becomes the flange and seal for chamber base and top (user) flanges.
- Ports must be a suitable length to ensure unobstructed insertion for screws into flanges. Suitable weld preparation must allow for adequate weld penetration. See Welds below.
- All internal edges must have a smooth radius. No sharp edges to be left after welding or surface finishing.
- All machined finishes other than vacuum seals to be equal to or better than 1.6 microns
- No sections within the vacuum space will have metal to metal contact where gas can be trapped unless it is at the edge of a seal joint. When this is necessary, pump out grooves must be incorporated in the design.
- Vacuum windows shall be made from toughened glass or quartz. No plastic to be used for windows unless the window is a flange on a chamber that is designed to withstand more than 100 kPa. The large plastic flange/windows should be made from >18 mm thick up to Ø200 and >25 mm thick for up to Ø400 in clear Perspex or polycarbonate.
- For ANTARES beam lines the preferred flanges are Dependex, nominal diameter 100 mm. See section 8 for detail on Dependex flanges.
- For STAR there is no preferred flange type. It must be selected depending on the desired base vacuum and interface to other adjoining flanges.
- As a guide, the MDC catalogue from Vacuum Products Corporation 2003 onwards, Building Blocks for Vacuum Science and Technology, provides vacuum tubing dimensions for most common sizes. The dimensions must be assumed to be minimum dimensions. Other tubing sizes should be calculated using the formulas in Appendix 1.
- Vacuum pumping ports leading to the vacuum pumps must be designed to optimise conductance.
- Pressure relief valves to be incorporated where a vacuum system can be vented to >10 kPa above atmospheric pressure.
- ASME state that the design of an external pressure vessel must consider Material type, diameter of chamber, unstiffened length, temperature and wall thickness

# 2. Welds

- TIG is preferred for vacuum sealing welds as minimal scale and flux is produced. Electron beam welding is suitable for thin walled materials. TIG produces high quality welds with or without filler rods to produce flat, smooth well penetrated welds. TIG comes into its own where the weld preparation allows for melting together of the parent materials without filler rods. MIG has a higher deposition of filler material so care must be taken to ensure welds are kept smooth and flat.
- No standard exists for vacuum welding of compact scientific vacuum equipment where pressures approaching zero are necessary. Equipment designed in the accelerator area and welded in the ANSTO workshops since 1990 has had penetrations of ≥1mm but ≤2mm on a butt weld and ≥2 mm on a fillet weld. Experience has demonstrated the adequacy of these figures. No failure reported.
- For thin walled materials ie < 1 mm, such as bellows, manufacturers' will use their industry standard. ANSTO does not weld thin wall materials.
- Weld preparation must be provided where possible to ensure even heat penetration.

- Electric arc with rods is not recommended due to flux trapping. This can cause pits which have long lasting outgassing periods.
- All welds that form a seal against atmospheric or other positive pressures must be made to the internal side (vacuum side) of the joint.
- Tack (stitch) welding is recommended on the external side only with less than 50% of the diameter covered in small intervals. This minimises the chance of trapped voids and facilitates He leak testing post welding.
- All welds must be free from pitting and scale. Must be cleaned to bare metal. If chemicals are used to clean welds care must be taken to remove all traces of chemicals.
- Some chambers such as magnet boxes will need to have some joints welded externally due to access problems on thin chambers. In this case, good penetration must be achieved to ensure minimal gas trapping points.

## 3. Surface finishes

- The inside of the chamber and ports must be 1.6 micron or better for mechanical finishes.
- Can be chemically cleaned (polished) but all traces of chemicals must be neutralised and removed prior to use in vacuum.
- All machining oil to be removed with an alcohol scrub then acetone rinse, minimum. The use of kerosene, turpentine or other oily solvents is not encouraged however where they are used the final clean must be with acetone followed by alcohol. Water based cutting fluids can be used as they are more readily removed therefore having less effect on outgassing.
- Nitrile gloves to be used when assembling cleaned components. Other un-powdered gloves may be used but only those that do not react with the solvents being used.
- Care must be given to cleaning and handling surfaces that will be exposed to vacuum. Gloves should be used to minimise the transfer of dirt and oils from the skin to vacuum surfaces.
- A suitably clean area should be used for the preparation of vacuum systems and all tolls used should also be cleaned (degreased).
- Stainless steel can be cleaned in nitric acid to remove scale that remains from rolling or wire cutting. It is may also be used to prepare stainless steel components for use in ultra-high vacuum systems.

# 4. Flanges

- Flanges must be standard flanges selected from the group referenced this document for compatibility reasons. Exceptions may be made but all 'o' ring seals must be to Australian Standard AS 2842-1986. External flange rings not in contact with the vacuum space may be aluminium alloy. Stainless flanges are not economical or necessary unless the seal requires forces to crush metal seals such as copper ConFlat, aluminium or indium wire.
- Minimum thicknesses for top and bottom flanges to be calculated using the standard in Appendix 1 Structural Calculations for Scientific Vacuum Vessel Design.
- All other flanges to have threads consistent with their design standard.
- ANTARES Primary flanges will be Dependex unless interface is necessary to accommodate other standard flanging.
- For Dependex, screws to be imperial sizes consistent with UNC series.

• STAR utilises many of the major vacuum flange/seal designs. The most appropriate design must be chosen that meets the specific application.

## 5. Seals

- 'O' ring sizes and sealing groove dimensions must comply with the Australian Standard AS 2842-1986. Adjustments to groove sizes can be made where specific 'crush' is necessary for special applications.
- Viton rubber 'o' rings to be used unless a special requirement for an alternate elastomer is identified.
- Standard flange and seal sizes to be used unless an application prohibits their use.
- It is recommended that vacuum sealing surfaces are cleaned just before a seal is made to minimise the possibility of dust and hair settling on sealing surfaces.
- Metal seals may be used in specifically designed sealing surfaces including, aluminium and indium wires. They are very good as ultra-high vacuum seals.

## 6. Screws

- All screws for flanges other than Conflat will be made from stainless steel in either 304 or 316 grades.
- Screws used in Conflat flanges will be made from A2-70 or A4-70.
- All screws to be socket head unless access necessitates hexagon or other special head. Slotted or Philips head must not be used.
- Stainless steel flat washers to be used under all screws where practicable. Do not use lock or star washers on standard flanges.
- In the vacuum space, no screws to be used in holes unless the holes are through holes and can be pumped from both sides. Screws can be used in blind holes if they have a minimum Ø1 mm hole drilled through the centre to allow trapped gas/air to escape.
- Unless a standard flange uses non metric thread, all screw threads must be within the metric series. Unless there is a specific need, avoid using intermediate metric sizes, that is, 0.5 mm steps such as M1.5 mm, M2.5 mm etc.
- Anti-seizing compounds can be used on threads but extreme care must be taken to ensure they do not enter the vacuum space. Graphite powder can be used as a lubricant inside the vacuum space. It can be applied using ethanol to wet the surface to be lubricated. Allow solvent to evaporate prior to pumping.

# 7. Feedthroughs

- Sliding seals should have dual 'o' ring seals with a gland formed seal at least at one end. The shaft finish must be 0.8 micron or better.
- All sliding shafts must have retainers to stop them being pulled through into the vacuum space.
- All coax feedthroughs to be glass or ceramic hermetically sealed. No plastic.

#### 8. Lubricants, vacuum greases and epoxies

- Only vacuum specific grease and oils to be used in vacuum systems.
- Silicone based or particular Apiezon family lubricants design for vacuum use, are recommended on sliding seals. Thin smears only.
- No vacuum greases should be used unless there is a small leak through a damaged sealing surface and all other options are not feasible. If grease must be used it must be designed for high vacuum use and only a very thin smear to be used.
- Do not use grease to fill suspected leaks in welds or fittings.
- Where a vacuum epoxy must be used the surface must be well prepared to ensure appropriate bonding. Care must be taken to ensure the epoxy seal doesn't create a trapped void of air which may become an outgassing problem. 'Torr-Seal' is commonly used due to its relatively low vapour pressure, and thermally stable to 120°C. (this is a Varian product)
- Vacuum oils are not recommended for general lubrication as they can creep on surfaces spreading contamination and increasing the outgassing surface.
- See Appendix 3 section 1.5.

#### 9. Mounting

- The load of the chamber assembly must rest on a standard accelerator beam line table ANSTO, HVEC, HVEE or NEC or a purpose built stand that minimises vibration.
- Vibration decoupling will be achieved by supporting the chamber separately from the vacuum pump and using an edge welded bellows between the chamber and pump.
- For ANTARES beam line vacuum equipment the beam axis alignment is nominally 1750 mm from floor height.
- For STAR beam line vacuum equipment the beam axis alignment is nominally 1193 mm from floor height.

#### 10. Access

- The working face of the chamber must face an area that is easily and comfortably accessible. The floor space immediately in front of the working face should be raised using steps to facilitate access.
- Heavy or awkward shaped vacuum equipment must be designed to be lifted with the aid of over cranes for example lifting lugs.

#### 11. Testing

- Vacuum equipment must be tested prior to routine use to minimise time spent chasing leaks in completed systems.
- Vacuum vessels must be helium leak tested to ensure all seals are better than  $1 \ge 10^{-10}$  Pa.m<sup>3</sup>/s ( $1 \ge 10^{-9}$  mbar.l/s). As a standard, during a leak test there should be no deviation from this baseline level for a leak-tight system. A record of the test is to be noted on the engineering drawing showing, date, maximum leak rate, leak location/s, leak detector used and operator name.

- For all other vacuum equipment in-service testing may be necessary to allow complete operation of various feedthroughs and positioners.
- Caution must be taken before positive pressure testing vacuum equipment as the equipment may be irreversibly damaged. Warning Do not use water testing on any vacuum equipment.
- A Residual Gas Analyser (RGA) can be used to give more detailed information about the gas composition inside a vacuum volume. An RGA can be used as a substitute for a helium leak detector with the benefit of providing more information about outgassing, or virtual leaks inside the vacuum volume. The pressure of the vacuum system must be  $< 1 \times 10^{-2}$  Pa to prevent damage to the filament inside the instrument. Instruction manuals must be checked before using the RGA head to ensure safe and effective use.

## 12. Operation and Continued Monitoring

- All chambers should have a high vacuum gauge sensing within the chamber space. This includes all adjoining compartments that may be isolated by a valve.
- The chamber must have valves attached that allow complete vacuum isolation from the beam lines and other equipment.
- To protect high vacuum gauges against the effects of operating in low vacuum, high vacuum gauges should be automatically isolated if the vacuum pressure rises above  $1 \times 10^{-2}$  Pa.
- High vacuum gauges must be cleaned at minimum every 3 years or whenever a gauge develops instability as compared with adjacent gauge readings.
- Systems indicating higher than normally observed pressure must be isolated and repaired. A helium leak test is recommended if the leak location is not clear.
- To avoid undesired contamination, vacuum chambers should be vented with dry nitrogen or argon instead of air. This minimises water vapour and oil from depositing on the vessel walls.

# Chapter 2

# Design Criteria, Information and Philosophies for Vacuum Systems used in the Accelerator Area

#### Overview

In an accelerator system ion beams are transported from ion sources through to end stations (or detectors) through vacuum space maintained inside of beam tubes (lines). The relatively low number of gas molecules in the vacuum ensures a higher mean free path for the accelerated ions and hence less loss of ions through collision which is crucial especially for AMS measurements when counting a very small number of ions. A high vacuum also minimises the formation of plasmas in an electric field at high voltages which can avalanche into spark breakdown damaging sensitive equipment.

The vacuum systems used in the accelerator area are designed to be fail-safe to protect people and equipment. A very high proportion of problems with vacuum systems relate to, seals leaking on first use, being incorrectly mounted or failing due to aging 'o' rings. Other problems, however less frequent, include mechanical damage to sealing surfaces or fatigued parts such as bellows. The problems are all characterised by a poor vacuum or a previously steady vacuum that begins degrading slowly as observed during the operation of the vacuum system over its life time. The remaining problems are due to poor welds or vacuum fittings breaking. Poor welds are usually found during the initial helium leak test. They are remedied by either removing and re-welding the section or using a commercial vacuum epoxy. The choice is dependent on whether the weld is on a structural component or just a seal. Broken vacuum fittings are replaced or repaired.

Within the accelerator area. physicists work directly with technicians who apply broad knowledge and experience in vacuum technology to develop their concepts for use in the accelerator environment. This mix has been successful for 50 years. However not all designs are completed in the accelerator area. There are cases where commercial projects call for a more production based design or designs of chambers with complex noncylindrical shapes. These may go to the ANSTO drawing office for completion or a specialist vacuum design company.



Fig 1. ANSTO designed and developed beam lines on the STAR accelerator in building 22

It is not possible and often not necessary to standardise the design requirements for all components used in the accelerator area so the following information is intended to help designers of vacuum equipment to comply with accelerator specific and Australian standards. As of 2011, there are no known commercial workshops in Australia who specialise in the production of vacuum vessels. Local representatives from vacuum suppliers can provide access to overseas specialist workshops.

# 1. Terminology

**ANTARES** – Australian National Tandem for Applied RESearch. The HVEC FN Tandem accelerator in B53.

Backing – To pump on the exhaust (back) of a high vacuum pump

Beam line – The evacuated tubes that carry the ion beams from the ion sources to the end stations

**Cryopump** – Cryogenic vacuum pump. Uses a helium cooling interface to the vacuum space to freeze gases.

**Dependex** – A type of flange which is the standard flange used on ANTARES and ANSTO built beam lines.

**Fore line** – The vacuum line, tube, pipe, whatever between the high vacuum pump and the backing pump

High vacuum – See details in Chapter 3, para 1.3

ISO - International Standards Organisation

KF – Klein (small) Flange

LF – Large Flange

Low Vacuum – See details in Chapter 3, para 1.3

Outgassing - the liberation of gaseous vapours from the surfaces within a vacuum space

Pa – Unit of pressure, Pascal. 1 Pa = 1 N/m<sup>2</sup> = 0.01 mBar = 7.5 mTorr = 1.45 x 10<sup>-4</sup> PSI

**Pump down = Pump out** – evacuating gas from a vacuum space

**Roughing or Roughing-out** – Usually used to describe the initial transfer of gas from atmospheric pressure to low vacuum.

STAR – Small Tandem for Accelerator Research. The HVEE Tandetron accelerator in B22.

Turbo – an abbreviation for turbomolecular vacuum pump

**Ultra-high vacuum –** See details in Chapter 3, para 1.3

Venting – the equalisation of negative pressure to atmospheric pressure in vacuum equipment

Vessel – generic for a vacuum chamber where an instrument is mounted or interfaced into the vacuum space.

# 2. Standard Units

#### 2.1 General

Although metric standard is adopted across all ANSTO accelerator facilities it does not mean that all equipment is metric as the US built FN Tandem is mostly imperial, conversely, the Dutch built HVEE Tandetron is all metric. There is a combination of metric and imperial devices used throughout the facilities but designers must ensure that where practicable all new designs are metric.

#### 2.2 Threads

New equipment for use in the accelerator area, where practicable will be designed with metric standard threads.

• Contrary to the standard, on ANTARES there is a combination of threads in use. The accelerator high voltage generator and parts that have originated from the United States are

mostly imperial. Typically, UNC and some UNF. Most beam lines and endstations are metric but all Dependex and most Conflat flanges are imperial.

• ON STAR all threads are metric. ANSTO designed and built beam lines are metric except for the Dependex flanges which are UNC.

Care must be taken to identify threads and use the correct screws. Where practicable, metric threads are to be used other than on Dependex flanging.

#### 2.3 Pressure

All pressure indicating devices shall be in the SI unit, Pascal. Historically, Torr was used but this was replaced by Pa in the late 1980's. Most international accelerator labs use mbar. The conversion is as simple as multiplying mbar by 100 to get Pa. Pascal refers to a physical force of newtons per metre squared.

# 3. General Design Philosophies

There are many varieties of vacuum chambers and equipment in use in the accelerator area. Most have been designed and made at ANSTO. Examples include vacuum equipment measurement chambers where samples are measured, beam line transport components, ion source components or sensitive detectors, to name a few. Each has its own unique characteristics and vacuum design considerations.

In order to maintain a suitable mean free path for ion beams whether being transmitted from a source or scattered from a sample into a detection system, high to ultra-high vacuums are necessary. In a high vacuum the density of gas is low enough to minimise electron flow from surfaces at high voltages to surfaces at lesser potentials. It is therefore prudent to ensure designs of vacuum equipment that directly couple to the accelerator are designed for a minimum of high vacuum operation.

In every case the designers must ask the following questions:

- What shape? How big? Where will it fit? How will it be mounted? What level of vacuum?
- Will it be ridged enough and have low enough distortion to mount ion optical components?
- Is the design flexible enough to enable small scope changes in the future? Is it designed with consideration for reuse in another application when the project finishes?
- Does the design use vacuum technology best practises?
- Does it meet Australian and local standards where applicable?
- Is it a safe design?
- Is it achievable with resources available?
- Is it cost effective. Can we modify existing equipment to achieve a similar result?
- Does it have low environmental impact?

#### 3.1 The Shape

The first consideration is to define the actual use of the vacuum equipment relative to the experiment. Where possible cylindrical shapes should be considered first as they are stronger under vacuum and consequently being round, ports can be welded into the chamber aligned radially to the centre which ideally aligns instruments including detectors and cameras with the intersection point of the ion beam and the sample being measured.

Non-cylindrical chambers must be carefully designed to included adequate strengthen ribs to minimise distortion and hence misalignment of instruments.



Fig 2. A few examples of vacuum vessels used in the accelerator area

# 3.2 The size

The size is dictated by the space needed inside of the vessel or equipment in order to meet the operational demands. In the case of measurement chambers too big may mean problems with the distance between samples and detection systems and too small may mean manual handling of samples may be inhibited. Large chambers may have to be certified as per AS 4343-2005, Hazard Levels of Pressure Equipment in Australian Standard.

#### 3.3 Location

It will be an agreement between the accelerator user groups as to where equipment and beam lines will be located. This agreement will endeavour to minimise conflicts in beam line space, usage, accessibility and resourcing.

#### 3.4 Mounting

In general equipment must be secured onto the floor unless it is designed as mobile. An accelerator compliant beam line table should be used with standard beam line supports. Vibration must be identified during the design stage and decoupling stages provided.

Insertion bellows must be provided in long sections of beam lines to enable compression of the beam lines to facilitate dismantling and to ensure minimal strain on vacuum joints.

Designs must consider trip, bump and fall hazards. Structures that support platforms or heavy equipment that may otherwise fall must not be reliant on the structure supporting the vacuum chambers and beam lines. For example, ladders, walkways, etc.

- For ANTARES beam line vacuum equipment the beam axis alignment is nominally 1750 mm from floor height.
- For STAR beam line vacuum equipment the beam axis alignment is nominally 1193 mm from floor height.

## 3.5 Flexible design

It is prudent to ensure where practicable designs ensure some redundancy for feedthroughs, access ports and internal space. A major problem with early chambers was the lack of space allowed for inserting samples by hand or manually manipulating internal equipment and wiring. It is easier to design flexibility into the chamber or equipment at the design stage rather than having to dismantle it and have it reworked in the workshops later.

## 3.6 Can it be made?

When designing any equipment seek advice from a qualified machinist. Often designs include machining that may be very expensive or not possible. Early in the design phase ensure that the resources are available to service the vacuum equipment. It is recommended to consider the following early in the design stage:

- Power
- Cooling water
- Air cooling
- Signal lines
- Control lines
- Add-on electronic and mechanical instrumentation

## 3.7 Safe design

There are no cases that the author has been able to find of scientific vacuum chambers or equipment, like those used in accelerator facilities, failing and causing injury. Vacuum pressure is relatively low, -100 kPa, or about half the equivalent magnitude in a car tyre.

For safe designs the structure must meet the minimum dimensions as calculated in Appendix 1 - Structural Calculations for Scientific Vacuum Vessel Design.

Care must be taken with the design of the following:

- Glass view ports (most glass breakages are in the form of a crack which leak to atmospheric pressure quickly without the spread of shards)
- No internal sharp edges; in the case where a person must insert their hands blindly to access components.
- Venting limits pressure relief for positive pressure?
- Evacuation rate regulation; especially in the case where very thin material windows separate different pressure systems within a single vacuum space for example beryllium windows on x-ray detectors.
- Equipment mounts/stands; where an operator must climb up high to operate devices on the system
- If oil type vacuum pumps are used then mist filters must be fitted to the exhausts.

# **3.8** Cost effective design

All *bells and whistles* may not be necessary even after considering some design redundancy for future changes. Where possible, designs should consider integrating components that are available off the shelf. Designing what has already been designed by someone else is wasting time and effort.

It may be cheaper to modify an existing chamber for use rather than start from the beginning. Many chambers used around the accelerator area have successfully had one or more lives.

## **3.9** Design by numbers

This is a matter of ensuring structural integrity and being able to prove it. Likewise, designing the vacuum pumping system requires calculating the most appropriate size pump rather than going for the "biggest"!

There are formulas in Chapter 3 that allow must crucial calculations to be made from wall thicknesses to conductance. Good enough is not acceptable. Replication of previously designed equipment is acceptable.

#### **3.10** Australian Standards

Compliance is mandatory however there are few standards that are relevant to scientific vacuum vessels and equipment. The industry has developed its own standards for flange and seal designs which can be seen in the catalogues of most leading manufacturers of vacuum equipment. It is the responsibility of the designer to ensure compliance with all relevant Australian Standards. Chapter 1 discusses what standards are included.

#### 3.11 Environmental impact

Most vacuum systems used in the accelerator area today are designed to be oil free. That is, they do not use oils for lubrication of vacuum seals. There is however some oil vane and diffusion pumps still in use but the oils are responsibly disposed of through waste management at ANSTO. Some old diffusion pumps had been used with mercury as the pumping agent. Personnel must be cautious of using old diffusion pumps and their associated backing pumps as there may be residual mercury on surfaces and in the backing pump oil.

Solvents used for cleaning are used in small amounts and usually for wiping rather than immersing. Where baths are needed the solvents are reused when possible. The solvent bath used for degreasing, self recycles the solvent by design however there is a small loss of solvent over time into the atmosphere.

Where possible solvents used are selected with the environmental impact in mind.

#### 3.12 Manufacture

If ANSTO cannot provide the level of manufacture quality in-house then scientific vacuum equipment is best manufactured by companies that specialise in high tolerance machining and welding. A general metal fabricator will not suffice.

Attention must be paid to considering standard sized tooling during manufacture. Some companies will charge for the purchase of tooling that they consider to be non-standard.

It is imperative that the drawings for manufacture state all tolerances and qualities for the finished product.

On all design drawings, where the angle of the welded ports is crucial, ensure angular tolerances are clear and achievable. If necessary, specify the use of mandrels to hold ports in place during welding.

Some success has been made outsourcing the manufacture through vacuum companies to overseas manufacturers who specialise in high quality small runs.

#### 3.13 Testing

Testing the vacuum equipment or chamber is essential to establish compliance with the design and the all important ultimate base vacuum. Helium leak testing is the standard method used for chambers and most equipment however on some equipment in-service testing may be the only option. This is where add on vacuum equipment doesn't change the original base vacuum of the system it is connected to.

#### 3.14 Inspection

All parts should be cleaned prior to inspection. This doesn't need to be the final clean but enough to ensure all welds and surfaces can be clearly inspected. Welds must be checked for pits and alignment along the weld lines and seals must be checked for surface finishes. All crucial dimensional must be checked. It is recommended that any non-complying findings are photographed and documented in the project file. All non-complying parts should be reworked rather than *patched up*.

#### 3.15 Certification

As most vacuum vessels and equipment in the accelerator area fall within Hazard Level E of AS 4343-2005, certification is a local process. For large measurement vacuum vessels, results from the inspection and leak testing will be recorded on the engineering drawing and registered in the local accelerator register and/or ANSTO drawing register as appropriate.

#### 3.16 Documentation

For all large measurement chambers requiring any level of design calculations, design documents must be kept with project files.

#### 3.17 Maintaining

Vacuum chambers and beam lines do not require high levels of maintenance other than a general periodic clean and occasional seal replacement. Vacuum fittings and devices will have their own level of maintenance so access to these components must not be difficult.

The design must be easy to maintain especially moving parts, sliding or rotating seal and other parts in the vacuum space that may wear. Also vacuum pumping systems that may be mounted under or near the vacuum chambers. Generally any part that will require any level of routine maintenance must be accessible without the need for a major disassembly.

Vacuum pumping systems other than oil free systems, connected onto beam lines and chambers must include suitable oil traps between the backing and high vacuum pumps and where mist filters on all exhausts.

Some high vacuum gauges will require periodic cleaning. On ANTARES high vacuum cold cathode gauges are scheduled for cleaning every 12-18 months or max 3 years for difficult to service or less crucial instrument locations. Otherwise gauges will be cleaned on demand within the periods.

# 4. Performance and Operations

#### 4.1 Ultimate Vacuum

The ultimate vacuum is the maximum vacuum or lowest pressure the vacuum equipment typically reached after 3-5 days of uninterrupted pumping. This is a benchmark value which is used to assess the long term quality of the vacuum system. It is sometimes referred to as the *base vacuum or static vacuum*.

Generally, vacuums in the beam lines and chambers are well within the high vacuum range. Vacuums lower than this range will indicate a higher than normal gas load, a failing seal or a pump malfunction. On the accelerators the two main types of high vacuum pumps are turbomolecular and cryogenic pumps. For a system designed for high vacuum or better with no leaks and low outgassing then vacuums better than  $4 \times 10^{-6}$  Pa should be readily achieved.

The ultimate vacuum is improved by the correct choice of materials, seal types, high vacuum pumps used cleanliness and correct operation. All fittings must be designed or selected deliberately for high to ultrahigh vacuum use. Surface preparation and cleanliness will contribute to improved vacuums. A finger print can outgas for long periods of time. A bigger (higher speed, higher ultimate vacuum) vacuum pump does not mean a better vacuum!

#### 4.2 Working Vacuum

The working vacuum may be lower than the ultimate vacuum due to sample cycling (changing), introduced gas loads from adjoining systems or outgassing samples. A system must be designed to ensure the high vacuum pumping system has the optimum pumping speed and ultimate vacuum. Once a vacuum system reaches the "working vacuum" little operator intervention is necessary to maintain it but there are some important points regarding ongoing monitoring to ensure a failure is not immanent.

For sections of beam lines that are holding a static vacuum a cursory glance at the local vacuum gauge will indicate the stability of the vacuum. Generally, the (vacuum) pressure will change slightly throughout a day for example due to diurnal changes in ambient temperature and perceived changes due to ionisation gauges "wandering" as they become dirty.

In sections of beam lines that are in the vicinity of operating beam lines or near end stations the vacuum will be continually changing. Factors that influence these changes include increased gas loads from samples outgassing or surfaces that have just be exposed to high humidity, outgassing due to temperature changes within the vacuum space (including bake outs), outgassing from ion beams striking plastics, etc. Gas molecules don't have a preference to whether they travel towards or away from a pump during molecular flow so the source of gas can come from just about anywhere in the open system. These factors must be considered when monitoring vacuums throughout an entire system.

If a vacuum system's pressure rises slowly over many days or weeks if it is not related to the cryopump loading then it may be a seal failing. In this case a helium leak test is recommended.

A fundamental flaw in many vacuum system designs is the neglect for monitoring the backing pressure between the backing pumps and turbo pumps. Pirani gauges which are best suited for this role are relatively cheap and are the best diagnostic tool for determining overall vacuum system performance. The backing pressure should be consistent with changes in the high vacuum pressure. By monitoring the backing pressure over time, the base vacuum will be realised relative to the high vacuum so a pending failure of the backing pump can be determined, hopefully, well before it happens.

Care must be taken to maintain systems that have the potential to degrade over time for example an oil vane roughing pump will back stream oil mist that may accumulate in the high vacuum side of the system over time. This will contribute to lower vacuums due to higher gas loads from the partial pressures of contaminants.

#### 4.3 Sustainable Operation

Now that the vacuum system is designed, manufactured and commissioned for routine service, operating procedures have to be defined and adhered to, to maintain reliable trouble-free service. There are 3 processes that summarise the continuous cycle of operating a vacuum system:

- Pumping down from atmospheric pressure
- Using the working vacuum
- Venting to atmospheric pressure

The accelerator beam lines are divided into stand alone vacuum systems but in essence function as one. Each system can impact on a neighbouring system if things go wrong. In the case of end stations which cycle up and down in pressure more than any other devices an operator must be 100% sure of the pump down sequence in order to avoid damaging, pumps, samples, high voltage devices, detectors, etc. It is possible to shut down the accelerators as well. Most sections of the accelerator vacuum systems are interlocked to isolate a pressure surge to small area. Beam line vacuum systems are design so that both the vacuum pump and the beam line are isolated via gate valves. This is a typical arrangement for endstations as well.

The following must be read remembering that all 3 processes work in a cyclic fashion in a continuous loop.

#### 4.4 **Operational Protocols**

Key vacuum systems operate continuously, 24 hours per day, seven days per week. There are several vacuum systems that are cycled on and off as usage demands. All vacuum systems have one or more high vacuum gauges, some of which are interlocked to make the systems fail-safe. Any slow leaks or major catastrophic failures will close valves and isolate the system. In the few cases where no interlocks are used, a catastrophic failure will result in the high vacuum pump shutting down.

- For a <u>turbo pump</u>, as the pressure rises the gas load on the pump will cause the pump to draw more current to maintain it's speed. It will eventually fail when the current trip level is reached in the controller.
- For a <u>cryopump</u>, as the pressure rises the inflowing gas will condense or freeze onto the in the pump until it begins to warm up. Eventually there will be a thermal trip activated o the compressor which will shut is down. In both cases it is fail-safe.

As part of the accelerator's maintenance system, daily monitoring of all vacuum pressures is carried out. Where a pressure is seen to rise by a few percent without a particular reason, further monitoring will occur and corrective action started. Instantaneous failures are dealt with immediately. All observations and whether any maintenance carried out is noted on the maintenance log.

Where a vacuum system is isolated for service or where it has failed the appropriate tags are placed on the power leads.

Unattended out of hour's operation notification is not normally used as vacuum systems run continuously.

#### 4.5 Pump down

#### General

Pump down time is when most mistakes are made and significant time lost. Before a pump down begins the operator must ensure that all seals that were disturbed had been cleaned and replaced if damaged. The system must then be sealed ready for pump down and the roughing-out valve on the rough-out port, closed.

If a dry pump is to be used, such as a scroll pump, for rough-out then the tube is connected to the pumping port. If an oil vane rotary pump is used then ensure the cold trap has been filled beforehand.

#### Rough-out

Start the roughing pump and allow it to reach base vacuum. This is your first indicator of whether you have a leak in the system being pumped and when you have reached the pumping limit of the

pump. Slowly open the pump out valve on the system. This is where mistakes are made and damaged caused. Relatively speaking the volume in most sections of beam lines and endstations is small and the bulk of the gas can be removed very quickly. The problem is that a reduction in pressure too quickly can cause mechanical shock to fragile parts that must adjust slowly in order to minimise stress such as thin windows on detectors and detectors or devices that contain carbon foils.

Operators must always open roughing out valves slowly then gradually increasing as the gas load reduces. The change in the sound of the loaded pump is a very good indicator. A good design for more sensitive pump outs is to use a metering valve in line with the rough-out port to act as a regulating orifice.

#### Cryogenic Vacuum Pumps

For systems using cryopumps as the primary high vacuum pump it is essential to bring the base pressure down to the limit of the roughing pump before changing over pumping to the high vacuum pump. This reduces the gas load to be trapped (captured) on the cryo pump and extends the service life of the cryopump. Cryopumps are widely used on ANTARES as the primary high vacuum pumps but their limitation is that the warm up as they trap more gas causing a rise in base pressure. That is, when a cryopump is reaching saturation the temperature on the surface increases. More energy is required to hold the molecules on the surface and since it is almost fixed by design the temperature increases. A cryopump's effective pumping speed depends on the freezing and boiling points of the gases being pump relative to the cryopump's temperature. As the temperature increases more molecules leave the surface (boil away) raising the pressure. Some molecules are re-trapped as they hit the surface of the arrays and loose kinetic energy but they have a short residency time and soon add to the rising pressure in the system. Eventually the base pressure in the system becomes unusable so the pump must be regenerated.

#### Turbomolecular Vacuum Pumps

The other type of high vacuum pump widely used in the accelerator area is a turbo pump. Unlike a cryo pump that traps or captures gas molecules a turbo pump transfers the gas by conducting it almost one-way out of the vacuum space. The lowest roughing pressure before changing to a high vacuum pump is not as crucial when a turbo pump is being used. In saying this, the bulk of the gas must be removed before changing from roughing out to high vacuum pumping otherwise the vanes on the turbo pump may be overly stressed due to the pressure surge. A typical changeover pressure range is 5 - 50 Pa.

The optimum changeover time to minimise pump down times can be estimated with the assistance of the formulas found within the document. Letting the roughing pressure reduce to the limit of the roughing pump before changing over to the turbo pump is an inefficient method of system pumping. A turbo pump is more efficient at removing gas molecules as the flow moves towards molecular flow. Changing pumping to a turbo pump just before molecular flow is reached will increase the pumping speed and base vacuum will be reached sooner.

#### Pumping Speed

Each type of gas will pump away at a different speed depending on the mean velocity of the molecule which is a function of its mass. A hydrogen molecule of 0.02 kg/mol and mean velocity of 1762 m/s will reach a pump much quicker than say a Xenon atom of 0.13 kg/mol with a mean velocity of 217 m/s.

#### **Limitations**

In rare circumstances roughing pumps may be left pumping high vacuum systems by mistake. It is unlikely to damage either pump but the ultimate vacuum will not be reached as the roughing pumps are ineffective at conducting away molecules in molecular flow. Water vapour and residual solvents are difficult to remove from a vacuum space with *normal* vacuum pumping. Most low vacuum backing pumps provide gas ballast which can speed up the removal of water vapour and solvents that may otherwise condense inside of the pump during the compression stage. By introducing a small amount of air near the outlet of the compression stage the small increase in pressure helps carry water vapour and solvents to the exhaust before they can condense.

Some vacuum systems have been designed to allow chambers and beam lines to be heated. Heating causes contaminants to be removed from surfaces at lower than normal vacuums. The higher the temperature the faster the removal will be. Care must be taken to ensure heating doesn't inadvertently cause plastics and elastomers used within the vacuum space to outgas. Most bakeable systems use ceramics and metals only.

It is important that where vacuum systems are permanently connected to high voltage devices for roughing out, the insulated (plastic) pump out lines between the roughing pump and high voltage device must be brought up to atmospheric pressure before high voltages are reapplied. Otherwise the low molecular density of gas in the tube may ionise causing burning of the tube or even spark damage that can puncture the tube causing a leak.

# 4.6 Venting

#### General

Venting can cause damage to vacuum components just like pumping out a system from atmospheric pressure too fast.

#### Venting Speed

As discussed earlier the volume of vacuum systems on the accelerators is measured in litres to a several tens of litres. A catastrophic loss of vacuum will be more of a brief audible event rather than a dangerous mechanical failure. Little energy is required to change these systems by 100 kPa. There are a few devices in the vacuum space that may be damaged due to pumping out too fast but none that are considered a danger to personnel.

Other than venting too fast the second point to consider is the introduction of water vapour if venting with air. Key vacuum systems on the accelerators are vented with either dry nitrogen or argon both of which are delivered through a closed reticulation system from gas bottles. The choice of gas is dependent on molecular contamination within the system for example nitrogen is not preferred to vent the 846 ion source as nitrogen is an element that is known to bond with other elements causing molecular ion beams.

#### Regulation of Flow

There are many low flow regulators and rotameters (tubular flow meters) available that provide adequate control over venting. A simple in-house gas regulating system which works well is a tee piece connected between the venting port and the venting gas supply line. The centre port of the tee piece faces upwards and a ping pong ball sits over the hole with a cage over it so that it cant be blown away. Under the ball is an 'o' ring seal. Before the venting valve is opened the venting gas is allowed to flow. The pressure of the gas lifts the ping pong ball of its seal. The gas flow is adjusted depending on the height of the ball above the seal. The ball can only travel a centimetre or so. Once the flow is set the venting valve is opened and the now regulated gas flows into the vacuum space and the balls drops onto the seal stopping air from entering the vacuum space. As the pressure reaches equilibrium the ball begins to rise off the seal until eventually it rises to the original set height indicating the vacuum space is now at atmospheric pressure.

#### Precautions

- Before venting a vacuum system it is wise to isolate all ports and beam lines that must remain under vacuum. To save stressing a beryllium window on an x-ray detector due to the changes in pressure the system should be designed so that the x-ray detector can be isolated with a gate valve. The same logic can be applied to all equipment that may be effected. It is also important to ensure all power supplies and high voltages that may be hazardous to personnel or equipment are isolated prior to venting. This can be done either by administrative protocols or using a vacuum switch such as programmable relays in a vacuum gauge controller.
- Some vacuum gauge controllers use the signal from a Pirani gauge measuring at the same location as a high vacuum gauge to switch off the high vacuum gauge when the pressure becomes too high. This saves the gauge from ionising air when the system is at atmospheric pressure which will significantly reduce its life time.
- Another problem with venting too quickly is the disturbance of dust. Accelerator tubes must be vented slowly to minimise equalisation stresses and also the disturbance of dust. In this case a metering valve is used to limit the vent flow rate.
- Venting can cause loud high frequency noise so care must be taken to dampen the noise to a comfortable level by controlling the air admittance rate, or use hearing protection.
- Most beam line vacuum pumping systems are interlocked to isolate either the beam line or pump or both. In the event where an accidental venting occurs the gate valves will close minimising the effect on surrounding systems.

# 5. Safety

#### 5.1 General

Catastrophic failure causing injuries are not common and no reference is known from overseas accelerator facilities. This is almost certainly due to the low pressures involved in vacuums and the size and nature of the scientific apparatus. There are of course many failures reported for industrial sized tanks and equipment.

The vacuum systems are essentially fail-safe from absolute vacuum to atmospheric pressure. The worst case has been a number of beam lines and measurement chambers developing leaks and thin windows that have ruptured. In each case there has been a gradual rise in pressure to atmospheric pressure.

However, a few rules do apply.

- All vacuum systems must have one or more vacuum gauges attached with the exception to some static chambers used for storing moisture sensitive items or radiation detectors.
- High vacuum gauges must be switched off before vacuum systems are vented to atmospheric pressure.
- Only nitrogen, argon or other inert gas to be used for venting. No flammable gases to be used such as hydrogen.
- Turbomolecular pumps must not be vented from full speed. See manufacturer's recommendations.
- All designs must be consistent or better than the best practises outlined in this document. Inferior equipment must be isolated and reported to the Leader of Accelerator Operations.

- Cryopumps must not be operated without a serviceable pop off valve.
- Cold traps must be warmed and blown dry at least every 6 months to minimise  $O_2$  concentrating (and for de-icing as necessary).

# 5.2 Interlocks

Nearly all vacuum systems on the accelerators are interlocked via a set of integrated relays in a vacuum gauge controller, to protect equipment. Generally, if a rise in pressure is detected above a particular set level then the interlock relays will activate to close corresponding gate and line valves. This is designed to minimise the number of vacuums that must be regenerated in a fault. The interlock system also protects accelerator tubes from exposure to moisture and in the event of a catastrophic failure of the accelerator tubes; the vacuum system will close the main accelerator gate valves to trap SF6 gas.

If an interlocked failed causing a gate valve to stay open in the event of a pressure rise then the next interlock should activate the next gate valve. If the next interlock fails it is possible that the two interlocks share the same vacuum gauge controller which is at fault. In that case the next interlock should activate. If no interlocks activated there would be no increased risk of injury to personnel but all vacuum systems will require regeneration or a restart after the interlocks were repaired and tested.

## 5.3 Fumes from exhausts

There are a number of oil vane pumps in use on the accelerators. In the past, the exhaust on the pumps has released oil mist into the atmosphere inside the buildings. Now, on nearly all oil vane pumps in continual service there are mist filters attached or the exhaust is plumbed into an extraction system that vents the exhaust fumes outside of the building.

#### 5.4 Earthing

Some vacuum gauge controllers are prone to high voltage transient surge damage. These sometimes cause unnecessary isolation of systems requiring resetting. It is very important that all vacuum gauge controllers are well earthed.

# 6. Serviceability

#### 6.1 Access

Most beam lines with the exception to some measurement chambers are static volumes of vacuum requiring little operator intervention other than during preventative maintenance. Vacuum pumping systems do require a higher level of intervention during maintenance and operation. With these points in mind the designer must ensure that all controls and monitoring devices are localised at the working face of the beam line or chamber.

Due to beam lines being so high above floor level, working on them usually requires some form of ladder or steps. When designing beam lines, be sure to have pump out ports facing the passageway beside the beam line. The same applies to vacuum chambers and other large volume vessels.

Measurement chamber will need to have the access port accessible from a location near the control side of the chamber. A platform is recommended to allow easy reach and eay viewing into the ports.

#### 6.2 Maintenance

A well designed vacuum system will require routine maintenance including:

- Changing faulty seals cleaning seals that are regularly opened
- Cleaning vacuum gauges especially cold cathode type high vacuum gauges
- General cleaning inside of measurement chambers and equipment that is; regularly opened and touched, whenever it looks dirty, when the base vacuum degrades or when the surfaces are exposed to the atmosphere for long periods at a time (weeks).
- Servicing of vacuum pumps the group of the most routine maintenance tasks includes; cryopump regeneration, turbo pump bearings, scroll pump tip seals and oil vane pump oil changes to name a few
- Actuator service sliding shafts that penetrate into the vacuum space

#### 6.3 Cleaning

Cleaning is by far the most essential part of general maintenance of a vacuum system. Poor cleanliness will lead to poor vacuums and possibly sample contamination. It is a general philosophy that cleaning is done with solvents, wiped over and/or scrubbed and thoroughly dried.

#### **Solvents**

Typically ethanol, methanol and acetone are used. These solvents remove most greases and oils that are encountered. What ever solvent is chosen the most crucial point to remember is that it must not leave any residue.

For surface finishing see section on 'Surface Finishes'.

Chemical polishing is not commonly used in the accelerator area due to the size of the baths that are needed for large parts, the problem of cleaning or neutralising residual chemicals and not having a suitable facility for handling the types of chemicals used. Although once used electro-polishing is no longer encourages for the same reasons. If suitable facilities were available both chemical and electro-polishing are real options for very good cleans.

A general clean will consist of a number of different levels of cleaning. A single clean with the same solvent will not remove some oils and greases therefore a progressive approached is used using a variety of solvents and wiping materials. The following is a guide only of some such techniques.

Using a dry gas or filtered compressed air, most loose dust can be blown away. This is sometimes the best starting point for cleaning. A final blow down is also useful to ensure complete removal of lint.

The first clean will take away coarse particulates or chemicals. Ethanol dampened tissues if good for this level of cleaning. The ethanol helps remove some oils and greases and mixes with water to reduce the evaporation time. It is also good for mopping up dirt and dust. Scour pads (or 1200 wet or dry paper) moistened with ethanol is good for removing stubborn dirt. This must be continued until better than 95% of dirt, dust, oil and grease is removed. For stubborn dirt it may be necessary to use a petroleum based solvent followed by an ethanol rinse and wipe. Wiping is key to cleaning. The light mechanical brushing dislodges most dirt.

The second clean starts with ethanol but finishes with acetone. Only lint free wipes are used such as Kimwipes. At the end of this level of cleaning the equipment must look ready for use in the vacuum. That doesn't mean it is. It will more than likely have residues remaining on the surface.

Final clean. It is recommended that methanol is used for the final clean or another low residue solvent. The final clean must use lint free wipes dampened with methanol and lightly rubbed over the entire surface. An alternative final clean is to use the solvent bath and suspend the equipment in the hot vapour for a few minutes. Care must be taken with the bath as the hot vapour will degrease skin very quickly.

If a shiny (healthy looking) clean is desired then a metal polish such as Brasso can be used. Whenever Brasso is used the residue must be cleaned off with ethanol. Care must be taken not to get metal polish pastes into gaps that cannot be thoroughly cleaned out. Brasso is an enemy of vacuum, it traps water.

#### 6.4 leak detection

In the accelerator area, a leak is characterised by a flow of gas molecules into a space that is below atmospheric pressure. The pressure of the gas entering the space will restrict the vacuum system from reaching its design pressure which is typically in the high vacuum range. As a benchmark, if a vacuum is  $1 \times 10^{-5}$  Pa or more then disregarding the effects from outgassing, there may be a leak. In system designed for ultrahigh vacuum the benchmark will be in the order of  $1 \times 10^{-7}$  Pa.

Leak detection is usually only necessary for new chambers or equipment. It can be done offline or after the part has been mounted into a beam line. The most common method of leak detection is a helium leak test. Helium atoms being so small will find their way through openings that are too small for most other molecules. Generally, the smaller the leak, the less helium that can make its way along a leak path. The leak detector is usually a mass spectrometer tuned to have highest sensitivity for helium. The detector is connected directly to the device being tested. Any helium that makes its way through the leak will eventually be measured in the mass spectrometer. Residual Gas Analysers (RGA), although not specifically leak detectors, can be used for just that. Working on the same principle, mass spectrometry, the RGA can detect helium as well as other gases.

In some cases leaks can be too large for a helium leak test, that is, the amount of gas in the device being tested is too high diluting the helium to an ineffective percentage. Also, if the pressure is too high the leak detector cannot be opened to the device as the pressure gradient will be too high. Leak detectors work best from the higher end of the low vacuum range.

Often there are multiple leaks in systems. It may mean repairing large leaks in order to find small leaks. Where a leak is too large for the helium leak detector a few other methods can be used to locate leaks.

- Solvents have a very low viscosity and vaporise very quickly in air. Wetting surfaces with small amounts of acetone or ethanol can show small changes on Pirani and Penning gauges.
- Isolation of seals can, by process of elimination, restrict the testing area allowing large leaks to be isolated. For example, if a chamber with many ports and devices attached has a leak. It may be prudent to close all valves leading away from the chamber except for the leak detector valve then sequentially blank off the ports one at a time (using a sound flange and new seal) and test for a leak.

# 7. Soldering wires vs mechanical joints

It is preferable that wires in a vacuum space are joined together with a mechanical clamping type joint. The clamp must allow for gas to escape. Where a direct joint is necessary then soldering is permitted. Standard lead/zinc with resin core is okay provided that ALL flux is removed with ethanol. This may require some soaking in solvent with gentle agitation. Solder without resin core

can be used on stainless steel with a zinc chloride based eutectic flux, used sparingly. Ensure all flux is removed especially between the wire strands.

Be aware that in Faraday cups the continual flexing of wires can work harden the wire causing it to break. These are typically soldered joints. If soldering, be sure not to melt the insulation. It is highly recommended to replace normal plastic insulation with loose fitting PTFE (Teflon) spaghetti.

# 8. Flanges and seals

## 8.1 Common vacuum seals used in the accelerator area

A variety of standard vacuum seals are used on all vessels depending on the degree of vacuum to be achieved and the type of fitting that will be used. The types are widely used in the vacuum industry. ANTARES uses the Dependex style seals and flanges. This was developed for use with HVEC accelerators over 50 years ago. Other types in use include Conflat, ISO, and KF are used as well.

Туре	Vacuum level	Flange type	Seal type
KF	Low to High	Clamp (typically)	Viton 'o' ring, Al, PTFE
Edwards	Low	Threaded retaining nut	Viton 'o' ring
Dependex	High	Ring retained by spring clip	Viton 'o' ring supported by a ring
LF or ISO	High	Ring or clamps	Viton 'o' ring supported by a ring or in a groove
Conflat	Ultra high	Ring fixed or rotating	Copper ring, Square section 'o' ring
Specials	Various	Various	Aluminium or indium wire

Table 1. Common types of vacuum flanges and seals. Each type is available in multiple sizes

# 8.2 KF – Klein Flange

A common flange and seal used for general low vacuum applications due its ease of assembly and vacuum performance. It is common to have KF flanges and seals within the high vacuum side of a system. Vacuum performance can be enhanced by the use of aluminium or PTFE seals which directly replace the Viton 'o' ring.

Many general and specialised vacuum equipment is fitted with KF fittings as the range of adaptors from KF to other major types is readily available.





Fig 3. Assembly drawing for a typical KF flanges seal and a photo of the flange, seal and clamp
# 8.3 Edwards

This is no longer a preferred type that is used in the accelerator area however some equipment still uses this type.

One end of the fitting is shaped to retain an 'o' ring while the other end has an angled sealing surface that mates and crushes the 'o' ring to make a seal. They are connected by a threaded nut which when tightened squeezes the two halves together. It is normally for low vacuum applications.

# 8.4 Dependex

This was designed for use on HVEC Van der Graaff accelerators and beam lines. It was introduced to ANSTO in the early 1960's when the 3MV Van de Graaff operated in B22. Here, 1", 2" and 4" Dependex was used. When ANSTO purchased the FN Tandem nearly all fittings used were 4" Dependex (100 mm nominal). It is suitable for high vacuum use and often mated with Conflat flanges in systems that operate near ultra high vacuum.

The Dependex seal consists of a supported 'o' ring sealing between two opposing  $5^{\circ}$  metal faces. The 'o' ring is supported around its inner diameter by a ring to ensure the 'o' ring aligns with the centre of the 2 faces. The inner ring also sets the maximum crush on the 'o' ring that is the ring fits into a step on both halves of the Dependex seal keeping them separated at a fixed distance. Another ring which fits on the outer diameter of the 'o' ring does little more than keep the 'o' ring clean. The rings are often called *egg rings* due to their resemblance.

The Dependex flanges are held in place on a tube, or other fitting that is designed to accept Dependex, by a large circlip that fits into an outer groove a few mm from the end of the sealing face.

The flanges are normally drilled such that the holes alternate between being tapped or clearance. 1" and 2" Dependex use <sup>1</sup>/<sub>4</sub>" UNC, cap (socket or Allen) head screws and 4" (100 mm) Dependex use 5/16" UNC, cap (socket or Allen) head screws. Only use stainless steel screws (of one length) and washers. Discard plated steel screws as they are identified during maintenance. Washers are recommended as most flanges are aluminium alloy.

To complete the Dependex assembly, the flanges must be placed over the tube ends and then retained in place with a circlip. Secondly, the 'o' ring is stretched onto an inner ring and then an outer ring is placed around the assembly. The two Dependex ends are brought close together then the seal assembly is placed between them. The inner ring is aligned with the step on one side then the other tube is aligned and than pushed onto the ring. At this point it is important to ensure the 'o' ring assembly stays in place until the bolts are tightened. Screws should be finger-tightened and the flange faces must be parallel. Tighten screws in an opposite pattern. Some old flanges will have worn threads so screws may only be turned with the aid of an Allen key.

#### Problems

There are a number of things that can go wrong with assembling Dependex fittings.

- There have been cases where inner egg rings have been made too wide so that when fitted between the tubes the 'o' ring is prevented from crushing adequately to form a vacuum seal.
- Another problem with the inner rings is where some Dependex seals have been designed with too shallow steps so that a standard inner ring becomes too wide. In this case special inner rings have been made which are narrower. Personnel servicing non-standard joints must take care to reuse the right sized inner ring. A label should be placed on all non-standard joints identifying them.

- Some steps for inner rings have also been found to be out of tolerance preventing standard size inner rings from locating in the step. In this case some operators have cut a small section from the inner rings so that they can be squeezed together. Cutting inner rings like this is only recommended to solve this type of problem.
- Some flanges have been design for special applications with slightly different PCD's. These have unfortunately bee mixed up with standard stocks. As personnel who identify them (sometimes after a frustrating encounter) should put them aside and label them accordingly.
- Old flanges that have been over-tightened may have stretched threads. They are identifiable by the difficulty in starting screws. Either re-tap the threads or discard the flange. There have been case were tight threads have mislead personnel into thinking they have tightened the Dependex joint only to find leaks.
- Circlips can stretch especially those in over or under sized slots. They are easily identified by there distorted shape. Only use circlips that are flat when not under tension. A discerning technician should discard rusty circlips that cannot be successfully cleaned.
- Care must be taken to make sure the two faces of the Dependex joint are parallel before the joint is closed and bolted together. Failing to do this may cause a leak in the joint. It is acceptable if a bellows is used between sections that have misalignments.
- Twisted 'o' rings can cause leaks although small. There have been cases where a twist has caused the 'o' ring to bulge towards the outer ring making a less than adequate seal.

# 8.5 Special Flanges

These consist of flanges that have been designed to fit application where "off-the-shelf" flanges and seals cannot be used. They are typically based on common designs but modified to suit the application.

# Example

The Alphatross ion source uses two stainless steel flanges to hold the heater reservoir in place. Here the vacuum sealing surface is a flat surface about 8mm wide on each flange. The fixed flange is threaded in an imperial thread and the reservoir flange is open hole. The gasket used is aluminium wire which has been fused together to make a ring. The joint is similar in size to the wire. The wire is supported in place using about 3 to 4 strips of aluminium foil looped around the wire and sticky taped to the edges of the flange. The flange is tightened in a circular pattern to ensure maximum crush on the wire.

# Problems

- The wire is essentially unsupported so vacuum must not be applied until the wire is completely crushed.
- Bulbous joints in the aluminium wire dent the sealing faces on the flanges as they are tightened so they must not be used.

# 8.6 ISO or LF (Large Flange)

This is a European range of fittings designed for high vacuums and to suit most tube sizes used in the vacuum industry. There are two types of design which meet most light and heavy duty applications.

ISO-K flanges are a robust joint which are suitable for supporting larger and heavier items such as vacuum pumps and large gate valves. They are characterised by the outer clamping system which holds the two halves of the joint together.

ISO-F flanges are more of a traditional bolted flange type joint like Dependex. ISO-F and ISO-K can be joined together using a half clamp which fits onto a standard ISO-F flange. The half clamps then attach to the ISO-K flange.

Available in sizes from 63 mm nominal bore to 500 mm NB.



Fig 4. ISO family of Flanges

# 8.7 Conflat CF

Conflat is designed as an all metal vacuum seal for ultra high vacuum applications. A copper gasket is sandwiched between two thick stainless steel flanges that have a knife edge machined into the sealing face. As the flanges are tightened together the knife edges "bite" into the copper to produce an all metal seal with very low outgassing.

The term "ConFlat" is a registered trademark of Varian, Inc., so "CF" is commonly used by other flange manufacturers. Conflat sealed systems can reach vacuums as high as  $1 \times 10^{-11}$  Pa.

The copper gasket locates into a partially recessed in a groove in each flange to provide alignment prior to tightening. It also stops the gasket from moving during bake out. Conflat can be baked up to  $450^{\circ}$ C.

Most Conflat flanges have imperial sized bolts. The original style of bolt had a multi-face head that a ring spanner would fit over. This is to allow more purchase on the head from many angles. It is not uncommon now to find hexagon and socket head screws in use. Be aware that the steel must be a high tensile grade to ensure optimum force can be used to crush the gasket. It is essential that the flange faces are parallel during and on final tightening. A torque wrench may be used to facilitate uniform tightening. It is highly recommended that a molybdenum grease or graphite loaded grease is used on the threads.

Conflat flanges are available in sizes from 10 mm nominal bore to 250 mm NB. Sometimes nominal bore is written DN in front of the size, eg DN63.

Square section Viton 'o' rings may be used with Conflat flanges however this compromises the ultra high vacuum design and ultra high vacuums may not be reached.

# Problems

- Copper gasket not aligned in groove and edge is partially clamped on the bolt hole face of the flange. This lead to partial seal and most likely, a leak. Most Conflat flanges have two small grooves a little more than a millimetre wide machined into the bolt face. These are for locating special clips that hold the gasket in place while the flanges are brought into place. Just prior to tightening they clips are removed. They are sometimes mistakenly called pressure relieving grooves.
- Copper gaskets must only be used once. Unless the copper hasn't been fully compressed after the first use there will not be enough material for the knife edge to reform a seal.
- Due to the forces required for a knife edge to indent a copper gasket the flanges are made of stainless steel and quite thick. Large flanges are quite heavy and care must be taken not to drop them.
- Where Conflat flange bolts have been used more than once the threads in the flange and screws may have stretched. In this case, it is recommended that the same screws are reused in the stretched threads (with a little molybdenum grease) as they will have the same profile. Sometimes using new screws in a stretched thread will cause binding. Re-tap the flange if necessary.
- Stretched threads are possible where nuts and bolts are used through open holes so ensure the nuts go onto the bolt the same way they were removed. Alternatively replace the nuts and bolts outright.
- For ultra high vacuum ensure the gaskets are handled with gloves. Copper reacts well with the sweat in a fingerprint leading to a long outgassing process.

# 9. Bellows

Bellows consist of two styles, edge welded for ease of extension or compression and corrugated style which is has minimal movement. Each style is made from a thin stainless steel that has been electron beam welded. The ends are usually finished in a standard flange to suit the application.

# 9.1 Corrugated style

These are typically used for two purposes. Firstly, to allow sections of beam lines or equipment to be compressed to allow easy removal of sections without dragging the sealing surfaces against each other. Secondly, as bellows can distort and still maintain their structural integrity they are ideal to

join sections of beam line or equipment that have a deliberate mis-alignment. They can be purchased with almost any style of flange including specials. See example pictures.



Large bellows. ~300mm diameter. Stainless steel, wall thickness ~ 0.8 mm. This bellows is used to correct alignment offsets in the magnet box

Medium bellows. ~ 100 mm diameter. Stainless steel. wall thickness <0.5 mm. Used to allow sections of the beam line to be compressed to facilitate beam line component removal.



Small bellows. Electron beam edge welded stainless steel. Material thickness <0.25 mm. Used to isolate vibrations in the beam line.

Fig 5. Examples of bellows

#### 9.2 **Edge Welded Bellows**

These bellows are made up from very thin stainless steel discs stacked together and welded along their edges. The advantage of the design is the concertina style bellows that is very flexible allowing designs that have long extensions and short compressions. They have two main applications. Firstly, applications that requires a device to extend or contract into the vacuum space where an elastomeric seal on a sliding shaft is not adequate. Such as a sample manipulator or Faraday cup. Secondly, where a device such as a detector or sample positioner is sensitive to vibration and must be decoupled from the vibration source.

# 10. Valves

Along the axis of the beam lines gate valves are used to enable the isolation of sections for maintenance and also to minimise loss of vacuum in the whole accelerator if one section develops a leak. Most gate valves are electro-pneumatically operated and many are interlocked to local high vacuum gauges. The few that are manually operated are for maintenance isolations.

On ANTARES the electro-pneumatically operated valves are interlocked to close when the pressure, as measured near the gate valve, rises above 5 x  $10^{-5}$  Pa. This figure can vary depending on the location of the valve. See the Accelerator WIKI for values around the ANTARES. Once an interlock has tripped the switch that operates the gate valve is bypassed. To reset the valve:

- The vacuum must be within the high vacuum range as read on the corresponding vacuum gauge controller with a pressure better than the trip point.
- Once this level is reached the bypass button is held down momentarily.
- At this point the pressure will rise then fall as the gas load is pumped away.
- When the system pressure is less than the trip level the bypass switch can be let go.
- An indicator light near the switch will show its status.

#### Notes:

- Operators and technicians must be aware that gate valves must be orientated so that the gate always seals towards the vacuum pump. That is, they seal best in one direction only. Failure to orientate the gate the correct way may cause a vacuum leak when atmospheric pressure forces the gate off its seal.
- In some cases gate valves may remain sealed after they have been switched to open. This can be due to the withholding (sometimes called back pressure) pressure not being adequate enough to lift the gate off the seal.
- Gate valves, while mostly metal can become hot if a gate is closed in front of an ion beam. If left for long periods with high current beams the elastomer seals may warm up and begin to outgas.
- For roughing ports typically 25KF bellows sealed valves are used. On STAR these same valves are used to isolate vacuum gauges for cleaning.
- Where fine flow control is required for either roughing out or venting, metering valves are used. These are basically multi-turn needle valves for fine adjustment.
- Valves do require maintenance to keep the seals clean and in working order. After long periods of time the 'o' ring seals will deform and often will not recover their original shape.

There have been attempts to build fast acting gate valves for the purpose of isolating the accelerator tank's load of  $SF_6$  in the event of a breach in a tube. While it seemed like a good idea the force of the valve closing at high speed could damage the gate seal causing the valve to leak. Designers must use care to ensure that a standard high differential pressure rated gate valve isn't adequate before launching into these expense type valves.

# **11. External Interfaces**

# **11.1 Electrical Feedthroughs**

a) Low Voltage, Low Current

There are few applications where voltages greater than 24Vac are used within the vacuum space. For these voltages at a few amps coaxial feedthroughs such as BNC, SHV, MHV, etc, are often used in place of more expensive dedicated commercial types. In one application 200Vdc is used for faraday Cup suppressors but current is only a few micro-amps at most.

Notes:

• Do not use multi-pin signal feedthroughs to carry voltages unless the pins used for voltage can be a few unused pins away from used signal pins.

- Feedthroughs can be purchased is almost any flange style but are usually restricted to small size flanges.
- Insulation must be used on all wires carrying voltage. It is highly recommended to use single or stranded wires (preferably nickel coated) with loose fitting PTFE (Teflon) spaghetti.

# b) Signal

Signal feedthroughs other than coaxial such as BNC, SMC, Microdot, etc are from one to multiple pins. It is recommended that connections to multi-pin feedthroughs are by the use of slide on pins. Many are either sliver of gold plated for good electrical contact. Avoid soldering unless care is used to remove all traces of flux and no pin-holes in the solder have been created

On coaxial feedthroughs the coaxial connector will be either both sides of the feedthrough and hermetically sealed or with the coaxial connector on the atmospheric side and a plain pin on the vacuum side, also hermetically sealed. For either type, where coaxial cable must continue into the vacuum space it is recommended to remove the outer plastic insulation and replace it with loose fitting PTFE spaghetti. This is also recommended for all single or stranded wires carrying signals.

# c) High Voltage, High Current

There is a wide range of high voltage and high current feedthroughs available in most flange types. High voltage feedthroughs usually have a larger insulator in ceramic and sometimes glass. These must be kept clean on the atmospheric side. Long term breakdown on high voltage feedthroughs can damage the insulator causing vacuum leaks.

For high current applications such as heaters, dedicated high current feedthroughs must be used. These are characterised by larger cross section wires to carry the higher currents. Like HV feedthroughs they must be kept clean. It is recommended to clean the insulators on signal feedthroughs periodically to remove any accumulation of dust.

# d) Power

There are few feedthroughs designed for powering devices at mains voltages. It is highly recommended that experiments are designed not to have mains voltages in vacuum vessels or equipment due to safety concerns.

# **11.2** Mechanical Feedthroughs

# a) Rotary

Rotary feedthroughs allow the transition of rotary movement into the vacuum space to, for example, operate shutters or position samples or detectors. They are widely available in many of the common flange styles. There are three common types both of which are very good in high vacuums.

The first type is a continuous shaft between the atmospheric side and vacuum side using rubber vacuum seals in the form of either supported 'o' rings, square or odd shaped rings (like common shaft seals), and flat rubber sheet seals. These are best for general rotary actuation rather than continually rotating. Okay in high vacuum but care must be taken to keep the seals clean and periodically replaced or whenever wear is noticed.

The second type is a ferro-fluidic type which also uses a continuous shaft between the atmospheric side and vacuum but the seal is made by encapsulating a dense fluid around the shaft. These types usually have bearings to keep the shaft centred and free moving. They are best for high revolutions in high vacuum applications.

The third type is magnetically coupled rotary feedthroughs. These are expensive. They provide a seal by magnetically coupling the shaft on the atmospheric side to the shaft on the vacuum side across a continuous sealing plate. If either shaft is removed the vacuum is not compromised. These are excellent for ultrahigh vacuums and lower. They are for moderate speed revolving applications and general actuations.

# **b**) Sliding

Usually these feedthroughs are a round shaft that can slide in and out of the vacuum as well as rotate. Sliding feedthroughs are used for positioning devices or where a linear actuation is necessary. The length can vary from a few millimetres to hundreds. In either case there is a shaft which penetrates from the atmospheric side to the vacuum side. The type of seals used varies like in the rotary seal. Sometimes dual seals are used to minimise leakage especially in high actuation rate applications. In some applications the void between the two seals is pumped increasing the reliance of the seal. These types are good in high vacuums. Notes:

- Care must be taken to ensure the shaft is lubricated with a suitable vacuum oil or grease. It is imperative that the oil or grease on the shaft is changed regularly to minimise dust build.
- There are bellows sealed sliding seals available. These types do not rotate and can only move in and out a few tens of centimetres at most. Very good in high to ultrahigh vacuums.
- Some sliding seals are able to move radially which will affect alignment. Where alignment is crucial a linear bearing type is recommended.

# c) Wobbler

A wobbler feedthrough allows users to use a shaft as a lever or pick up. They are designed around a flexible rubber or bellows seal they allows a greater range of circular movement.

# **11.3 Manipulators**

These are a class of feedthroughs design for high to extremely high precision positioning of samples, detectors or other instruments. They can be purchased having all or either X, Y, Z and rotary movement. The X and Y axes can have various travel lengths however they are typically less than 100 mm each. The Z axis (vertical) can have greater lengths but is limited as with the X and Y axes by the physical limitations of the vacuum bellows.

# 11.4 Actuators

'Actuators', combine sliding and rotary feedthroughs with pneumatic, electro-pneumatic or mechanical actuators to do the work.

In the accelerator area, actuators can be found on Faraday Cups to insert the cup into the beam line on a bellows sealed sliding feedthrough and inside of the ANTARES pressure vessel to move the stripper tube in and out of the beam axis.

# 11.5 Load locks

Load locks are not common in the accelerator area but are ideal for moving samples into a high vacuum space without significant loss of vacuum pressure. A sample or device is mounted onto the end of the load lock shaft. It is inserted into a tube connected to a chamber but isolated from the chamber vacuum by an in-line gate valve. The tube is sealed and then evacuated to a vacuum pressure similar to the chamber. Once the pressures equalise the gate valve is opened and the shaft pushed into the vacuum space. From this point the sample or device can be removed from the shaft by other remote means.

# **12. Internal Devices**

There are many devices that are commonly used inside of vacuum systems. Not all are represented in this section however similar reasons are use to assess new and unusually devices for use in vacuum.

# 12.1 Lighting

Lighting is necessary in most sample measurement chambers where the operator must be able to see the sample in order to position it in front of the ion beam. In most cases a standard bare 12Vac halogen bulb is used with good results. Care must be taken to ensure the bulb can be dimmed as when running at maximum power the heat load can only be dissipated through the connecting wires. That is, there is little gas inside a vacuum to conduct head away from the bulb. Bright white LED's are also used with mixed success. They have less radial light output as compared with a bare halogen bulb. If the LED is setup correctly the light can be focussed towards the necessary target.

Preferably, wires must be stranded or solid wire with a loose Teflon sleeve but normal plastic coated hook up wire works in high vacuums. In some cases where the wires as not likely to come into contact with other components then they are used uninsulated. All wiring must be restrained so that it cannot move into the path of the ion beam. As a rule-of-thumb wires should be at least 20 mm from the beam.

A standard double BNC feedthrough can be used, that is, only the centre core connections are used. There are many off-the-shelf electrical feedthroughs available that will be more suited for this specific use.

# 12.2 Heating

Providing power to a heating stage on a sample (target) stick requires a high current feedthrough. If a feedthrough isn't provided from the supplier of the heating stage then many types can be found from larger vacuum suppliers. Ensure the feedthrough selected is designed to carry the maximum current that can be supplied and not the maximum current likely to be used. Like in the lighting section above, care must be taken to ensure wires are insulated and away from the beam path.

Heating stages rely on heat dissipation through the support for the stage which is usually made from stainless steel, a relatively poor conductor. When opening devices for handling be sure they are cool to touch first.

# 12.3 Cooling

Cooling stages come in 2 distinctly different types. The first is the Peltier cooled stage which is all electric. Powering the stage can be managed as for lighting and heating. The second type is a liquid nitrogen cooled system. It uses an insulated tube to carry LN2 through a feedthrough into the vacuum space. A typical design is where LN2 is pumped through a coil attached to a heavy copper braid. The braid is then connected to the cooling stage where heat is conducted away from the mounting stage. Thermal isolation of the cooling stage from the holder can be Teflon or sapphire standoffs or similar materials.

These cooling devices must only be switched on when the vacuum is at the working level otherwise condensation and possibly ice can form on their surface becoming a large outgassing problem. Likewise ensure the stage is close to room temperature before opening up the vacuum system to atmospheric pressure. Always vent with a dry gas.

# **13. Pumping**

# 13.1 General

It is not the intention of this document to describe the fundamental operation of particular vacuum pumps. Adequate information is available from manufacturer's catalogues. The following is information on what type of systems are used in the accelerator area and the reasons why.

Vacuum pumps don't suck. They act essentially as one way valves in a system where gas is flowing towards them. This is discussed in Chapter 3 of this document. There effectiveness is there ability to stop recoiling gas molecules returning into the vacuum space such as a beam line or vacuum chamber.

When considering what type of pump to use for a vacuum system the following points must be considered.

- What is the application?
- Will it have a relatively high gas load, that is;
- Will it be pumping a static gas load or changing gas load?
- Will there be a high outgassing rate?
- Are there known hidden/trapped voids to be pumped?
- It is a clean or dirty device? Is it contaminated with grease and oil?
- What pumping speed will be needed to maintain a high vacuum?
- What is the volume of the system? What is the conductance of the system? Will a small pump be just as effective as a large pump?
- What gases will be pumped?
- What type of pumps and how many are being used in the vicinity?
- Will the pumping speed need to be regulated such as for pumping near foils or delicate samples, detectors, etc?
- Cycling time whether it will be required to regularly pump from low vacuums
- Will the pumps operate reliably in their chosen application?
- Are they compatible with other pumps used on the accelerators? Are they cost effective?

Another consideration is vibration. Pump vibration can be significant leading to micrphonic interference in detectors and vibration of samples being measured. Vibration can be reduced by selecting pumps with less inherent vibration like turbomolecular pumps or the more fickle ion pumps. If a pump is a source of vibration it can be decoupled from the detector or measurement chamber by the use of a flexible bellows. An fine edge welded bellows is recommended.

# **13.2 High Vacuum Pumps**

See section 4.5, Pump Down characteristics for cryogenic and turbomolecular vacuum pumps.

# <u>Cryopumps</u>

For ANTARES the main beam line pumps in use are cryopumps. The types of pumps trap (or immobilise) gas molecules. They are used because they have a long lifetime between major

services, can be regenerated without removing them from service, have a high pumping speed for most gases and can maintain high vacuums for long periods in a static vacuum application.

#### Turbomolecular pumps

Where a vacuum system is cycled such as at a measurement chamber, then turbo pumps are used. Turbo pumps are transfer type pumps meaning, gas is transferred from the vacuum space into the atmosphere. A turbo pump can routinely recover a vacuum from low vacuum after a measurement chamber has been roughed out without needing regeneration as for a cryopump.

Turbo pumps are also more effective at pumping lighter molecules such as helium and hydrogen as compared with cryopumps. Using turbo pumps near systems that use these gases will reduce the effect of loading on cryopumps.

Most new turbo pumps can be serviced in-house. Some models purchased require special balancing of the bearings after they had been changed. In this case they are sent back to the manufacturer at great cost. It is important that models chosen can be maintained in-house.

#### Other high vacuum pumps

There have been few applications where titanium sublimation or ion pumps have been used in the accelerator area. Most have been stand alone UHV systems or cryostats for specialised applications. The practicality of cleaning and regenerating these gas trapping type vacuum pumps on systems requiring relatively frequent cycling is prohibitive.

Getter materials once used widely in electronics in tubes are becoming more used within larger static volumes such as beam lines and associated equipment. They can be coated onto surfaces to assist in maintaining uniform high vacuums in spaces that have poor pumping speeds.

# **13.3** Low Vacuum, Roughing or backing pumps

#### Oil free pumps

These pumps are categorised by either being an oil type pump or dry (oil free) pump. Oil free vacuum systems are of choice for accelerator systems now that the technology has been proven to be reliable and cost effective. The majority of roughing and backing pumps now in use are scroll pumps as well. Piston pumps have been trialled with little success due to the short life of the seals. Some applications have used diaphragm pumps and multistage roots pumps but scroll pumps remain the most cost effective and easiest to maintain option. The Edwards type scroll was chosen over other brands as this type of scroll pump is designed with the bearings mounted outside of the vacuum which in other brands often caused the bearings to fail from lubricant loss.

#### Oil vane pumps

Low Vacuum, Roughing or backing pumps are all the same type of pump. Oil vane type pumps were widely used in the accelerator area until 2006 when a systematic change began on the STAR accelerator to replace oil vane pumps with oil free scroll pumps. Some oil vane pumps remain in use where some back streaming oil vapour is not considered a problem. If an oil vane pump is to be used for roughing out purposes, it is high recommended that a liquid nitrogen cold trap is used to trap vapours. It is the ultimate aim to have oil free pumping on all accelerators in the future.

# **14. Monitoring**

# 14.1 Vacuum Gauges

The standard type of high vacuum gauge in use is the cold cathode penning type gauge.

- On ANTARES the Pfeiffer brand is in use in two versions; the standard cold cathode type and full range type which has an integrated Pirani gauge.
- On STAR the most widely used brand is Leybold.

Generally, vacuums are measured as an indication only for process controlling and establishing basic quality to ensure pressures are within levels that are accepted for "normal" operation. This varies depending on location and use of the vacuum system for example a measurement chamber is cycled often and affects surrounding vacuum systems. It will more than likely be a much high pressure than surrounding static systems.

In most cases, absolute vacuums are not required to be measured. However on some AMS beam lines where thin windows are used it is important to have accurate measurement of pressures when cycling pressures in the gas detectors. If accuracy is required it must be remembered that different gases ionise at different pressures and so pressures readings from the same volume will be different depending on the dominant background gas. Most gauges have characteristics related to Nitrogen (air).

# **14.2 Gauge Controllers**

On ANTARES the most widely used gauge controller is the Pfeiffer TPG300. This is an aging model and is being replaced in some key areas with the Pfeiffer Maxi-gauge which allows 6 heads to be connected at once.

The TPG300 has inputs for up to 4 heads whether Penning or Pirani. The limitation with the TPG300 is the electrical robustness when operated near the accelerator high voltage generator. Many failures have been attributed to high voltage transient surges. The TPG300 is now technologically aged and will ultimately be replaced outright. Various other models of TPG's are in use on ANTARES especially where single gauges are required. It is the TPG300 controllers on ANTARES that provides the interlocking capabilities.

The MAXI gauge is still being trialled as a replacement to the TPG300. A unit under test for many years at the HE end of the accelerator has already failed during an accelerator sparking event. Its real place may be away from the accelerator where the 6 heads allow consolidation of a whole beam line's vacuum monitoring system.

On STAR the common high vacuum gauge is the Leybold cold cathode type. It has been integrated into the STAR control system via cat 5 cable. They operate using a similar principle of measuring current flow across the plasma of the ionising gas. These gauges have been far more unreliable than the Pfeiffer equivalent. Unfortunately they are not interchangeable.

If venting with gas other than air or nitrogen be aware that the response on the Pirani gauges may show lower pressure than actual pressure.

# 14.3 Bourdon Gauges

Bourdon gauges are normally used for indicating that pressure is below atmospheric. They are not used on accelerator systems as they have poor accuracy in the range of interest i.e., below  $1 \times 10^{-3}$  Pa.

# Chapter 3



# **Overview of Basic Vacuum Technology**

The ANSTO accelerators consist of 140 metres of beam lines under vacuum. Supporting these beam lines are 60 individual high vacuum pumping stations. There are over 70 isolation zones along the beam lines to allow isolation of a zone without interrupting the adjacent zones. This facilitates isolations to repair leaking sections, routine maintenance and cycling of pressure in zones to change samples, etc.

The following information are the general tools used by technicians and engineers to design reliable and high performance vacuum systems for the accelerator facilities.

# 1. Vacuum Overview

# 1.1 What is vacuum?

A vacuum is defined as a diluted gas, or the corresponding state at which its pressure or density is lower than that of the ambient surrounding atmosphere.

Due to the Earth's gravity, atmospheric pressure varies with altitude. As a standard atmospheric pressure at sea level  $p_o = 101.325$  kPa. A Pascal is unit of vacuum relating to a force of newtons per metre squared.

An absolute vacuum of 0 kPa is not practically achievable so in real terms we can only approach zero for example on the ANSTO accelerators we have "operating" vacuums better than  $1 \times 10^{-4}$  Pa. In outer space the vacuum is less than  $10^{-12}$  Pa but not 0 Pa as there are some molecules of gas present which exert a very small partial pressure.

The pressure exerted from a vacuum lies between 0 to 100 kPa (see table below). In an ideal pumped vacuum system the pressure will continue to decease approaching 0 kPa. In accelerator systems the working ranges are medium to ultra high vacuums. In some special cases ion sources may operate in the low vacuum range but generally vacuum systems for the majority of accelerator systems are in the high vacuum range with some vacuum end stations close to ultra high vacuum.

# 1.2 Units used

The SI unit  $Pa = N/m^2$  or kg·m<sup>-1</sup>·s<sup>-2</sup>, will be used where any vacuum quantity is referenced. In the accelerator area the vacuum units used are in Pascals (Pa) and although the vacuum pressures are below atmospheric pressure "negative" pressures are not represented as -kPa but of a magnitude which approaches absolute zero using scientific notation, e.g. 1 x 10<sup>-6</sup> Pa.

#### **1.3** Vacuum categories

Vacuums used in accelerator applications vary in magnitude from low vacuum to ultra high vacuum. These categories describe the pressure ranges which in the vacuum world become references for vacuum quality in a given system. Low vacuum is managed differently from high and ultra high vacuums due to the physical limits of the system designs. Generally, a vacuum system must be designed with the desired ultimate vacuum in mind to ensure the most appropriate materials, seals, pumps and vacuum management are used.

The table below shows each of the ranges based on the European system. This is the closest to that used for the ANSTO accelerators. The shaded section represents the working ranges used throughout the accelerator for example, ion sources may have low to high vacuums, beam lines may have very high vacuums and some endstation vacuum chambers may use high to very high vacuums. This document describes the design factors and particular management controls for achieving vacuums in the various ranges.

Range	Pressure (Pa)
Atmospheric pressure	1.013 x 10 <sup>5</sup>
Low vacuum	$1 \times 10^5$ to $3 \times 10^3$
Medium vacuum	$3 \times 10^3$ to $1 \times 10^{-1}$
High vacuum	$1 \times 10^{-1}$ to 1 x $10^{-4}$
Very high vacuum	$1 \ge 10^{-4}$ to $1 \ge 10^{-7}$
Ultra high vacuum (UHV)	$1 \ge 10^{-7}$ to $1 \ge 10^{-10}$
Extremely high vacuum	< 1 x 10 <sup>-10</sup>
Outer Space	$< 1 \times 10^{-12}$
Perfect vacuum	0 Pa

Table 2. Reference pressure ranges (European scale).

A. Berman, Vacuum Engineering Calculations, Formulas, and Solved Exercises, Academic Press, Inc. (1992).

In the accelerator area:

Low Vacuum = atmospheric pressure to medium vacuum

High Vacuum = high vacuum to very high vacuum

Ultra high vacuum = all those equal to and higher than ultra high vacuum

# 2. Pressure Fundamentals

# 2.1 General

To understand the reason why vacuum systems must be carefully engineered to achieve high vacuums consideration must be given to how gases act in a closed chamber.

Any gas enclosed within a volume (a chamber or pipe work) is uniformly distributed or moving towards uniform distribution as in the case of accelerator tubes connected to stripper gas enclosures. The individual gas particles are constantly moving back and forth at high-speed within the volume; upon striking the vessel wall, they exert a force F on surface A due to pulse transmission. The pressure p that is exerted on the wall is defined as:

$$p = F/A$$

If the gas is made up of different types of gases, each of these gases will exert a pressure that corresponds to its concentration; this is called <u>partial pressure</u>. The sum of all partial pressures equals the total pressure. Air is a good example of this: In addition to its main constituents of nitrogen, oxygen and water vapour, air also contains many trace gases, the sum of which equal  $\sim$ 101.3 kPa, total pressure (atmospheric pressure). See table below.

In an accelerator system it would not be uncommon to have various carbon based compounds, sulphur hexafluoride and specific stripper gases present in trace amounts.

Gas	% by volume	Partial Pressure (Pa)		
Ar	0.92	934		
CO <sub>2</sub>	3.26 x 10 <sup>-2</sup>	33		
H <sub>2</sub>	4.9 x 10 <sup>-5</sup>	4.94 x 10-1		
He	5.16 x 10 <sup>-4</sup>	5.23 x 10-1		
Kr	1.1 x 10 <sup>-4</sup>	1.15 x 10-1		
$N_2$	77.17	78180		
Ne	1.8 x 10 <sup>-3</sup>	1.82		
O <sub>2</sub>	20.7	20970		
Xe	8.59 x 10 <sup>-6</sup>	8.7 x 10-3		
H <sub>2</sub> O	1.18	1200		
Other	remainder	remainder		
Total	100 %	101325 Pa		
Notes: H <sub>2</sub> O may vary depending on environment				
"Other" may include: $CH_4$ , $O_3$ , $N_2O$				

Table 3. Partial Pressures in AirThe Vacuum Technology Book – Pfeiffer Vacuum September 2008

# 2.2 Ideal (General) gas equation

1 mole of any gas at STP occupies 22.414 litres. At a temperature 273.15 K (0 °C) and a pressure of 101,325 Pa (standard pressure) 1 mole of any gas contains 6.02 x  $10^{23}$  particles. This is referred to

as Avogadro's number. The mass of the gas thus enclosed is its molecular weight in grams. The ideal gas equation describes the state of a gas as a function of pressure, temperature and volume.

$$pV = nRT$$

Example:

For a chamber, Ø300 mm x 500 mm long, a volume of 0.035 m<sup>3</sup>, calculate the mass of gas in the chamber at a vacuum of 1 x  $10^{-6}$  Pa.

pV = nRT

n = pV/RT  $n = 1 \times 10^{-6} \times 0.035 / 8.31451 \times 293 = 1.43 \times 10^{-11} \text{ moles}$ Mass of remaining volume = moles x mass = 1.43 x 10<sup>-11</sup> x 29 (air) = 0.415 ng No. of molecules = moles x Avogadro's Number = 1.43 x 10<sup>-11</sup> x 6.02 x 10<sup>23</sup> = 8.6 x 10<sup>12</sup> Per cm<sup>3</sup> = 8.6 x 10<sup>12</sup> / 35000 cm<sup>3</sup> = 246 x 10<sup>6</sup> molecules per cm<sup>3</sup>

Where:

 $p = \text{pressure (Pa)}, V = \text{volume (m}^3), n = moles, R = \text{general gas constant} = 8.314510 \text{ kJ/(kmol K)}, T = \text{thermodynamic temperature (K)}, Avogadro's number = 6.02 x 10^{23}$ 

# **3.** Flow Fundamentals

#### **3.1** Mean free path $(\lambda)$

The mean free path is the mean path length that a molecule traverses between two successive impacts with other molecules. High vacuums provide a longer mean free path than low vacuums. In accelerator systems high vacuums are crucial as ions that collide with gas molecules in beam lines loose energy and are not transported to the point of measurement. In the accelerator tubes high mean free paths are crucial to ensure low energy loss in the ion beam and optimised ion beam transmission.

Gas (at 0°C and 1 atm)	λ (nm)
Air	68
Ar	62.6
Cl <sub>2</sub>	27.4
CO	58.6
CO <sub>2</sub>	39
$H_2$	110.6
Не	173.6
Kr	36
$N_2$	58.8
Ne	124
02	63.3
Xe	26

Table 4. Mean free Paths for various gases

Hirschfelder, Curtiss and Bird (1954) Molecular Theory of Gases and Liquids, Wiley, New York

Pressure (Pa)	Molecules / cm <sup>3</sup>	Molecules / m <sup>3</sup>	Mean free path
101325	$2.7 \times 10^{19}$	$2.7 \times 10^{25}$	68 nm
	<b>—</b> 11 <b>—</b> 31	1	

Table 5. Other characteristics of Air

#### 3.2 Mean Velocity (u) m/s

The residency time of various gases in a system (or the time taken to remove various gases) relates to their individual mean velocity. The following table shows mean velocities for some gases. It can be seen that very light gases travel significant faster than heavy gases. It should be noted that Hydrogen and Helium do not pump efficiently in turbo pumps or cryopumps but heavy gases generally do.

Gas	Molar Mass kg/mol	Mean Velocity m/s	Mach Number
Air	0.028966	463	1.4
Ar	0.039948	394	1.2
Cl <sub>2</sub>	0.0709	296	
СО	0.028011	471	
CO <sub>2</sub>	0.04401	376	1.1
$H_2$	0.002016	1762	5.3
He	0.00402	1246	3.7
Kr	0.0838	272	
$N_2$	0.02801	471	1.4
Ne	0.020179	555	
O <sub>2</sub>	0.03199	441	
Xe	0.1313	217	
H <sub>2</sub> O	0.01802	587	1.8

Table 6. Mean Velocity for gases at 20°C

The Vacuum Technology Book – Pfeiffer Vacuum September 2008

$$u = \sqrt{\frac{8.R.T}{\pi.M}}$$

Where: *M* = Molar mass (kg/mol)

For example: Mean velocity of Argon @  $20^{\circ}$ C u =SQRT (8 x 8.31451 x 293 / 0.039948 x 3.1416) u =394 m/s

#### 3.3 Types of flow

The different flow phases need to be understood in order to select the appropriate pumping system for a particular application. It may be that in most cases a chamber or other piece of equipment is

pumped at the highest flow rate achievable with a given pump but it could be the case for example where a chamber has delicate films inside and gas movement past the foils has the potential to tear them apart. In this case a low steady flow rate is needed.

The flow phases in a vacuum system characterise the gas molecule movement related to pumping speed in a vacuum system. Factors that determine flow include pressure differentials, mean free paths, gas type, geometry, dimensions of the system and temperature. There are many different nomenclatures used to represent flow phases but 3 types in particular are used in the accelerator area, Turbulent, Laminar (both in Viscous phase), and Molecular. Less emphasis is placed on the intermediate or transitional flow as once this state is reached it is assumed that the system is pumping well. At atmospheric pressure up to about 100 Pa, the mean free path of the gas molecules is very small. Therefore, the gas flow is limited by the viscosity of the gas being pumped so the type of flow is called Viscous.

In an *ideal* system, if a roughing pump has a pumping speed of say 22 m<sup>3</sup>/hr and there is negligible flow resistance between the pump and the chamber then a chamber of  $\emptyset$ 300 mm ID x 500 long (a volume of 0.35 m<sup>3</sup>) at atmospheric pressure, will take approximately 1 minute to remove the bulk of the gas. During this time the flow remains in the [Viscous] Laminar and/or Turbulent phase.

Where a system has a leak, depending on the magnitude of the leak it is possible that the pumping will plateau at either the Turbulent, Laminar or Molecular flow phase. With outgassing systems the less referred to transitional state may be reached and with systems designed to reach ultra high vacuums outgassing may be occurring well into the molecular flow state.

Pumping	Flow Phase		Description	Type of vacuum	Flow rate Pa-l/sec
Pumping begins		Turbulent Flow	Initial flow experienced at start of pumping or in a low vacuum environment where the flow rate is high. Chaotic movement of molecules. Eddies and other non-parallel flows occur.	Low Vacuum	>1.01
	Viscous	Laminar Flow	Initial flow experienced at start of pumping or in a low vacuum environment where the flow rate is low. Gas molecules bouncing off other gas molecules (gas-gas) in the vacuum system. Short mean free path. Molecules streaming from system.	Medium Vacuum	10.1 to 1.01 x 10 <sup>-4</sup>
	Transit (Kn	ional Flow udsen)	Intermediate flow between laminar and molecular. Flow is dominated by both gas-gas and gas-wall collisions.	Medium to High Vacuum	1.01 x 10 <sup>-2</sup> to 1.01 x 10 <sup>-5</sup>
Base vacuum reached	Molec	ular Flow	Remaining gas molecules bounce off the walls (gas-wall) of the vacuum system and not each other. Long mean free paths. Flow rate very small.	High to Ultra-High Vacuum	<1.01 x 10 <sup>-5</sup>

Table 7. Vacuum flow phases

By evaluating the Knudsen (Kn) and Reynolds (Re) number, one can predict the various flow phases. These phases can be seen in the picture below which also shows how the gas molecules behave in the different flow phases moving through the opening d.

#### 3.4 Knudsen Number (Kn)

The Knudsen number is a dimensionless number defined as the ratio of the molecular mean free path length to a representative physical length scale. The Knudsen number in a viscous flow phase < 0.01 and molecule-molecule collisions dominate gas behaviour which behaves as a fluid. In molecular flow Kn > 1 and molecule-surface collisions dominate.

$$Kn = \frac{\lambda}{d}$$

 $\lambda$  = Mean free path

d = diameter of the opening that gas will pass through

#### 3.5 Reynolds Number (Re)

Reynolds number indicates whether the flow of a gas is absolutely steady (laminar flow) or on average steady but with small, unsteady changes (turbulent flow). The Reynolds number, Re, has no dimensions and is defined as the size of the flow.

Osborne Reynolds demonstrated in 1883 that the change from laminar to turbulent flow in a pipe occurs when the value of the Reynolds number exceeds 2,100. The exact value of Re for which the flow changes from laminar to turbulent depends on the geometry of the component, its surface roughness and other experimental factors. During evacuation of a vessel, turbulent flow normally occurs only for a short period of time at the beginning. It has been found to be approximately proportional to the root of the pressure gradient.

$$\operatorname{Re} = \left(\frac{\rho}{\eta}\right) d.v$$

 $\rho$  = gas density (kg/m<sup>3</sup>)  $\eta$  = viscosity (Pa.s) (Pascal-second) v = flow velocity (m/s) d = tube diameter (m)

Gas	Formula	Molecular weight	Gas Density [2] kg/m <sup>3</sup>	Viscosity η Pa.s
Air [1]	$N_2 + O_2$	29	1.1839	1.79 x 10 <sup>-5</sup>
Ammonia	NH <sub>3</sub>	17.031	0.7449	1.01 x 10 <sup>-5</sup>
Argon	Ar	39.948	1.723	2.24 x 10 <sup>-5</sup>
Carbon Dioxide	$CO_2$	44.01	1.9105	1.51 x 10 <sup>-5</sup>
Carbon Monoxide	CO	28.01	1.2082	1.74 x 10 <sup>-5</sup>
Chlorine	Cl <sub>2</sub>	70.906	3.1124	1.34 x 10 <sup>-5</sup>
Helium	He	4.02	0.171	1.99 x 10 <sup>-5</sup>
Hydrogen	$H_2$	2.016	0.0868	8.80 x 10 <sup>-5</sup>
Hydrochloric Acid	HCl	36.5	1.5844	1.46 x 10 <sup>-5</sup>
Hydrogen Sulphide	$H_2S$	34.076	1.4876	1.26 x 10 <sup>-5</sup>
iso-Butane	$C_4H_{10}$	58.12	2.3758	7.49 x 10 <sup>-5</sup>
Methane	$CH_4$	16.043	0.6556	1.11 x 10 <sup>-5</sup>
Nitrogen	$N_2$	28.02	1.2088	1.76 x 10 <sup>-5</sup>
Oxygen	$O_2$	32	1.381	2.02 x 10 <sup>-5</sup>
Propane	C <sub>3</sub> H <sub>8</sub>	44.09	1.8024	8.26 x 10 <sup>-5</sup>
Sulphur Hexafluoride	$SF_6$	146.5	6.27	$1.53 \times 10^{-5}$

Table 8. Gas density and viscosities

Unless otherwise indicated gases referenced from McGraw Hill Chemical Properties Handbook 1 atm 25°C. [1] Air STD Atmosphere at Sea Level: 1 atm 15°C [2] Gas Density Values Interpolated From 15°C Data using Charles Law

http://pipeng.com/index.php/gsts/itdmodflup002a/itddaflup00201

# 3.6 Viscous Flow

# Low vacuum, p = 100000 - 100 Pa, where $\lambda \ll d$

What characterises viscous flows, is frequent contact between gas molecules, yet less frequent contact with the walls of the vessel. The mean free path of the gas molecules is therefore very small and the gas flow is limited by the viscosity of the gas being pumped (Viscous Flow). In this case, the mean free path of the gas molecules is significantly shorter than the dimensions d of the vacuum equipment. For both Laminar and Turbulent Flow, Kn < 0.01. In addition, the term viscous flow is used if the product of pressure p and diameter d of the components through which gas is flowing is p.d  $\geq$  60 Pa.cm for air. Whether the flow is in the Laminar or Turbulent flows phases can be calculated using the Reynolds Number formula above.

# 3.7 Knudsen flow

# Medium vacuum, p = 100 - 0.1 Pa with $\lambda \le d$

If the Knudsen number is between 0.01 and 0.5, this is termed Knudsen flow. Because many process pressures are in the medium vacuum range, this type of flow occurs with corresponding frequency. Since this is a transitional flow, this range is transited relatively quickly when pumping down vacuum chambers and hence not usually considered in accelerator systems. This means that the influence of this conductivity on pump-down times is correspondingly low. A simple approximation for the Knudsen range can be obtained by adding the laminar and molecular conductivities. The

table in "Conductivities" below shows the conductivities of round, one meter long tubes of differing diameters in all three flow ranges.

# 3.8 Molecular flow High vacuum, $(p = 0.1 - 10^{-5} \text{ kPa})$ , where $\lambda > d$ and in UHV $(p < 10^{-5} \text{ kPa})$ , with $\lambda >> d$

At Knudsen numbers of Kn > 0.5 molecule-molecule collisions virtually no longer occur. What prevails is molecular flow. In this case, the product of pressure p and component diameter d is p x d  $\leq$  1.3 Pa.cm.



Fig 5. Molecular paths during different flow phases Re reference: http://www.engineersedge.com/fluid\_flow/pressure\_drop/pressure\_drop.htm

# **3.9 Pump throughput qpV**

The concept of pump throughput is of major significance in practice and should not be confused with the pumping speed! The pump throughput is the quantity of gas moved by the pump over a unit of time, expressed in Pa.1.s<sup>-1</sup>. Conversely, the pumping speed is the capacity of the pump to remove a volume of gas within a specific unit of time, measured in  $m^3/h$  or 1/s.

The throughput value is important in determining the size of the backing pump in relationship to the size of a high vacuum pump with which it is connected in series in order to ensure that the backing pump will be able to take away the gas moved by the high vacuum pump.

The pumping capacity (throughput) for a pump is equal either to the mass (m) flow through the pump intake port in a specified time:

$$q_m = \frac{m}{t}$$

Or to the pV (quantity of gas) flow through the pump's intake port:

$$q_{pV} = \frac{pV}{t}$$

It is normally specified in Pa.l.s<sup>-1</sup>. Here p is the pressure on the intake side of the pump. If p and V are constant at the intake side of the pump, the throughput of this pump can be expressed with the simple equation:

 $q_{pV} = p.S$ 

Where:

S = pumping speed of the pump at intake pressure of p.

#### 3.10 Speed of vacuum pump S

The speed of a vacuum pump is defined as

$$S = \frac{q_{pV}}{p}$$

p = Pressure at the pump inlet

However, pumps are usually connected to vacuum chambers via hoses resulting in a lower effective pumping speed  $S_{eff}$  at the chamber. If  $p_1$  is the pressure at the pump and  $p_2$  is the pressure in the vacuum chamber then:

$$q_{pV} = S.p_1 = S_{eff}.p_2$$

for continuous flow. Combining this with  $q_{pV} = C(p_1 - p_2)$  (see Conductance below) results in an expression for S<sub>eff</sub>

$$S_{eff} = \frac{S.C}{S+C}$$

#### **3.11** Conductance C

In a vacuum system, the volume that is pumped by a vacuum pump exhibits a level of flow resistance (Z) due to chamber and pipe sizes and their geometries, and the various sized orifices and other in-stream devices. The terminology used to describe this effect is the reciprocal of flow resistance which is *conductance* (C) or the systems ability to conduct gas.

Conductance is expressed in either l/s or sometimes  $m^3/h$  and is usually for steady, continuous flow through one or more components in a vacuum system. It is affected by the geometry of the piping element and relative to the flow phase and capacity of the pump used. In the high and ultrahigh vacuum ranges (molecular flows), C is a constant which is independent of pressure. In the rough and medium-high vacuums (turbulent and laminar) it is, by contrast, dependent on pressure. As a consequence, the calculation of C for the piping (beam line) elements must be carried out separately for the individual pressure ranges.

Flow resistance (sec/litre) 
$$Z = \frac{P_1 - P_2}{q_{_{PV}}}$$

Conductance (litre/sec) therefore

 $C = \frac{1}{Z} = \frac{q_{_{PV}}}{P_1 - P_2} \qquad (continued over)$ 

$$q_{pV} = C(p_1 - p_2)$$

Where:  $q_{pV}$  = Throughput flow rate p = Pressure (Pa)

At the molecular level, the conductivity of a system is reduced due to the friction of both the walls of the system and other molecules. As discussed above in "Types of Flow", these two affects occur at different flow phases. The total effect of conductance is the sum of the various conductance elements. In the case where a valve, a trap and an elbow in series each having a different conductance, the sum is:

$$\frac{1}{C_{total}} = \frac{1}{C_{valve}} + \frac{1}{C_{trap}} + \frac{1}{C_{elbow}}$$

If the components with different conductances are connected in the flow path in parallel, for example, 2 different diameter tubes connecting a chamber to a pump then the equation will be:

$$C_{_{Total}} = C_{_{tube1}} + C_{_{tube2}}$$

For the design of vacuum systems used on the accelerators conductance is mostly calculated for systems in molecular flow.



*Fig 6. Diagram for estimating pipe conductance Pupp/Hartmann, Vakuumtechnik, Grundlagen und Anwendugen, Hanser Verlag* 

# 4. Limitations

The most fundamental problems with vacuum systems are leaks and outgassing. Leaks can be categorised as either real leaks where gas enters the vacuum system from outside of the vessel or leaks form trapped voids which is in effect an internal leak. Trapped voids can contain gas that will continue to outgas for long periods reducing the ultimate vacuum that can be reached in a vessel. Real leaks can be found with the aid of a helium leak detector whereas internal leaks from trapped voids may never be found.

Outgassing can be minimised through careful selection of materials used within the vacuum space. The appendices have helpful tables to estimate outgassing rates. Outgassing may be in the form of desorption, diffusion or vaporisation. Permeation is not outgassing as the source of gas originates from outside of the vacuum space.

Examples commonly used substances in the accelerator systems that outgas,:

- Teflon, PVC, Ertalyte
- Viton, neoprene
- Copper, aluminium, stainless steel, brass, tantalum, rubidium, caesium, lithium, zinc
- Vacuum pump oil, vacuum greases, vacuum epoxies

These are just a few but it demonstrates that all materials will outgas at some point when the temperature and vacuum pressure reach their individual vapour pressure.

Neither of the problems is resolved by increasing the pumping speed (capacity) of the vacuum pump. There will be a point where the rate of gas entering the system will equalise with the pumping speed of the pump. The rate may vary depending on the vapour pressure of the substance outgassing and the temperature of that substance.



Fig 7. Limitation of Pumping From: http://www.mse.ncsu.edu/WideBandgaps/classes/MSE%20751/Handouts/HO02\_Leakage.pdf



Fig 8. Unwanted gas source wheel

# 4.1 Contamination

Vacuum chambers must be clean in order to reach the desired pressure as quickly as possible when they are pumped down. Typical contaminants include oil and grease on screws and seals, process reaction products or condensed vapours, particularly water that is adsorbed on the walls of the vessel. Consequently, it is necessary to ensure that the components are clean when installing vacuum equipment. All components attached in the vacuum chamber must be clean and grease-free. All seals must also be installed dry. If high or ultra high vacuum is to be generated, clean gloves must be worn during the assembly process.

Examples of common contaminants in accelerator vacuum systems

- Rotary pump oil
- Water
- Plasticisers from various plastics
- General airborne dust
- Machining oil
- Residual gases from stripper gases, venting gases, ion source gases
- Sample breakdown

# 4.2 Condensation and vaporisation

All substances can occur in a liquid, solid or gaseous state. Their aggregate status is a function of pressure and temperature. Liquids are transformed into their gaseous state through vaporisation, solids through sublimation. The separation of liquids or solids out of the gaseous phase is termed condensation. Since normal room air contains approximately 10g of water vapour per m<sup>3</sup>, condensed water vapour is always present on all surfaces.

Adsorption on surfaces is especially pronounced due to the strong polarity of the water molecules. Natural fibres, in particular, such as paper, contain large quantities of water that escape during drying processes under vacuum. Cooled condensers are used to separate the water vapour in this connection. Even some metals (Cd, Zn, Mg) can vaporise in noticeable quantity at temperatures of several 100 °C. Consequently, use of these metals is avoided in plant construction.

#### 4.3 Desorption

In addition to water, other substances (oil) can be adsorbed on surfaces. Substances can also diffuse out of the metal walls, which can be evidenced in the residual gas. In the case of particularly rigorous requirements, stainless steel vessels can be baked out under vacuum, thus driving the majority of the volatile components out of the metal walls.

Gas molecules, (primarily water) are bound to the interior surfaces of the vacuum chamber through adsorption and absorption, and gradually desorb again under vacuum. The desorption rate of the metal and glass surfaces in the vacuum system produces a gas yield that is a function of time, however. A good approximation can be obtained by assuming that after a given point in time  $t > t_0$  to the reduction will occur on a linear basis over time.  $t_0$  is typically assumed to be one hour.



Fig 9. Desorption curves



Fig 10. Adsorption Curves From: http://www.mse.ncsu.edu/WideBandgaps/classes/MSE%20751/Handouts/HO02\_Leakage.pdf

#### 4.4 Diffusion with desorption

At operation below  $10^{-4}$  kPa, desorption of plastic surfaces, particularly the seals, assumes greater significance. Plastics mainly give off the gases that are dissolved in these plastics, which first must diffuse on the surface.

Following extended pump downtimes, desorption from plastics can therefore dominate the metal surfaces. Although the surface areas of the seals are relatively small; the decrease in desorption rate over time occurs more slowly in the case of metal surfaces. As an approximation it can be assumed that the reduction over time will occur at the square root of the time.

The gas produced from plastic surfaces can thus be described as:

Desorption from plastic material  $Q_{diff} = q_{diff} \cdot A \sqrt{t_o/t}$ 

Where Ad denotes the surface area of the plastics in the vacuum chamber and  $q_{diff}$  denotes the surface area-specific desorption rate for the respective plastic. At even lower pressures, similar effects also occur with metals, from which hydrogen and carbon escape in the form of CO and CO<sub>2</sub> and can be seen in the residual gas spectrum.

#### 4.5 **Permeation and leaks**

For a gas passing through small holes in a thin wall in the Knudsen Flow regime, the number of molecules that pass through a hole is proportional to the pressure of the gas and inversely proportional to its molecular weight.

To Convert to Leakage	Multiply Helium Leak Rate by:			
Rate of:	Laminar Flow Molecular Fl			
Argon	0.88	0.316		
Air	1.08	0.374		
Nitrogen	1.12	0.374		
Water vapour	2.09	0.469		
Hydrogen	2.23	1.410		

Table 9. Conversation table for leak rates

Seals, and even metal walls, can be penetrated by small gas molecules, such as helium, through diffusion. Since this process is not a function of time, it results in a sustained increase in the desired ultimate pressure. The permeation gas flow is proportional to the pressure gradient  $p_0 / d$  (d = wall thickness,  $p_0 =$  atmospheric pressure = ambient pressure) and to the permeation constants for the various materials  $k_{perm}$ .

Permeation

 $k_{perm}$ .  $Q_{perm} = k_{perm}$ .  $A \cdot p_0/d$ 

Permeation first manifests itself at pressures below  $10^{-6}$  kPa.  $Q_1$ , denotes the leakage rate, i.e. a gas flow that enters the vacuum system through leaks at a volume of V. The leakage rate is defined as the pressure rise  $\Delta p$  over time  $\Delta t$ :

Leakage rate  $Q1 = (\Delta p \cdot V)/\Delta t$ 

If a vessel is continuously pumped out at a volume flow rate S, an equilibrium pressure  $p_{gl}$  will be produced. Throughput is equal to the leakage rate  $Q_l = S \cdot p_{gl}$ . A system is considered to be

adequately tight if the equilibrium pressure  $p_{gl}$  is approximately 10 % of the working pressure. If, for example, a working pressure of  $10^{-4}$  kPa is attained and the vacuum pump that is being used has a pumping speed of 100 I/s, the leakage rate should not be more than  $10^{-3}$  kPa I/s. This corresponds to a leak of approximately 20.20  $\mu$ m<sup>2</sup> in size. Leakage rates  $Q_l$  of less than  $10^{-6}$  kPa I/s can usually be easily attained in clean stainless steel vessels. The ultimate pressure achievable after a given period of time *t* primarily depends upon all of the effects described above and upon the pumping speed of the vacuum pump. The prerequisite is naturally that the ultimate pressure will be high relative to the base pressure of the vacuum pump.



Fig 11. Permeation of Different Gases From: http://www.mse.ncsu.edu/WideBandgaps/classes/MSE%20751/Handouts/HO02\_Leakage.pdf

# 4.6 Leaks

The source of leaks is as wide and varied as can be imagined. There is no such thing as a common leak however there are several types generally seen. The following lists types in rough order of prevalence:

- Dirty seals elastomer type seals with traces of dust, dirt and fibres on the surface
- Jarred seals ill fitted flanges where the seal lays across the sealing surface
- Vacuum pumping tubes where a fitting has been fitted without care and the pump cannot reach its full capability
- Distortion over tightened or misaligned flanges and fittings that "lift" seals off the sealing surface
- Stress cracks usually in places around flanges and fittings that have been incorrectly tightened
- Pressure dependant mostly related to accelerator tubes that develop leaky seals allowing insulation gas to enter when the pressure is greater than atmospheric pressure.

# 4.7 Bake-out

The following prerequisites must be satisfied in order to achieve lower pressures ( $<10^{-6}$  kPa):

- The base pressure of the vacuum pump should be a factor of 10 lower than the required ultimate pressure
- Stainless steel vacuum recipients and components must be used
- Metallic seals (CF flange connections) are required
- Leaks must be avoided and eliminated prior to activating the heater (use helium leak detectors!)
- Clean work is a must, i.e. all parts must be thoroughly cleaned and must be installed with grease-free gloves
- Pump and equipment must be baked out

Bake-out significantly increases desorption and diffusion rates, and this produces significantly shorter pumping times. Bake-out temperatures of up to 300°C are used. The instructions of the pump manufacturers relating to maximum bake-out temperatures and maximum permissible radiation levels in the pump flange must be observed.

Following installation the equipment is switched on, and after reaching a pressure of  $P < 10^{-3}$  kPa the heater is then switched on. During the heating process, all gauge heads must be operated and degassed at intervals of 10 hours. In the case of stainless steel vessels and the use of metallic seals, bake-out temperatures of 120°C and heating times of approximately 48 hours are sufficient for advancing into the pressure range of  $10^{-8}$  kPa. Bake-out should be continued until 100 times the expected ultimate pressure is attained. The heaters for the pump and vacuum chamber are then switched off. After cool-down, the desired ultimate pressure will probably be achieved. In connection with pressures  $P < 5 \times 10^{-8}$  kPa and large interior surface areas, it will be advantageous to use a titanium sublimation pump that pumps the hydrogen escaping from the metals at a high volume flow rate.

# 4.8 Residual gas spectrum

When leaks have been solved in a vacuum system and poor vacuum persists than a residual gas analysis (RGA) measurement should be made to determine the composition of the gas load. This will give vital information on where the vacuum problem may be. Interpreting the information will be trial and error as users will have to think about all possible sources of gases (and outgassing) that may be present. Also relative ratios of gases should be considered to make sense of the measurement. Care should be taken especially if making a measurement near a source such as a cryopump. Some gases that have been trapped on the cryopump may be liberating from the cold surface and will make up a part of the gas spectrum. Other places to take care are near the accelerator tubes and ion sources.

If developing an ultra high vacuum system it is important to know all sources of gas otherwise moving lower than  $1 \times 10^{-6}$  Pa will be an unnecessary challenge. For all other systems it is nice to know but not essential unless residual gases affect ion beam measurements. The dominate gases (and their masses) that may be seen include:

Gas	Mass	Gas	Mass
Water	18	Carbon Monoxide	28
fragment HO	17	Carbon Dioxide	44
Nitrogen	28	Argon	40
Oxygen	32	Sulphur Hexafluoride	146
Hydrogen	2	Helium	4

Table 10. Table of masses for common "in vacuum" gases

# **Appendix 1 – Structural Calculations for Scientific Vacuum Vessel Design**

Calculations for Vacuum Vessel wall and end plate thickness

The following formulae are from the Vacuum Society of Australia (VSA) training course notes which have been in circulation since the early 1980's. VSA continues to support these calculations for training vacuum technologists around Australia. The calculations provide conservative estimates of vacuum vessel wall thicknesses as compared with formulas used in ASME Section VIII – Division 1 UG-28 (Thickness of Shells and Tubes under External Pressure) and associated documents, which have been developed for larger industrial type vacuum vessels.

#### Cylindrical Vessel – Stainless Steel @ 20°C

Thickness of plate in a given diameter (h):

$$\frac{D}{h} \le 105, \ \frac{h}{D} \ge \frac{1}{105}$$

Where: D = diameterh = thickness

Maximum length of vessel for a given diameter (L<sub>c</sub>):

$$\frac{L_c}{D} \le 11.5$$

Where: D = diameter $L_c = \text{length}$ 

# **End Plates – Stainless Steel**

Thickness of plate in a given diameter (h<sub>1</sub>):

$$\frac{D_1}{h_1} \le 89$$
 ,  $\frac{h_1}{D_1} \ge \frac{1}{89}$ 

Where:  $D_1$  = diameter  $h_1$  = thickness

Minimum thickness for given deflection at centre:

 $\frac{h_1}{\delta} \ge 3$ 

Where:  $\delta$  = deflection  $h_1$  = thickness For Hemispherical End (h<sub>2</sub>):

$$\frac{R}{h_2} \le 830 \text{ requires } \frac{h_2}{R} \ge \frac{1}{830}$$

Where: R =radius  $h_2 =$  thickness

**Example 1:** To construct Stainless Steel vacuum chamber at  $20^{\circ}$  C as shown – Length L = 50 cm = 500 mm, D = 40 cm = 400 mm, Maximum allowable deflection at centre  $\delta = 1$  mm. Find  $h, h_1, h_2$  to satisfy vacuum chamber strength requirements.

# For Cylinder

1. 
$$\frac{D}{h} \le 105$$
,  $\frac{h}{D} \ge \frac{1}{105}$   $h \ge 400/105$ ,  $h \ge 3.8 \text{ mm}$   
2.  $\frac{L_c}{D} \le 11.5$ ,  $L_c \le 11.5 \times 400$   $L_c \le 4600 \text{ mm}$  with  $L = 500 \text{ mm} \checkmark$ 

#### **For End Plates**

1. 
$$\frac{D_1}{h_1} \le 89$$
,  $\frac{h_1}{D_1} \ge \frac{1}{89}$   $h_1 \ge 396.2/89$ ,  $h_1 \ge 4.45 \text{ mm}$   
2.  $\frac{h_1}{\delta} \ge 3$ , with  $\delta = 1 \text{ mm}$  then require  $h_1 \ge 3 \text{ mm}$ , however  $h_1 = 4.45 \text{ mm} \checkmark$ 

#### For Hemispherical End

1. 
$$\frac{R}{h_2} \le 830$$
 requires  $\frac{h_2}{R} \ge \frac{1}{830}$   $h_2 \ge 198.1/830 \ge 0.24$ mm

Dimension of cylindrical, planar and hemispherical parts of vacuum enclosures

	At	Cylinders		End Plates		Hemispherical
Material	Temp (°C)	D/h	$L_c/D$	$D_1/h_1$	$h_l/\delta$	<i>R/h</i> <sub>2</sub>
Copper	20	84	10	52	15	600
Copper	500	58	8.5	-	-	-
Nickel	20	100	11	73	8	780
Nickel	500	90	10.5	-	-	-
Aluminium Alloy	20	70	9	37	57	470
Aluminium Alloy	500	62	8.7	-	-	-
Stainless Steel (304)	20	105	11.5	89	3	830
Stainless Steel (304)	500	89	10.5	-	-	-
Glass (hard)	20	70	9	16	117	470
Neoprene	20	2.5	1.7	10	0.2	30
Teflon	20	12	3.8	14	9	-
PVC (Tygon)	-	3.7	2.1	-	-	-
Perspex	-	-	-	30	-	-
Mica	-	-	-	58	15	-

Factor tables for thickness calculations of common materials used in high vacuum systems

 $\delta$  = maximum permissible deflection at centre of plate L<sub>c</sub> = critical length for cylinder = 1.11D(D/h)1/2

**Example 2:** Using the ASME Boiler and Pressure Vessel Code (the Code) Section VIII, Division 1 for the safe design pressures for a vacuum vessel is used to calculate the maximum external pressure acceptable for a given wall thickness. The following example uses the same vessel dimensions as in Example 1, with a wall thickness of 3.8 mm as calculated using the VSA method.

The calculations reference the Vacuum Vessel Cylindrical Shell Thickness using paragraphs UG-28, UG-27 of ASME Boiler and Pressure Vessel Code.

Vessel shell thickness t = 3.8 mm  $D_o = 400$  mm (outer diameter of shell)  $D_o/t = 105.26$  Since this ratio is greater than 10, follow UG-28(c)(1) L = 500 mm (length of stiffened shell)  $L/D_o = 1.25$ E = 28 x 10<sup>6</sup> (modulus of elasticity for 304 stainless steel)

From Table G in Sec II, Subpart 3 to determine Factor A

A = 1 x 10<sup>-3</sup> (Factor A, from Table G of Sec. II, Subpart 3)  $P_{ext} = \frac{2AE}{3(\frac{D_o}{t})}$ 

 $P_ext = 177.33$  psi (max allowable working external pressure for given *t*) = 1222 kPa

A wall thickness of t = 3.8 mm results in a maximum allowable working external pressure of P\_ext = 325 kPa, which is greater than the external pressure that the vacuum vessel will see.

**Example 3:** For comparison, calculate the maximum external pressure as in Example 2 but with a wall thickness of 2.5 mm.

 $D_{o} = 400 \text{ mm}$ t = 2.5  $D_{o}/t = 160$ L = 500 L/D\_{o} = 1.25 E = 28 x 10<sup>6</sup> (modulus of elasticity for 304 stainless steel)

From Table G in Sec II, Subpart 3 to determine Factor A

A =  $5.3 \times 10^{-4}$  (Factor A, from Table G of Sec. II, Subpart 3)

$$P\_ext = \frac{2AE}{3(\frac{D_o}{t})}$$

P\_ext = 61 psi (max allowable working external pressure for given t of 2.5 mm) = 420 kPa

Therefore a wall thickness of 2.5 mm has a P\_ext of 420 kPa.

**Example 4:** This is a worked example for a large vacuum vessel from ASME Boiler and Pressure Vessel Code (the Code) Section VIII, Division 1, paragraph UG-28. It is in imperial units.

Calculate the maximum allowed external working pressure following UG-28. The minimum required thickness takes into account the support of the stiffening rings. Note that the actual vessel shell thickness t = 0.375-inch

 $D_o = 38.0$  inch (965 mm) (outer diameter of shell) t = 0.261 inch (6.63 mm) (minimum required thickness of shell for external pressure of 14.5-psia)  $D_o/t = 145.594$  Since this ratio is greater than 10, follow UG-28(c)(1) L 235.7 inch (6057.5 mm) (length of stiffened shell)  $L/D_o = 6.203$ E =29x10<sup>6</sup> psi (modulus of elasticity for SA-516 Gr 60 carbon steel)

From Table G in Sec II, Subpart 3 to determine Factor A A =  $1.108 \times 10^{-4}$  (Factor A, from Table G of Sec. II, Subpart 3)

$$P\_ext = \frac{2AE}{3(\frac{D_o}{t})}$$

P\_ext = 14.709 psi (maximum allowable working external pressure for given t) = 101.35 kPa

A wall thickness of t = 0.261 inch results in a maximum allowable working external pressure of P\_ext = 14.7 psia, which is greater than the external pressure that the vacuum vessel will see. Since the actual wall thickness is 0.375 inch (9.5 mm), the vessel design is adequate for the working external pressure.



Factor A table for Stainless Steels

# **Appendix 2 – Low Outgassing Specialist Non Metal Materials**

# • Celazole® PBI (PolyBenzImidazole) http://www.boedeker.com/celazo\_p.htm

Celazole® is the highest temperature-capable plastic available. However, it is very brittle (almost ceramic-like) and quite difficult to machine. That said, is it frequently used for bushings, bearings, rollers, and spacers in extreme environments. Its outgassing values are listed as 2.50% TML, 0.00% CVCM, 0.40% WVR.

# • Vespel® (Polyimide)

http://www2.dupont.com/Vespel/en\_US/assets/downloads/vespel\_gen/E61500.pdf

DuPont Vespel® SP-1 is one of the most-used high-temperature plastic materials used in applications where high-purity and electrical properties are needed. Vespel is frequently used in ultra-clean semiconductor and chemical applications. It is also one of the most expensive materials sold, but is flight-approved for NASA, USAF and other aerospace agencies. Its NASA outgassing values are listed as 1.09% TML, 0.00% CVCM, 0.40% WVR.

# • Duratron® XP (Polyimide)

http://www.portplastics.com/download/pdf/plastics/highPerformance/highPerformance26.pdf

Duratron® XP is the first real alternative to Vespel ... it was developed specifically to replace Vespel in extreme applications at a slightly lower price. It contains less than 1% metallic impurities as measured using the ICP-MS test standard. Duratron XP is ideal for use in high-energy gas plasma etch and strip processes. Outgassing values for Duratron XP are 0.75% TML, 0.00% CVCM, 0.49% WVR.

# • Torlon® 4203 unfilled PAI (PolyAmide-Imide) http://www.boedeker.com/torlon-4203.htm

Unfilled Torlon® 4203 has high dielectric properties and low thermal expansion, and is much less expensive than some advanced polymers. Torlon 4203 is typically used for insulators, spacers, and mechanical parts up to 520°F. Its outgassing values are listed as 1.85% TML, 0.00% CVCM and 0.49% WVR.

# • Torlon® 5530 glass-filled PAI (PolyAmide-Imide) http://www.boedeker.com/torlon-5530.htm

Torlon 5530 (30% glass-filled) is typically used for applications where dimensional stability over a wide temperature range is needed, as with temperature test sockets, nests, and fixtures. Its outgassing values are listed as 0.58% TML, 0.00% CVCM (% WVR is not shown). NOTE: Torlon's moisture absorption is a bit high, so critical dimensional stability can be an issue.

# • Semitron® ESd 500HR (filled PTFE)

http://www.portplastics.com/download/pdf/plastics/staticControl/staticControl18.pdf

Semitron® ESd 500HR is antistatic/conductive PTFE. This material is relatively clean, readily machinable, dissipates static electricity reliably ... as a result it is used in test handling equipment, fixtures, and other applications where static generation may cause failures and/or errors in production environments. This material has low outgassing values of 0.04% TML, 0.00% CVCM and 0.01 % WVR. PTFE has good mechanical properties up to approximately 500°F.

# • Neoflon® PCTFE (PolyChloroTetraFluoroEthylene)

http://www.aftonplastics.com/materials/pdfs/neoflan\_pctfe.pdf

PCTFE exhibits high chemical resistance, low and high temperature capability, resistance to most chemicals (including strong acids and bases), low friction, electrical and thermal insulation, and "slipperiness". PCTFE has the lowest outgassing values of any thermoplastic material we sell ... 0.01% TML, 0.00% CVCM, 0.00% WVR.

• **PEEK (PolyEtherEtherKetone)** http://www.dotmar.com.au/ketron-peek-1000/ketron-peek-1000-polyetheretherketone.html

PEEK is pure, easily machinable, chemically resistant, stable, and also has relatively low outgassing values (0.31% TML, 0.00% CVCM, 0.06% WVR). PEEK has good mechanical properties, but will not take temperatures over  $350^{\circ}$ F, so it may not have the mechanical or thermal performance needed.

• **Techtron® PPS (PolyPhenylene Sulfide)** http://www.dotmar.com.au/techtron-hpv-pps/techtron-hpv-pps.html

Techtron® PPS is easily machined to close tolerance, has excellent mechanical, thermal and chemical stability and has one of the lowest outgassing values of any thermoplastic material we offer (0.04% TML, 0.00% CVCM ... % WVR is not shown). Techtron PPS is generally a bit less expensive than PEEK or Torlon, but again, will not take as high temperatures.

• Ultem® PEI (PolyEtherImide) http://www.boedeker.com/ultem\_p.htm

Ultem<sup>®</sup> has good dielectric properties and low thermal expansion, and is considerably less expensive than some other polymers. PEI is also clean and stable, but is not particularly resistant to chemicals or solvents ... it has outgassing values of 0.40% TML, 0.00% CVCM and 0.06 % WVR. PEI has good mechanical properties up to approximately 410°F.

• Semitron® ESd 410C (filled PEI) http://www.boedeker.com/sem410\_p.htm

Semitron® ESd 410C is antistatic/conductive PEI. This material is relatively clean, readily machinable, dissipates static electricity reliably ... as a result it is used in test handling equipment, fixtures, and other applications where static generation may cause failures and/or errors in production environments. This material has outgassing values of 0.46% TML, 0.00% CVCM and 0.17 % WVR. PEI has good mechanical properties up to approximately 340°F.

# • Ertalyte® PET-P (Polyethylene Terephthalate) http://www.boedeker.com/ertaly\_p.htm

Ertalyte® offers the dimensional stability of acetal with the wear resistance of nylon. Ertalyte® PET-Polyester is clean, chemically resistant, stable, and also has relatively low outgassing values (0.13% TML, 0.00% CVCM ... % WVR is not shown). PET-P is considerably less expensive than most of the other materials listed above, but may not have the mechanical or thermal performance needed for all applications.

# • Semitron® ESd 225 (filled acetal) http://www.boedeker.com/sem225\_p.htm

Semitron® ESd 225 is antistatic/conductive acetal. This material is relatively clean, readily machinable, dissipates static electricity reliably ... as a result it is used in test handling equipment, fixtures, and other applications where static generation may cause failures and/or errors in production environments. This material has low outgassing values of 1.00% TML, 0.05% CVCM and 0.60 % WVR. Acetal has good mechanical properties up to approximately 180°F.
### Appendix 3 – Materials for use in Vacuum

The choice of materials is limited for vacuum systems to a range that has little impact on vacuum production and ultimate base vacuums.

#### 1.1 Metals

- <u>Stainless Steel</u> is used for the majority of vacuum system designs due to its:
  - o Durability
  - Surface finishing
  - Ability to withstand moderate temperatures for system bake out
  - Low magnetic susceptibility
  - Low thermal conductivity
  - Resistance to oxidisation
  - Low vapour pressure

Type 304 or 316 stainless steel is ideally suited for vacuum vessel construction because of its machining/welding characteristics, excellent corrosion resistance and overall cost effectiveness.

Not all stainless alloys are acceptable. Free-machining 303 steel contains sulphur, which tends to outgas. Alloys with good weldability using TIG or MIG welding are usually chosen.

- 304 or 316 stainless steel is a common choice of a stainless steel.
- $\circ~$  304L stainless steel, a low-carbon variant of 304 steel, is used for ultra-high vacuum systems.
- 347 stainless steel does not accept high polish.
- $\circ$  321 stainless steel is chosen when low magnetic permeability is needed.
- <u>Mild steel</u> is okay for low to moderate vacuums above 10<sup>-4</sup> Pa. Outgassing can be reduced with suitable plating such as nickel. It has high permeability to hydrogen and tendency to rust. Mild steel must not be used for any components or chambers used near the bending magnets
- <u>Aluminium alloys</u> are easily machined and have a low vapour pressure, unless the alloys contain high proportion of zinc. 6061 grade is good for general use. Components used in high vacuums or better must not be anodized, as the oxide layer can trap water vapour and outgas. Aluminium and its alloys have low strength at high temperatures, distort when being welded, and the copper-containing ones are poorly weldable. Aluminium wire rings can be used as cheap gaskets in demountable seals. Soft alloys must be used to ensure wire gaskets do not damage the seal faces. Aluminium has high thermal conductivity, good corrosion resistance, and low solubility of hydrogen. Loss of strength at high temperatures limits its use in bakeable applications, but aluminium is advantageous for large-size systems due to its lower weight and lower cost than stainless steel. Aluminium is not recommended for vacuum chambers.
- <u>Brass</u> is not acceptable for vacuum chambers however it may be suitable for some small beam line inserts for specific applications. Once used widely but copper can cause problems with neutron production if struck by proton beams of particular energies. Brass is for high thermal conduction applications in cooling baffles or sample mounts that may be heated. Although bare

brass has good corrosion resistance the zinc content may cause outgassing problems. This can be reduced by plating with nickel.

- <u>Nickel</u> is widely used in vacuum technology, e.g. as mechanical parts in vacuum tubes. It is relatively low-cost, can be spot welded, can be easily machined, has high melting point and is resistant to many corrosive fluids and atmospheres. Its potential drawback is its ferromagnetism, which restricts applications that would be influenced by magnetic fields.
- <u>Beryllium</u> is used primarily for x-ray windows.
- <u>Oxygen-free copper</u> is okay for high vacuum but it is difficult to outgas completely. Copper is insensitive to hydrogen and impermeable to hydrogen and helium, has low sensitivity to water vapour, but is attacked by mercury. Although, oxygen-free copper is widely used as it is easily machined and has good corrosion resistance. It is unsuitable for bakeable vacuum envelopes due to its tendency to oxidize and create scales. Conflat flange seals are made from copper. Its strength falls sharply above 200 °C. Its vapour pressure becomes significant at above 500 °C.
- <u>Indium</u> wire is used as a gasket in demountable seals. Not suitable for high temperature above  $\sim 100^{\circ}$ C applications.
- <u>Gold</u> wire is used as a gasket in demountable seals for ultra-high vacuum.
- <u>Tantalum</u> is acceptable in vacuum systems but it is difficult to work with due to its hardness. It is expensive so it is usually purchased in sheet form. Typically mounted as a shield where ion beams contact a surface as it has a low production of secondary electrons. Good for apertures and defining slit faces and anywhere that a metal edge is used to intersect the ion beam.
- <u>Zirconium</u> is corrosion-resistant. Like tantalum, it has low production of secondary electrons, so it is used as a shield of areas where reducing their production is important. It is used for neutron windows. It is expensive and rarekly used. Zirconium and zirconium hydride are used for gettering.

#### **1.2 Plastics**

Plastics or ceramics are not to be used as the primary vacuum chamber structure without engineering advice from ANSTO or a specialist scientific vacuum instrument company. Plastic is very good for electrical insulators, bushes or light weight components are necessary in vacuums.

- <u>Polytetrafluoroethylene</u>, PTFE or Teflon as it is commonly known is suitable for use inside of vacuum systems from low to ultra high vacuums. Being soft PTFE can flow (creep) so for applications such as sample positioning systems or other systems requiring high stability then other plastics may need to be considered. PTFE has be far the highest dielectric strength, for extruded PTFE, 19.7 kV/mm 60 173 kV/mm for an insulating film. This compares with air which is around 1 kV/mm. It is self-lubricating, tolerant to fairly high temperatures, and has low outgassing. It is not suitable for barrier between vacuum and atmosphere, due to its permeability.
- <u>Ertalyte</u> offers the dimensional stability of acetal with the wear resistance of nylon. It is stable with relatively low outgassing values and has very low water absorption. It is very good for small mechanical loads requiring electrical isolation in vacuum systems.

- <u>Polyvinyl Chloride (PVC)</u> is acceptable in vacuum systems but not where heat is above 50°C. It is usually an amorphous thermoplastic material with excellent chemical resistance and dielectric properties, good tensile, flexural and mechanical strength, low moisture absorption, exceptional dimensional stability and good flammability characteristics.
- Other plastics can be used in vacuum systems but must have low vapour pressure. Care must be taken to ensure they are not used in applications that may require temperatures that will cause high outgassing rates.
- <u>Perspex</u> (acrylic plastic) is another plastic which has good dielectric properties but a high vapour pressure especially at raised temperatures making it unsuitable in vacuums however it can be used in small amounts in a well pumped high vacuum system.
- <u>Vespel</u>, a polyimide, is very expensive, but machines well, has good electrical insulator properties and is compatible with ultra-high vacuum. It does however absorb moisture and requires a longer pump down time. It also performs well in extremely low cryogenic temperatures. Good for bushes or where a mechanically stable plastic is required.
- <u>Nylon</u> is self-lubricating but has high outgassing rate and relatively high water absorption.
- <u>Polycarbonates and polystyrene</u> are good electrical insulators with moderate outgassing.
- <u>Mylar</u> is used to make thin windows that allow ion beams to penetrate from vacuum to positive pressures with little energy loss. For example gas detectors mounted onto beam lines for AMS have gas inside of the detector separated from the beam line which is under vacuum. Ion beams pass through the window as they move from the vacuum side to the positive pressure side. Other materials in this category include Kapton which is rated for higher temperatures.

#### **1.3 Elastomers**

Some elastomers have sufficient vacuum properties and are used widely as vacuum seals in the form of 'o' rings.

- <u>Viton</u> is the standard seal used throughout the accelerator area as it is long lasting, low vapour pressure as compared with other elastomers and is bakeable to 200 °C.
- <u>Nitrile</u> rubber is used for vacuum seals. Does tend to break down over time. First signs of breakdown are small splits. If used for vacuum seals then they must be inspected regularly.
- <u>Natural</u> rubber is not typically used in vacuum systems other than inside of vacuum pumps usually on the low vacuum side of the system.
- <u>Silicone</u> rubber is not generally used as a seal in high vacuum environments. Silicone is soft when compared with other elastomers. It has a relatively low vapour pressure.

Further reading: Materials for high vacuum technology: an overview, S. Sgobba, CERN, Geneva, Switzerland. http://cdsweb.cern.ch/record/983744/files/p117.pdf

#### 1.4 Ceramics and glass

• <u>Alumina</u> ceramic based ceramics in vacuum systems perform well provided the ceramic is not porous which can trap gas bubbles. Ceramics are readily available bonded onto metal flanges or mounting plates. Ceramic can also be baked if necessary to speed up the outgassing of surfaces.

Glass is similar to ceramics in this application. It is very low vapour pressure but difficult to work with. Glass viewports can be purchased already mounted in a metal flange.

- <u>Borosilicate glass</u> is often used for smaller assemblies and for viewports.
- <u>Porcelain</u> ceramics, when fully vitrified and therefore non-porous, are excellent insulators usable to 1500 °C. it is generally commercially available bonded to metal in electrical insulators and other feedthroughs.
- <u>Mica</u>, although it is neither ceramic or glass it best fits in this category. Mica has been used in vacuum systems for both electrical and thermal insulation but because Mica is a series of laminations it contains trapped air which is not suitable in high to ultra-high vacuums.
- <u>Macor</u> is a machinable glass made by Corning Incorporated with similar properties to ceramics. It has a low vapour pressure and thermally stable up to 1000°C making it bakeable. It is made from mica and borosilicate glass. It is by far the most flexible of the glass-ceramic options.

 Leakage Testing Handbook, Prepared for Liquid Propulsion Section, Jet Propulsion Laboratory, National Aeronautics and Space Administration, Pasadena, California
 Nondestructive Testing Handbook, Volume One, Leaktesting, American Society for Nondestructive Testing.
 Leakage Testing Handbook, Revised Edition, July 1969, General Electric.
 Fluid Flow in Small Passages, Mars Hablanian, J.W.Marr, Varian

#### 1.5 Greases and oils

There are few acceptable greases and oils for use in vacuum systems. Users must ensure they purchase greases and oils specifically designed for use in vacuums. Typically they will have low vapour pressures, extremely low water absorption and easy to remove for cleaning.

- <u>Apiezon family</u> For further information http://www.apiezon.com/ . These have been used throughout the history of accelerators at ANSTO with excellent results. Wherever greases are used remember to use the minimalist amount.
- <u>Greases</u>
  - Apiezon H High Temperature Vacuum Grease, silicone and halogen free
  - Apiezon L Ultra high vacuum grease, silicone and halogen free
  - Apiezon M High vacuum grease, silicone and halogen free
  - Apiezon N Cryogenic high vacuum grease, silicone and halogen free
  - Apiezon T Medium temperature grease, silicone and halogen free
  - o Apiezon AP100 Ultra High Vacuum Lubricating Grease, silicone free
  - o Apiezon AP101 Anti seize Vacuum Grease, silicone free
- <u>Waxes</u>
  - Apiezon® Waxes and Compounds
  - Apiezon Wax W (Hard Vacuum Sealing)
  - Apiezon Wax W40 (Softer Vacuum Sealing)
  - Apiezon Wax W100 (Softest Vacuum Sealing)

- Apiezon Sealing Compound Q
- <u>Vacuum Oil</u> is used in few applications around the accelerator area. With a change to oil free pumps most oil consuming pumps are redundant. However these types of pumps may still in use in other applications. Oil vane roughing pumps and diffusion pumps were the largest consumers of oil. More recent brands of vane pumps were specifically tuned to oils of particular specifications so generic oils may not be compatible. Diff pumps are more forgiving and high quality generic synthetic oils may be used. The list below is a small sample of what is available and previously used.
  - $\circ\,$  Shell Vitrea 68 oil for roughing pumps. Check specification on pump to ensure compatibility with this mineral oil
  - Fomblin PFPE (Perfluorpolyether) oils designed for rotary pumps. Check specification on pump to ensure compatibility with this mineral oil
  - Apiezon® Diffusion Pump Fluids Apiezon AP 201 The Apiezon diffusion pump fluid is known for its exceptional higher degree of oxidation resistance.

### **Appendix 4 – Surface finishing**

In most cases a clean polished surface will yield the least outgassing as there are minimal surface traps for gas. Also, a polished surface is easier to clean and keep clean. Achieving a polished surface may not be practicable due to accessibility into tubes, chambers and small components but a combination of other methods may provide an adequate surface finish that will allow the desired vacuum level to be reached. The best method for a given surface will depend on the material, geometry and accessibility to its surfaces.

- <u>Polishing</u> A polished surface may be either smooth or uneven but the metal will lustre in either case. Polishing is a method of removing a layer of metal to expose clean metal underneath. This can be done with a mechanical buff or by hand with the aid of a polishing compound. Care must be taken to ensure all traces of the polishing compound are removed during final cleaning. All residues must be removed.
- <u>Grinding, sanding and other coarse to fine abrasive methods</u> These methods tear the surface dragging metal over metal. Some metal breaks away exposing clean metal underneath. The problem with this type of surface preparation is that the metal can fold over and not break away leading to voids of trapped gases. It is important that a fine grit is the last grit used to ensure larger metal drifts on the surface are torn away.
- <u>Grit blasting with grit or beads</u> This method propels grit or beads onto the metal surface causing fragments of metal to be removed with the impact. Like the abrasive techniques metal can be layered on metal (peening) if the grit is too coarse. Best to finish with a fine grit then a mechanical polish. Grit blasting can provide a more uniform surface finish and is very good for removing scale that may be present from rolling or extrusions.
- <u>Pickling</u> by far a very good way to clean welds and other heat induced decolourisations in the metal. It will remove a thin layer of metal, scale, oils, etc. Care must be taken to ensure any chemicals used are neutralised and thoroughly cleaned away. Residues cannot be tolerated. Makes general maintenance of the surface easier.
- <u>Passivating</u> a form of chemical polishing and surface protection. Can be stimulated with an electric current. Passivating is the spontaneous formation of a hard non-reactive surface film that inhibits further corrosion. It isn't a preferred method of surface preparation in vacuum systems as damaged surfaces can form micro cracks trapping water and other gases. For example, damage to anodised aluminium may allow aluminium oxide to form which can readily trap water. Passivation is not typically used for stainless steel as the surfaces are self healing, provided sufficient oxygen is available.

Further reading on surface passivation can be found at: http://www.euro-inox.org/pdf/map/Passivating\_Pickling\_EN.pdfGetter surfaces

• Nickel Plating – has been used on vacuum components to provide stability to the surface of normally oxidising metals such as steel, copper and brass. It is important to ensure that the nickel has uniform bonding to the parent metal. Cases have been observed where the plating has lifted creating voids in the vacuum space. Although nickel is resistant to corrosion it will form a thin layer if exposed to moist atmospheres. A periodic clean is recommended to minimise problems with corrosion.

Element	Melting Point °C	Critical Temp °C	Phase at Critical T
Aluminium	660	1124	Liquid
Americium	1176	896	Solid
Antimony	631	455	Solid
Arsenic	84	270	Liquid
Barium	729	574	Solid
Beryllium	1289	1102	Solid
Cadmium	321	226	Solid
Caesium	29	114	Liquid
Calcium	847	538	Solid
Californium	900	1402	Liquid
Cerium	798	1602	Liquid
Chromium	1863	1220	Solid
Cobalt	1492	1418	Solid
Copper	1084	1146	Liquid
Dysprosium	1412	1025	Solid
Erbium	1529	1139	Solid
Europium	822	547	Solid
Gadolinium	1312	1450	Liquid
Gallium	30	954	Liquid
Germanium	938	1260	Liquid
Gold	1064	1291	Liquid
Hafnium	2231	2255	Liquid
Holmium	1474	1146	Solid
indium	157	836	Liquid
Iridium	2447	1199	Solid
Iron	1538	1253	Solid
Lanthanum	918	1586	Liquid
Lead	328	622	Liquid
Lithium	181	465	Liquid
Lutetium	1663	1517	Solid
Magnesium	651	386	Solid
Manganese	1246	887	Solid
Mercury	-39	22	Liquid
Molybdenum	2623	2319	Solid
Neodymium	1021	1219	Liquid
Neptunium	639	1781	Liquid
Nickel	1455	1409	Solid
Niobium	2468	2501	Liquid
Osmium	3033	2721	Solid
Palladium	1555	1348	Solid
Platinum	1769	1800	Liquid
Plutonium	640	1506	Liquid
Potassium	64	165	Liquid
Praseodymium	931	1381	Liquid
Protactinium	1572	2636	Liquid
Rhenium	3186	2817	Solid
Rhodium	1963	1874	Solid
Rubidium	39	129	Liquid

# **Appendix 5 – Critical Vapour Pressures**

Substance	Vapour Pressure (Pa)	Temperature (°C)
Acetaldehyde	98.7	20
Acetone	24640	20
Butane	22	20
Carbonyl sulphide	1255	25
Ethanol	5.83	20
Ethylene glycol	0.5	20
Formaldehyde	435.7	20
Freon 113	37.9	20
Methanol	12800	20
Methyl isobutyl ketone	26.48	25
Nitrogen (N <sub>2</sub> )	63200	20
Oxygen (O <sub>2</sub> )	54200	20
Propane	2200	55
Propanol	2.4	20
Tungsten	0.1	3203
Water (H <sub>2</sub> O)	2.3	20
Xenon difluoride	0.6	25

# **Appendix 5 – Vapour Pressures continued**

Substance	Vapour Pressure (Pa)	Melting point (°C)
Apiezon Oil J	0.13 at 200°C 1.3 x 10 <sup>-4</sup> at 20°C	-
Apiezon Oil K	0.13 at 300°C 1.3 x 10 <sup>-7</sup> to 10 <sup>-8</sup> at 20°C	-
Apiezon Grease L	0.13 at 100°C 1.3 x 10 <sup>-7</sup> to 10 <sup>-9</sup> at 20°C	47
Apiezon Grease M	0.13 at 200°C 1.3 x 10 <sup>-5</sup> to 10 <sup>-6</sup> at 20°C	44
Apiezon Grease N	0.13 at 200°C 1.3 x 10 <sup>-6</sup> to 10 <sup>-7</sup> at 20°C	43
Apiezon Grease T	About 1.3 x 10 <sup>-6</sup> at 20°C	125
High Vacuum Grease Dow Corning	< 1 x 10 <sup>-4</sup> at 20°C	-
Nylon	~ 1 x 10 <sup>-3</sup> at 20°C	-
Teflon	< 1 x 10 <sup>-4</sup> at 20°C	-
Viton	1 x 10 <sup>-6</sup> at 20°C	-

# Appendix 6 – Outgassing tables for various materials Reference site: http://home.fnal.gov/~mlwong/outgas\_rev.htm

					i sampies.	
Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	(hours)	Test method	Reference	Year
Aluminium	None	1x10 <sup>-6</sup>	1h		Schamus (ref Markley, et al)	1999
Aluminium	Degassed	$1.7 \times 10^{-7}$	1h		Schmaus (ref Markley, et al)	1999
Aluminium	Degassed	2.7x10 <sup>-8</sup>	10h		Schmaus (ref Markley, et al)	1999
Aluminium 6061-T6	Baked 13.5h @ 300°C	1.4x10 <sup>-8</sup>	10h		Schmaus (ref Das)	1999
Aluminium	Cleaned	8x10 <sup>-9</sup>	10h		Schmaus (ref Blears, et al)	1999
Aluminium	Fresh, degreased w/ trichloroethylene & cleaned w/ ethyl alcohol	6.3x10 <sup>-9</sup>	1h	conductance	Elsey (ref Schram)	1975 (1963)
Aluminium	Degassed	$4.6 \times 10^{-9}$	100h		Schmaus (ref Markley, et al)	1999
Aluminium 6061-T6	Baked @ 200°C	4.5x10 <sup>-9</sup>	10h		Schmaus (ref Das)	1999
Aluminium	Degassed 24h, degreased w/ trichloroethylene & cleaned w/ ethyl alcohol	4.14x10 <sup>-9</sup>	1h	conductance	Elsey (ref Schram)	1975 (1963)
Aluminium 6061-T6	None	2.5x10 <sup>-9</sup>	10h		Schmaus (ref Das)	1999
Aluminium 5083-O	Bell jar, as received, room temp.	2.18x10 <sup>-9</sup>	10.3h	Rate-of-rise	Schrank, et al	1964
Aluminium 5083-O	Bell jar, as received, room temp.	1.27x10 <sup>-9</sup>	8h	Conductance	Schrank, et al	1964
Aluminium 5083-O	Bell jar, as received, room temp.	6.6x10 <sup>-10</sup>	24h	Conductance	Schrank, et al	1964
Aluminium 5083-O	Bell jar, as received, room temp. after baking @ 220°C	$4.6 \times 10^{-10}$	50h (baked @27-31h)	Conductance	Schrank, et al	1964
Aluminium 6061-T6	Baked 15h @ 300°C	$1.6 \times 10^{-10}$	10h		Schmaus (ref Das)	1999
Aluminium	Degassed 24h, degreased w/ trichloroethylene & cleaned w/ ethyl alcohol	3.06x10 <sup>-10</sup>	1h	conductance	Elsey (ref Schram)	1975 (1963)
Aluminium 5083-O	Bell jar, no. 4 finish (lapped), room temp.	$2.87 \times 10^{-10}$	6.0h	Rate-of-rise	Schrank, et al	1964
Aluminium	Fresh, degreased w/ trichloroethylene & cleaned w/ ethyl alcohol	6.0x10 <sup>-10</sup>	10h	conductance	Elsey (ref Schram)	1975 (1963)
Aluminium, type 1100	Cleaned w/ detergent, rinsed w/ acetone, pumped 24 hours	~10 <sup>-10</sup>	0	conductance	Young	1968
Aluminium	LEP vacuum chamber, chem clean, baked in situ @ 150°C	$2.3 \times 10^{-11}$	24h		Mathewson, et al	1988
Aluminium 6061-T4	Degreased in acetone w/ methanol rinse; baked 100°C	6x10 <sup>-12</sup>	24h	Rate-of-rise & conductance	Halama, Herrera	1976

Outgassing rates of aluminium Note the different methods of measurement and treatment of samples.

Aluminium 6061-T4	Degreased in acetone w/ methanol rinse, chem polished, baked 100°C	3x10 <sup>-12</sup>	24h	Rate-of-rise & conductance	Halama, Herrera	1976
Aluminium 6061-T4	Degreased in acetone w/ methanol rinse, chem polished, glow discharge in Ar, baked 100°C	5x10 <sup>-13</sup>	24h	Rate-of-rise & conductance	Halama, Herrera	1976
Aluminium, type 1100	Above plus baked 15h @ 250°C under vacuum	$4x10^{-13}$	24h @ room temp	conductance	Young	1968
Aluminium	PETRA vacuum chamber, glow discharge@145°C	$1 \times 10^{-13}$	Up to 200h	In situ glow discharge, conductance	Mathewson, et al	1977

# Outgassing rates of stainless steels, along with the surface treatment and pumping time.

Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	Time (hours)	Test method	Reference	Year
Stainless steel	None	6.4x10 <sup>-7</sup>	1h		Schamus (ref Markley, et al)	1999
Stainless steel	degreased	4x10 <sup>-7</sup>	1h		Schamus (ref Markley, et al)	1999
Stainless steel	None	2x10 <sup>-7</sup>	1h		Schamus (ref Blears, et al)	1999
Stainless steel	None	2x10 <sup>-8</sup>	10h		Schamus (ref Blears, et al)	1999
Stainless steel NS22S	Fresh	1.4x10 <sup>-8</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel ICN 472	fresh	1.3x10 <sup>-8</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel	None	1.2x10 <sup>-8</sup>	10h		Schamus (ref Das, et al)	1999
Stainless steel ICN 472	sanded	8.3x10 <sup>-9</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel NS22S	electro polished	4.3x10 <sup>-9</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel NS22S	mechanically polished	1.7x10 <sup>-9</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel	fresh	1.5x10 <sup>-9</sup>	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel ICN 472	Polished & vapor degreased	1.4x10 <sup>-9</sup>	10h		Schamus (ref Dayton, et al)	1999
Stainless steel	None	1.4x10 <sup>-9</sup>	10h		Schamus (ref Das, et al)	1999
Stainless steel NS22S	fresh	1.3x10 <sup>-9</sup>	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel ICN 472	sanded	1.0x10 <sup>-9</sup>	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel	None	7.6x10 <sup>-10</sup>	1h		Schamus (ref Das, et al)	1999
Stainless steel NS22S	mechanically polished	$4.6 \times 10^{-10}$	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel NS22S	electro polished	$4.3 \times 10^{-10}$	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel	Baked 24h @ 200°C	$1.5 \times 10^{-10}$	1h		Schamus (ref Das, et al)	1999

Stainless steel	None	$1.1 \mathrm{x} 10^{-10}$	100h		Schamus (ref Das, et al)	1999
Stainless steel 304	Degrease + water rinse	$4.0 \times 10^{-11}$	40h	Conductance	Strausser	1973
Stainless steel U15C	Baked 25h @ 300°C	$4.5 \times 10^{-12}$			Elsey (ref Calder, et al)	1975
Stainless steel 304	Degrease + water rinse, baked in vacuum 150°C for 12h	4.0x10 <sup>-12</sup>	5h after bakeout	Conductance	Strausser	1967
Stainless steel 304	Electropolished, baked 30h @ 250°C	$3.0 \times 10^{-12}$			Elsey (ref Young)	1975
Stainless steel U15C	Baked 45h @ 360°C	$2.6 \times 10^{-12}$			Elsey (ref Calder, et al)	1975
Stainless steel	Baked 24h @ 200°C	$9.3 \times 10^{-13}$	100h		Schamus (ref Das, et al)	1999
Stainless steel U15C	Baked 3h in vacuum @ 1000°C + 25h in situ @ 360°C	1.6x10 <sup>-14</sup>			Elsey (ref Calder, et al)	1975

Other cleaning methods although outgassing rates are not measured:

Material	Treatment	Results	Reference	Year
Aluminium	Quench a hot aluminium extrusion in an Ar- $O_2$ atmosphere	Dense, thin (~20Å thick) oxide layer	Sasaki, Y.T.	1990
Aluminium 6063	Clean with an alkaline detergent (Almeco 18)	Removes MgO, C, and Al <sub>2</sub> O <sub>3</sub>	Sasaki, Y.T.	1990
Aluminium	Vapour degreasing, pump for 120h before and after bake	Dominant residual gases: H <sub>2</sub> O, H <sub>2</sub> , CO, CO <sub>2</sub> , & CH <sub>4</sub> ; after baking @ 150°C and pumping, H <sub>2</sub> O & CH <sub>4</sub> gone, outgassing rates of other gases reduced 1 order of magnitude except H <sub>2</sub>	Mathewson, A.G., et al	1989
Aluminium	Vapour degreasing, then light alkaline etch (CERN basic procedure), pump for 120h before and after bake	Dominant residual gases: H <sub>2</sub> O, H <sub>2</sub> , CO, & CO <sub>2</sub> ; after baking @ 150°C and pumping, only H <sub>2</sub> exists	Mathewson A.G., et al	1989
Aluminium	Strong alkaline etching in NaOH, pump for 120h before and after bake	Dominant residual gases: H <sub>2</sub> O, H <sub>2</sub> , CO, CO <sub>2</sub> , & CH <sub>4</sub> ; after baking @ 150°C and pumping, only H <sub>2</sub> exists	Mathewson A.G., et al	1989
Aluminium	Vapour degreasing, Ar glow discharge @ 150°C	On initiation of discharge, large pressure increase (normal in Ar glow discharge) was too large for accurate measurement	Mathewson A.G., et al	1989
Aluminium	Vapour degreasing, then light alkaline etch (CERN basic procedure), Ar glow discharge @ 150°C	H <sub>2</sub> , CO dominant gases desorbing from surface during discharge cleaning	Mathewson A.G., et al	1989
Aluminium	Strong alkaline etching in NaOH, Ar glow discharge @ 150°C	H <sub>2</sub> , CO dominant gases desorbing from surface during discharge cleaning at order of magnitude less than light alkaline etch	Mathewson A.G., et al	1989
SS	H <sub>2</sub> glow discharge - 22°C	Dominant residual gases: $CH_4$ , $CO$ , & $C_2H_4$ ; water production insignificant; frequent arcing	Dylla, H.F.	1988

		on all exposed surface noted during initial discharge period		
SS	H <sub>2</sub> glow discharge – 150°C	Dominant residual gases: $CH_4$ , $CO$ , & $C_2H_4$ , and $H_2O$ ; frequent arcing on all exposed surface noted during initial discharge period	Dylla, H.F.	1988
SS	Ar glow discharge – ambient temp	Dominant residual gases that were significantly removed: $CO_2$ , $H_2$	Dylla, H.F.	1988
SS	Ar/O <sub>2</sub> glow discharge	Improved removal of CO and CO2; minor amts of implanted Ar that can be removed by baking @ 350°C	Dylla, H.F.	1988
	Ar/O <sub>2</sub> glow discharge 2h, exposed to air, then Ar/O <sub>2</sub> glow discharge 2h	Some degree of passivation: residual gases were removed with 1 <sup>st</sup> cleaning & did not show up during the 2 <sup>nd</sup> cleaning	Dylla, H.F.	1988
	O <sub>2</sub> glow discharge	Rapid removal of carbon, not of hydrocarbons (which can be baked out initially), increased surf. Oxidation, sputtering of the base metal, and possible enhanced outgassing of O <sub>2</sub> - containing molec.	Dylla, H.F.	1988

### Different outgassing measurements of epoxies

Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	Time (hours)	Test method	Reference	Year
NMA w/ solid alumina filler	Cured 300°F	$1.6 \times 10^{-7}$			Hanson, Patel	1970
Polyimide polymer	Thermosetting; measured @ 40°C	~1.1x10 <sup>-7</sup>	4h+9min	conductance	Kendall, Zabielski	1965
Polyimide polymer	Thermosetting; measured @ 155°C	~5x10 <sup>-8</sup>	4.5h increase temp + 6h	conductance	Kendall, Zabielski	1965
Silicone resin	Needs dicumyl peroxide catalyst; thermosetting, measured @ 35°C	~5x10 <sup>-8</sup>	4h	conductance	Kendall, Zabielski	1965
Araldite epoxies	Cured ~150°C for 15h	~10 <sup>-8</sup>	51h pumping	conductance	Barton, Govier	1965

# Kapton is a newer material and little data exists about its outgassing rate. Below are 3 references

Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	Time (hours)	Test method	Reference	Year
Kapton cable		1.0x10 <sup>-5</sup>	0h	conductance	Postma	1999
Kapton foil		$1.0 \times 10^{-7}$	40h		Ferro-Luzzi	1999
Polyimide	Bake @ 300°C	$4.0 \mathrm{x} 10^{-8}$	12h		Weston	1970
Kapton cable		$2.4 \times 10^{-8}$	168h	conductance	Postma	1999
Kapton cable		2.7x10 <sup>-9</sup>	336h	conductance	Postma	1999
Kapton cable		$6.5 \times 10^{-10}$	504h	conductance	Postma	1999

### Outgassing rates of several types of plastics

Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	Time (hours)	Test method	Reference	Year
Nylon		$1.2 \times 10^{-5}$	1h		Elsey (ref	1975
					Power, et al)	
Nylon		6.0x10 <sup>-6</sup>	1h		Elsey (ref	1975

					Power, et al)	
PVC	24h @ 95% relative	8.5x10 <sup>-7</sup>	1h	conductance	Elsey (ref	1975
	humidity				Santler)	
PTFE	Fresh	$1.7 \mathrm{x} 10^{-7}$	1h	conductance	Elsey (ref	1975
					Santler)	
Teflon		6.5x10 <sup>-8</sup>	1h	conductance	Elsey (ref	1975
					Santler)	
PTFE	Fresh	3.3x10 <sup>-8</sup>	10h	conductance	Elsey (ref	1975
					Santler)	
Teflon		$2.5 \times 10^{-8}$	10h	conductance	Elsey (ref	1975
					Santler)	
PVC	24h @ 95% relative	$2.0 \times 10^{-8}$	10h	conductance	Elsey (ref	1975
	humidity				Santler)	
G-10		~10 <sup>-8</sup>			Beams	2001
					Division -	
					FNAL	

Reference to the following has been lost but it is believed to have originated in an early Vacuum Catalogue from the 1990's

 $K_1$  is the outgassing rate (air equivalent) after 1 hour pumping  $\alpha_1$  is the slope of the (log K - log t) curve at 1 hour

Material	K <sub>1</sub> Torr litre/sec cm <sup>2</sup> (x 10 <sup>-10</sup> )	α1	K <sub>10</sub> Torr litre/sec cm <sup>2</sup> (x 10 <sup>-10</sup> )	a <sub>10</sub>
Aluminium Alloy (fresh)	63	1	6.0	1
Aluminium Alloy (degassed 24 hours)	41.4	3.2	3.06	0.9
Aluminium Alloy (3 hours in air)	65.5	1.9	4.75	0.9
Aluminium Alloy (anodised -2µ pores)	2760	0.9	322	0.9
Aluminium Alloy (bright rolled)	-	-	75	1
Duralumin	1700	0.75	350	0.75
Brass (wave guide)	4000	2.0	100	1.2
Copper (fresh)	400	1	41.5	1
Copper (mechanical polish)	35	1	3.56	1
OFHC Copper (fresh)	118	1.3	12.6	1.3
OFHC Copper (mechanical polish)	19	1.1	1.63	1.1
Gold (fresh wire)	1580	2.1	5.1	1
Mild steel	5400	1	500	1
Mild steel (slightly rusty)	6000	3.1	130	1
Cr plated steel (fresh)	70.5	1	6.8	1
Cr plated steel (polished)	91	1	8.0	1
Ni plated steel (fresh)	42.4	0.9	4.84	0.9
Ni plated steel	27.6	1.1	2.33	1.1
Chemically Ni plated steel (fresh)	83	1	7.05	1
Chemically Ni plated steel (polished)	52.2	1	4.6	1
• • • • •				

Araldite (moulded)	116	0.8	35.2	0.8
Araldite D	800	0.8	220	0.78
Araldite D	190	0.3	125	0.5
Araldite F	150	0.5	73	0.5
Celluloid	860	0.5	430	0.5
(PTFE) (fresh)	16.6	0.8	3.31	0.9
Kel-F Oak ridge	4	0.57	1.7	0.53
Methyl methacrylate	420	0.9	140	0.57
Mylar V-200 (24 hr at 95% RH)	230	0.75	40	-
Nylon	1200	0.5	600	0.5
Pertinax	620	0.18	290	0.5
Perspex	72	0.44	27	0.44
Perspex	310	0.4	180	0.4
Polyamid	460	0.5	230	0.5
Polyester - glass laminate	250	0.84	80	0.81
Polyethylene	23	0.5	11.5	0.5
Polystyrene	2000	1.6	200	1.6
Polystyrol	56	0.6	12	0.61
Polyvinylcarbazol	160	0.5	80	0.5
PTFE	30	0.45	15	0.56
P.V.C. (24 hr at 95% RH)	85	1.00	2	-
Teflon	6.5	0.6	2.5	0.2
Terephenil (fresh)	62.2	0.5	16.8	0.5
Neoprene	3000	0.4	-	-
Viton	114	0.8	-	-
	1	1		

# **Appendix 7 – Local Plant and Safety Regulations**

Regular maintenance inspections are carried out on ANTARES and STAR accelerator vacuum equipment to ensure operational status, vacuum quality, vacuum pump performance, and the mechanical and electrical integrity of the systems.

Generally, the integrity of the vacuum systems can be measured as a direct function of the known static vacuum pressures in a given vacuum system. That is, a lowering of vacuum from the known average vacuum pressure in a given system will indicate either/and, a leak in a seal, a failing vacuum pumping system or a structural failure.

The following is an overview of the application of the ANSTO OHSE Plant and Safety Standard, AS 2309, to vacuum systems in the accelerator facilities. *Reference: http://docushare.ansto.gov.au/Get/File-32340/Plant\_Safety\_Standard.pdf* 

Risk	Hazard	Risk rating	Controls	Risk rating (with controls in place)
Catastrophic failure vacuum vessels	Implosion – scattering of material fragments and loose fittings becoming missiles	Very low	<ul> <li>Use of vacuum standards to design equipment for safe usage.</li> <li>Protective barriers around equipment and fittings that may be susceptible to knocking</li> </ul>	Very low
Catastrophic failure vacuum beam lines and associated equipment	• Implosion – scattering of material fragments and loose fittings becoming missiles	Very low	<ul> <li>Use of vacuum standards to design equipment for safe usage.</li> <li>Purchased equipment from reputable companies/designers only</li> </ul>	Very low
	• Electric shock from fault in high vacuum gauge	Very low	• Vacuum gauges using high voltages in key areas will automatically switch off at low vacuums/atmospheric pressure	Very low
	• Failure of welded joint	Very low	• System will leak to atmospheric pressure at a rate commensurate with the conductance of the failed joint. No control is deemed necessary.	Very low
Electric shock from a failed vacuum pump or powered fitting	Electric shock from frayed lead or failure of earth inside of equipment	Low	<ul> <li>All equipment is protected from short circuits by circuit breaker protection.</li> <li>Most circuits now have RCD protection.</li> <li>A program is in place to put all circuits onto RCD protection</li> </ul>	Very Low
Failure of support structures	Crush, fall, bump, trip hazards during and after failure	Low	<ul> <li>Use of vacuum standards to design equipment for safe usage</li> <li>Access to equipment at height via approved ladders or dedicated platforms</li> <li>Non acceptance of standing on beam line stands and mounts</li> </ul>	Very Low
breakages to	<ul> <li>Fragments of broken</li> </ul>	LOW	<ul> <li>Barriers around exposed equipment</li> </ul>	LOW

1) Risk assessment of plant – Accelerator Facilities Only

fittings and equipment protruding from vessels and beam lines	<ul> <li>parts become missiles.</li> <li>Cuts to personnel bumping into fittings</li> <li>Secondary injuries including electric shock from exposed wiring</li> </ul>		<ul> <li>that is deemed to be hazardous</li> <li>Access control into accelerator areas restricted to accelerator personnel and visitors who are escorted</li> <li>See electric shock hazard controls above</li> </ul>	
Over pressure of vacuum systems during venting	Positive pressure in beam lines when venting with gases from high pressure storage	Very Low	<ul> <li>Pressure relief valves in use at systems where regular vacuum cycling is necessary</li> <li>Use of portable pressure relief fittings where local venting is required.</li> </ul>	Very Low
Implosion of glass view ports	Shards of glass scattering towards operators	Low	<ul> <li>Use purpose designed commercial vacuum windows</li> <li>Use small diameter (&lt;50mm x 6 mm thick) quartz windows on vacuum systems where ion beams can strike. Quartz is not known to crack through but to develop small surface anomalies</li> <li>Use double rubber seals to support windows to minimise mechanical stresses</li> </ul>	Low
Broken film windows	Sudden venting of vacuum to atmospheric pressure	Very Low	<ul> <li>Damage to equipment – high vacuum gauges must have auto shutdown at low vacuums</li> <li>Vacuum system isolated with gate valve to minimise loss of vacuum in adjoining systems</li> </ul>	Very Low
Breach of accelerator tubes with SF6	Vacuum systems pressurised with SF6 gas	Low	<ul> <li>Large gate valves either end of the accelerator vacuum tubes will shut if pressure rises to maintain SF6 within the vacuum vessels</li> <li>Some gas will enter the vacuum systems outside of the isolated section. The pressure will be relieved through blow off venting ports at each end of the vessel.</li> <li>Oxygen depletion alarms will sound if dangerous levels of SF6 are released. Personnel will be ordered to leave building through PA announcement</li> </ul>	Low

- 2) Risk control measures for eliminating or reducing the assessed risks *See table above*
- 3) Registration or notification of design of plant *Not deemed necessary. See AS 4343-2005*
- 4) Licensing of plant Not deemed necessary. See AS 4343-2005
- 5) Maintenance of plant

*Regular maintenance applied via Accelerator Maintenance system. See ENV-I-076-004 Vacuum Systems Maintenance manual* 

- 6) Plant operations and risk controls *See table above*
- 7) Requirements in relation to specific types of plant *Only vacuum system plant*
- 8) Training and supervision Vacuum technology training is provided in-house to world's best practices. Additional training has been provided by the Vacuum Society of Australia through their training courses.
- 9) Monitoring and evaluation Regular maintenance applied via Accelerator Maintenance system. See ENV-I-076-004 Vacuum Systems Maintenance manual

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# Vacuum Technology

# and

# Vacuum Design Handbook

# for

# **Accelerator Technicians**

Prepared by David Garton November 2011 Revision 0 Vacuum Technology and Vacuum Design Handbook for Accelerator Technicians

This handbook is a compilation of information gathered from over 50 years of direct hands-on experience to applicable information widely available from the vacuum technology industry. It seeks to address common and specific vacuum technology problems whilst clarifying the design standards and philosophies adopted for use in the ANSTO accelerator facilities. The author wishes to thank the reviewers and the many technicians from ANSTO that have contributed directly and indirectly to this booklet.

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### Chapter 1

### Vacuum design standards and good practises for the Accelerator Area

- \* This chapter must be read in conjunction with the detailed vacuum information in this document.
- \* Based on best practises developed at ANSTO and other local Australian accelerator laboratories in conjunction with Australian Standards and industry technical guides, referenced throughout this document.
- \* Where a standard is unavailable for a specific design, best practices are used from industry's best practises.
- \* *Refers to all vacuum chambers, beam lines and other enclosures used on the accelerators or stand alone.*

#### 1. Basic design

- All vacuum vessel designs must be consistent with the design standards in Australian Standard AS 1210-2010 and maintain a Hazard Level of E as per the design conditions tabled in "Hazard Levels of Pressure Equipment" in Section 2, Hazard Levels of Pressure Equipment in Australian Standard AS 4343-2005.
  - The vacuum equipment designed by/for the accelerator area maintains a Hazard level of E. All pressure equipment with a pV  $\leq$  30, pressure (p in MPa) x volume (litres)  $\leq$  30 is classed as Hazard level of E. For vacuum equipment in Hazard Level E the derating value of 0.1 is applied, ie 0.1pV  $\leq$ 30. Note the amount of stored energy at this level is very small. For example a Ø 1000 mm chamber, 1000 mm high will have a pV for vacuum equal to 7.85.
  - AS 4343-2005, Section 2.1.2 Typical hazard levels, part (d), states for Hazard Level E, "This equipment is usually exempt from special regulatory control but is <u>covered</u> by general plant safety regulations". See Appendix 7.
- All designs of chambers excluding general beam lines and fittings should be checked for structural integrity using finite elements analysis. Sound designs with acceptable structural integrity will be released for manufacture. A copy of the design acceptance tests must be filed with the project file. The standard finite element analysis can be performed utilising Solidworks. (Modelling for irregular shaped chambers, calculations Appendix 1 where appropriate for regular shapes)
- Stainless steel (304 or 316) will be used to construct chambers, beam lines and general fittings unless it is a *special* requirement to use other metals. Do not choose substances that have high outgassing rates or can contaminate vacuum systems.
- Measurement chambers and other large volume chambers should be cylindrical unless the chamber is an enclosure between pole faces (eg magnet box) or is an odd shape to accommodate steering plates or devices. When non-cylindrical chambers are required deflection of chamber walls should be modelled and taken into consideration.
- Other chambers including magnet boxes, ESA and deflector coffins to have suitable rib strengthened rectangular or curved sides where it is not practicable to increase the wall thicknesses.
- Minimum wall thickness calculation methods can be found in Appendix 1. As a general rule care must be taken when using formulas for vacuum calculations as historically many units other than SI units have been used to form some equations. For example length in cm, and pressure in torr is common in old notes.

- Top and bottom flanges to have integrated structural support rim which becomes the flange and seal for chamber base and top (user) flanges.
- Ports must be a suitable length to ensure unobstructed insertion for screws into flanges. Suitable weld preparation must allow for adequate weld penetration. See Welds below.
- All internal edges must have a smooth radius. No sharp edges to be left after welding or surface finishing.
- All machined finishes other than vacuum seals to be equal to or better than 1.6 microns
- No sections within the vacuum space will have metal to metal contact where gas can be trapped unless it is at the edge of a seal joint. When this is necessary, pump out grooves must be incorporated in the design.
- Vacuum windows shall be made from toughened glass or quartz. No plastic to be used for windows unless the window is a flange on a chamber that is designed to withstand more than 100 kPa. The large plastic flange/windows should be made from >18 mm thick up to Ø200 and >25 mm thick for up to Ø400 in clear Perspex or polycarbonate.
- For ANTARES beam lines the preferred flanges are Dependex, nominal diameter 100 mm. See section 8 for detail on Dependex flanges.
- For STAR there is no preferred flange type. It must be selected depending on the desired base vacuum and interface to other adjoining flanges.
- As a guide, the MDC catalogue from Vacuum Products Corporation 2003 onwards, Building Blocks for Vacuum Science and Technology, provides vacuum tubing dimensions for most common sizes. The dimensions must be assumed to be minimum dimensions. Other tubing sizes should be calculated using the formulas in Appendix 1.
- Vacuum pumping ports leading to the vacuum pumps must be designed to optimise conductance.
- Pressure relief valves to be incorporated where a vacuum system can be vented to >10 kPa above atmospheric pressure.
- ASME state that the design of an external pressure vessel must consider Material type, diameter of chamber, unstiffened length, temperature and wall thickness

#### 2. Welds

- TIG is preferred for vacuum sealing welds as minimal scale and flux is produced. Electron beam welding is suitable for thin walled materials. TIG produces high quality welds with or without filler rods to produce flat, smooth well penetrated welds. TIG comes into its own where the weld preparation allows for melting together of the parent materials without filler rods. MIG has a higher deposition of filler material so care must be taken to ensure welds are kept smooth and flat.
- No standard exists for vacuum welding of compact scientific vacuum equipment where pressures approaching zero are necessary. Equipment designed in the accelerator area and welded in the ANSTO workshops since 1990 has had penetrations of ≥1mm but ≤2mm on a butt weld and ≥2 mm on a fillet weld. Experience has demonstrated the adequacy of these figures. No failure reported.
- For thin walled materials ie < 1 mm, such as bellows, manufacturers' will use their industry standard. ANSTO does not weld thin wall materials.
- Weld preparation must be provided where possible to ensure even heat penetration.

- Electric arc with rods is not recommended due to flux trapping. This can cause pits which have long lasting outgassing periods.
- All welds that form a seal against atmospheric or other positive pressures must be made to the internal side (vacuum side) of the joint.
- Tack (stitch) welding is recommended on the external side only with less than 50% of the diameter covered in small intervals. This minimises the chance of trapped voids and facilitates He leak testing post welding.
- All welds must be free from pitting and scale. Must be cleaned to bare metal. If chemicals are used to clean welds care must be taken to remove all traces of chemicals.
- Some chambers such as magnet boxes will need to have some joints welded externally due to access problems on thin chambers. In this case, good penetration must be achieved to ensure minimal gas trapping points.

#### 3. Surface finishes

- The inside of the chamber and ports must be 1.6 micron or better for mechanical finishes.
- Can be chemically cleaned (polished) but all traces of chemicals must be neutralised and removed prior to use in vacuum.
- All machining oil to be removed with an alcohol scrub then acetone rinse, minimum. The use of kerosene, turpentine or other oily solvents is not encouraged however where they are used the final clean must be with acetone followed by alcohol. Water based cutting fluids can be used as they are more readily removed therefore having less effect on outgassing.
- Nitrile gloves to be used when assembling cleaned components. Other un-powdered gloves may be used but only those that do not react with the solvents being used.
- Care must be given to cleaning and handling surfaces that will be exposed to vacuum. Gloves should be used to minimise the transfer of dirt and oils from the skin to vacuum surfaces.
- A suitably clean area should be used for the preparation of vacuum systems and all tolls used should also be cleaned (degreased).
- Stainless steel can be cleaned in nitric acid to remove scale that remains from rolling or wire cutting. It is may also be used to prepare stainless steel components for use in ultra-high vacuum systems.

#### 4. Flanges

- Flanges must be standard flanges selected from the group referenced this document for compatibility reasons. Exceptions may be made but all 'o' ring seals must be to Australian Standard AS 2842-1986. External flange rings not in contact with the vacuum space may be aluminium alloy. Stainless flanges are not economical or necessary unless the seal requires forces to crush metal seals such as copper ConFlat, aluminium or indium wire.
- Minimum thicknesses for top and bottom flanges to be calculated using the standard in Appendix 1 Structural Calculations for Scientific Vacuum Vessel Design.
- All other flanges to have threads consistent with their design standard.
- ANTARES Primary flanges will be Dependex unless interface is necessary to accommodate other standard flanging.
- For Dependex, screws to be imperial sizes consistent with UNC series.

• STAR utilises many of the major vacuum flange/seal designs. The most appropriate design must be chosen that meets the specific application.

#### 5. Seals

- 'O' ring sizes and sealing groove dimensions must comply with the Australian Standard AS 2842-1986. Adjustments to groove sizes can be made where specific 'crush' is necessary for special applications.
- Viton rubber 'o' rings to be used unless a special requirement for an alternate elastomer is identified.
- Standard flange and seal sizes to be used unless an application prohibits their use.
- It is recommended that vacuum sealing surfaces are cleaned just before a seal is made to minimise the possibility of dust and hair settling on sealing surfaces.
- Metal seals may be used in specifically designed sealing surfaces including, aluminium and indium wires. They are very good as ultra-high vacuum seals.

#### 6. Screws

- All screws for flanges other than Conflat will be made from stainless steel in either 304 or 316 grades.
- Screws used in Conflat flanges will be made from A2-70 or A4-70.
- All screws to be socket head unless access necessitates hexagon or other special head. Slotted or Philips head must not be used.
- Stainless steel flat washers to be used under all screws where practicable. Do not use lock or star washers on standard flanges.
- In the vacuum space, no screws to be used in holes unless the holes are through holes and can be pumped from both sides. Screws can be used in blind holes if they have a minimum Ø1 mm hole drilled through the centre to allow trapped gas/air to escape.
- Unless a standard flange uses non metric thread, all screw threads must be within the metric series. Unless there is a specific need, avoid using intermediate metric sizes, that is, 0.5 mm steps such as M1.5 mm, M2.5 mm etc.
- Anti-seizing compounds can be used on threads but extreme care must be taken to ensure they do not enter the vacuum space. Graphite powder can be used as a lubricant inside the vacuum space. It can be applied using ethanol to wet the surface to be lubricated. Allow solvent to evaporate prior to pumping.

#### 7. Feedthroughs

- Sliding seals should have dual 'o' ring seals with a gland formed seal at least at one end. The shaft finish must be 0.8 micron or better.
- All sliding shafts must have retainers to stop them being pulled through into the vacuum space.
- All coax feedthroughs to be glass or ceramic hermetically sealed. No plastic.

#### 8. Lubricants, vacuum greases and epoxies

- Only vacuum specific grease and oils to be used in vacuum systems.
- Silicone based or particular Apiezon family lubricants design for vacuum use, are recommended on sliding seals. Thin smears only.
- No vacuum greases should be used unless there is a small leak through a damaged sealing surface and all other options are not feasible. If grease must be used it must be designed for high vacuum use and only a very thin smear to be used.
- Do not use grease to fill suspected leaks in welds or fittings.
- Where a vacuum epoxy must be used the surface must be well prepared to ensure appropriate bonding. Care must be taken to ensure the epoxy seal doesn't create a trapped void of air which may become an outgassing problem. 'Torr-Seal' is commonly used due to its relatively low vapour pressure, and thermally stable to 120°C. (this is a Varian product)
- Vacuum oils are not recommended for general lubrication as they can creep on surfaces spreading contamination and increasing the outgassing surface.
- See Appendix 3 section 1.5.

#### 9. Mounting

- The load of the chamber assembly must rest on a standard accelerator beam line table ANSTO, HVEC, HVEE or NEC or a purpose built stand that minimises vibration.
- Vibration decoupling will be achieved by supporting the chamber separately from the vacuum pump and using an edge welded bellows between the chamber and pump.
- For ANTARES beam line vacuum equipment the beam axis alignment is nominally 1750 mm from floor height.
- For STAR beam line vacuum equipment the beam axis alignment is nominally 1193 mm from floor height.

#### 10. Access

- The working face of the chamber must face an area that is easily and comfortably accessible. The floor space immediately in front of the working face should be raised using steps to facilitate access.
- Heavy or awkward shaped vacuum equipment must be designed to be lifted with the aid of over cranes for example lifting lugs.

#### 11. Testing

- Vacuum equipment must be tested prior to routine use to minimise time spent chasing leaks in completed systems.
- Vacuum vessels must be helium leak tested to ensure all seals are better than  $1 \ge 10^{-10}$  Pa.m<sup>3</sup>/s ( $1 \ge 10^{-9}$  mbar.l/s). As a standard, during a leak test there should be no deviation from this baseline level for a leak-tight system. A record of the test is to be noted on the engineering drawing showing, date, maximum leak rate, leak location/s, leak detector used and operator name.

- For all other vacuum equipment in-service testing may be necessary to allow complete operation of various feedthroughs and positioners.
- Caution must be taken before positive pressure testing vacuum equipment as the equipment may be irreversibly damaged. Warning Do not use water testing on any vacuum equipment.
- A Residual Gas Analyser (RGA) can be used to give more detailed information about the gas composition inside a vacuum volume. An RGA can be used as a substitute for a helium leak detector with the benefit of providing more information about outgassing, or virtual leaks inside the vacuum volume. The pressure of the vacuum system must be  $< 1 \times 10^{-2}$  Pa to prevent damage to the filament inside the instrument. Instruction manuals must be checked before using the RGA head to ensure safe and effective use.

#### 12. Operation and Continued Monitoring

- All chambers should have a high vacuum gauge sensing within the chamber space. This includes all adjoining compartments that may be isolated by a valve.
- The chamber must have valves attached that allow complete vacuum isolation from the beam lines and other equipment.
- To protect high vacuum gauges against the effects of operating in low vacuum, high vacuum gauges should be automatically isolated if the vacuum pressure rises above  $1 \times 10^{-2}$  Pa.
- High vacuum gauges must be cleaned at minimum every 3 years or whenever a gauge develops instability as compared with adjacent gauge readings.
- Systems indicating higher than normally observed pressure must be isolated and repaired. A helium leak test is recommended if the leak location is not clear.
- To avoid undesired contamination, vacuum chambers should be vented with dry nitrogen or argon instead of air. This minimises water vapour and oil from depositing on the vessel walls.

### Chapter 2

### Design Criteria, Information and Philosophies for Vacuum Systems used in the Accelerator Area

#### Overview

In an accelerator system ion beams are transported from ion sources through to end stations (or detectors) through vacuum space maintained inside of beam tubes (lines). The relatively low number of gas molecules in the vacuum ensures a higher mean free path for the accelerated ions and hence less loss of ions through collision which is crucial especially for AMS measurements when counting a very small number of ions. A high vacuum also minimises the formation of plasmas in an electric field at high voltages which can avalanche into spark breakdown damaging sensitive equipment.

The vacuum systems used in the accelerator area are designed to be fail-safe to protect people and equipment. A very high proportion of problems with vacuum systems relate to, seals leaking on first use, being incorrectly mounted or failing due to aging 'o' rings. Other problems, however less frequent, include mechanical damage to sealing surfaces or fatigued parts such as bellows. The problems are all characterised by a poor vacuum or a previously steady vacuum that begins degrading slowly as observed during the operation of the vacuum system over its life time. The remaining problems are due to poor welds or vacuum fittings breaking. Poor welds are usually found during the initial helium leak test. They are remedied by either removing and re-welding the section or using a commercial vacuum epoxy. The choice is dependent on whether the weld is on a structural component or just a seal. Broken vacuum fittings are replaced or repaired.

Within the accelerator area. physicists work directly with technicians who apply broad knowledge and experience in vacuum technology to develop their concepts for use in the accelerator environment. This mix has been successful for 50 years. However not all designs are completed in the accelerator area. There are cases where commercial projects call for a more production based design or designs of chambers with complex noncylindrical shapes. These may go to the ANSTO drawing office for completion or a specialist vacuum design company.



Fig 1. ANSTO designed and developed beam lines on the STAR accelerator in building 22

It is not possible and often not necessary to standardise the design requirements for all components used in the accelerator area so the following information is intended to help designers of vacuum equipment to comply with accelerator specific and Australian standards. As of 2011, there are no known commercial workshops in Australia who specialise in the production of vacuum vessels. Local representatives from vacuum suppliers can provide access to overseas specialist workshops.

### 1. Terminology

**ANTARES** – Australian National Tandem for Applied RESearch. The HVEC FN Tandem accelerator in B53.

Backing – To pump on the exhaust (back) of a high vacuum pump

Beam line – The evacuated tubes that carry the ion beams from the ion sources to the end stations

**Cryopump** – Cryogenic vacuum pump. Uses a helium cooling interface to the vacuum space to freeze gases.

**Dependex** – A type of flange which is the standard flange used on ANTARES and ANSTO built beam lines.

**Fore line** – The vacuum line, tube, pipe, whatever between the high vacuum pump and the backing pump

High vacuum – See details in Chapter 3, para 1.3

ISO - International Standards Organisation

KF – Klein (small) Flange

LF – Large Flange

Low Vacuum – See details in Chapter 3, para 1.3

Outgassing - the liberation of gaseous vapours from the surfaces within a vacuum space

Pa – Unit of pressure, Pascal. 1 Pa = 1 N/m<sup>2</sup> = 0.01 mBar = 7.5 mTorr = 1.45 x 10<sup>-4</sup> PSI

**Pump down = Pump out** – evacuating gas from a vacuum space

**Roughing or Roughing-out** – Usually used to describe the initial transfer of gas from atmospheric pressure to low vacuum.

STAR – Small Tandem for Accelerator Research. The HVEE Tandetron accelerator in B22.

Turbo – an abbreviation for turbomolecular vacuum pump

**Ultra-high vacuum –** See details in Chapter 3, para 1.3

Venting – the equalisation of negative pressure to atmospheric pressure in vacuum equipment

Vessel – generic for a vacuum chamber where an instrument is mounted or interfaced into the vacuum space.

#### 2. Standard Units

#### 2.1 General

Although metric standard is adopted across all ANSTO accelerator facilities it does not mean that all equipment is metric as the US built FN Tandem is mostly imperial, conversely, the Dutch built HVEE Tandetron is all metric. There is a combination of metric and imperial devices used throughout the facilities but designers must ensure that where practicable all new designs are metric.

#### 2.2 Threads

New equipment for use in the accelerator area, where practicable will be designed with metric standard threads.

• Contrary to the standard, on ANTARES there is a combination of threads in use. The accelerator high voltage generator and parts that have originated from the United States are

mostly imperial. Typically, UNC and some UNF. Most beam lines and endstations are metric but all Dependex and most Conflat flanges are imperial.

• ON STAR all threads are metric. ANSTO designed and built beam lines are metric except for the Dependex flanges which are UNC.

Care must be taken to identify threads and use the correct screws. Where practicable, metric threads are to be used other than on Dependex flanging.

#### 2.3 Pressure

All pressure indicating devices shall be in the SI unit, Pascal. Historically, Torr was used but this was replaced by Pa in the late 1980's. Most international accelerator labs use mbar. The conversion is as simple as multiplying mbar by 100 to get Pa. Pascal refers to a physical force of newtons per metre squared.

### 3. General Design Philosophies

There are many varieties of vacuum chambers and equipment in use in the accelerator area. Most have been designed and made at ANSTO. Examples include vacuum equipment measurement chambers where samples are measured, beam line transport components, ion source components or sensitive detectors, to name a few. Each has its own unique characteristics and vacuum design considerations.

In order to maintain a suitable mean free path for ion beams whether being transmitted from a source or scattered from a sample into a detection system, high to ultra-high vacuums are necessary. In a high vacuum the density of gas is low enough to minimise electron flow from surfaces at high voltages to surfaces at lesser potentials. It is therefore prudent to ensure designs of vacuum equipment that directly couple to the accelerator are designed for a minimum of high vacuum operation.

In every case the designers must ask the following questions:

- What shape? How big? Where will it fit? How will it be mounted? What level of vacuum?
- Will it be ridged enough and have low enough distortion to mount ion optical components?
- Is the design flexible enough to enable small scope changes in the future? Is it designed with consideration for reuse in another application when the project finishes?
- Does the design use vacuum technology best practises?
- Does it meet Australian and local standards where applicable?
- Is it a safe design?
- Is it achievable with resources available?
- Is it cost effective. Can we modify existing equipment to achieve a similar result?
- Does it have low environmental impact?

#### 3.1 The Shape

The first consideration is to define the actual use of the vacuum equipment relative to the experiment. Where possible cylindrical shapes should be considered first as they are stronger under vacuum and consequently being round, ports can be welded into the chamber aligned radially to the centre which ideally aligns instruments including detectors and cameras with the intersection point of the ion beam and the sample being measured.

Non-cylindrical chambers must be carefully designed to included adequate strengthen ribs to minimise distortion and hence misalignment of instruments.



Fig 2. A few examples of vacuum vessels used in the accelerator area

#### 3.2 The size

The size is dictated by the space needed inside of the vessel or equipment in order to meet the operational demands. In the case of measurement chambers too big may mean problems with the distance between samples and detection systems and too small may mean manual handling of samples may be inhibited. Large chambers may have to be certified as per AS 4343-2005, Hazard Levels of Pressure Equipment in Australian Standard.

#### 3.3 Location

It will be an agreement between the accelerator user groups as to where equipment and beam lines will be located. This agreement will endeavour to minimise conflicts in beam line space, usage, accessibility and resourcing.

#### 3.4 Mounting

In general equipment must be secured onto the floor unless it is designed as mobile. An accelerator compliant beam line table should be used with standard beam line supports. Vibration must be identified during the design stage and decoupling stages provided.

Insertion bellows must be provided in long sections of beam lines to enable compression of the beam lines to facilitate dismantling and to ensure minimal strain on vacuum joints.

Designs must consider trip, bump and fall hazards. Structures that support platforms or heavy equipment that may otherwise fall must not be reliant on the structure supporting the vacuum chambers and beam lines. For example, ladders, walkways, etc.

- For ANTARES beam line vacuum equipment the beam axis alignment is nominally 1750 mm from floor height.
- For STAR beam line vacuum equipment the beam axis alignment is nominally 1193 mm from floor height.

#### 3.5 Flexible design

It is prudent to ensure where practicable designs ensure some redundancy for feedthroughs, access ports and internal space. A major problem with early chambers was the lack of space allowed for inserting samples by hand or manually manipulating internal equipment and wiring. It is easier to design flexibility into the chamber or equipment at the design stage rather than having to dismantle it and have it reworked in the workshops later.

#### 3.6 Can it be made?

When designing any equipment seek advice from a qualified machinist. Often designs include machining that may be very expensive or not possible. Early in the design phase ensure that the resources are available to service the vacuum equipment. It is recommended to consider the following early in the design stage:

- Power
- Cooling water
- Air cooling
- Signal lines
- Control lines
- Add-on electronic and mechanical instrumentation

#### 3.7 Safe design

There are no cases that the author has been able to find of scientific vacuum chambers or equipment, like those used in accelerator facilities, failing and causing injury. Vacuum pressure is relatively low, -100 kPa, or about half the equivalent magnitude in a car tyre.

For safe designs the structure must meet the minimum dimensions as calculated in Appendix 1 - Structural Calculations for Scientific Vacuum Vessel Design.

Care must be taken with the design of the following:

- Glass view ports (most glass breakages are in the form of a crack which leak to atmospheric pressure quickly without the spread of shards)
- No internal sharp edges; in the case where a person must insert their hands blindly to access components.
- Venting limits pressure relief for positive pressure?
- Evacuation rate regulation; especially in the case where very thin material windows separate different pressure systems within a single vacuum space for example beryllium windows on x-ray detectors.
- Equipment mounts/stands; where an operator must climb up high to operate devices on the system
- If oil type vacuum pumps are used then mist filters must be fitted to the exhausts.

#### **3.8** Cost effective design

All *bells and whistles* may not be necessary even after considering some design redundancy for future changes. Where possible, designs should consider integrating components that are available off the shelf. Designing what has already been designed by someone else is wasting time and effort.

It may be cheaper to modify an existing chamber for use rather than start from the beginning. Many chambers used around the accelerator area have successfully had one or more lives.

#### **3.9** Design by numbers

This is a matter of ensuring structural integrity and being able to prove it. Likewise, designing the vacuum pumping system requires calculating the most appropriate size pump rather than going for the "biggest"!

There are formulas in Chapter 3 that allow must crucial calculations to be made from wall thicknesses to conductance. Good enough is not acceptable. Replication of previously designed equipment is acceptable.

#### **3.10** Australian Standards

Compliance is mandatory however there are few standards that are relevant to scientific vacuum vessels and equipment. The industry has developed its own standards for flange and seal designs which can be seen in the catalogues of most leading manufacturers of vacuum equipment. It is the responsibility of the designer to ensure compliance with all relevant Australian Standards. Chapter 1 discusses what standards are included.

#### 3.11 Environmental impact

Most vacuum systems used in the accelerator area today are designed to be oil free. That is, they do not use oils for lubrication of vacuum seals. There is however some oil vane and diffusion pumps still in use but the oils are responsibly disposed of through waste management at ANSTO. Some old diffusion pumps had been used with mercury as the pumping agent. Personnel must be cautious of using old diffusion pumps and their associated backing pumps as there may be residual mercury on surfaces and in the backing pump oil.

Solvents used for cleaning are used in small amounts and usually for wiping rather than immersing. Where baths are needed the solvents are reused when possible. The solvent bath used for degreasing, self recycles the solvent by design however there is a small loss of solvent over time into the atmosphere.

Where possible solvents used are selected with the environmental impact in mind.

#### 3.12 Manufacture

If ANSTO cannot provide the level of manufacture quality in-house then scientific vacuum equipment is best manufactured by companies that specialise in high tolerance machining and welding. A general metal fabricator will not suffice.

Attention must be paid to considering standard sized tooling during manufacture. Some companies will charge for the purchase of tooling that they consider to be non-standard.

It is imperative that the drawings for manufacture state all tolerances and qualities for the finished product.

On all design drawings, where the angle of the welded ports is crucial, ensure angular tolerances are clear and achievable. If necessary, specify the use of mandrels to hold ports in place during welding.

Some success has been made outsourcing the manufacture through vacuum companies to overseas manufacturers who specialise in high quality small runs.

#### 3.13 Testing

Testing the vacuum equipment or chamber is essential to establish compliance with the design and the all important ultimate base vacuum. Helium leak testing is the standard method used for chambers and most equipment however on some equipment in-service testing may be the only option. This is where add on vacuum equipment doesn't change the original base vacuum of the system it is connected to.

#### 3.14 Inspection

All parts should be cleaned prior to inspection. This doesn't need to be the final clean but enough to ensure all welds and surfaces can be clearly inspected. Welds must be checked for pits and alignment along the weld lines and seals must be checked for surface finishes. All crucial dimensional must be checked. It is recommended that any non-complying findings are photographed and documented in the project file. All non-complying parts should be reworked rather than *patched up*.

#### 3.15 Certification

As most vacuum vessels and equipment in the accelerator area fall within Hazard Level E of AS 4343-2005, certification is a local process. For large measurement vacuum vessels, results from the inspection and leak testing will be recorded on the engineering drawing and registered in the local accelerator register and/or ANSTO drawing register as appropriate.

#### 3.16 Documentation

For all large measurement chambers requiring any level of design calculations, design documents must be kept with project files.

#### 3.17 Maintaining

Vacuum chambers and beam lines do not require high levels of maintenance other than a general periodic clean and occasional seal replacement. Vacuum fittings and devices will have their own level of maintenance so access to these components must not be difficult.

The design must be easy to maintain especially moving parts, sliding or rotating seal and other parts in the vacuum space that may wear. Also vacuum pumping systems that may be mounted under or near the vacuum chambers. Generally any part that will require any level of routine maintenance must be accessible without the need for a major disassembly.

Vacuum pumping systems other than oil free systems, connected onto beam lines and chambers must include suitable oil traps between the backing and high vacuum pumps and where mist filters on all exhausts.

Some high vacuum gauges will require periodic cleaning. On ANTARES high vacuum cold cathode gauges are scheduled for cleaning every 12-18 months or max 3 years for difficult to service or less crucial instrument locations. Otherwise gauges will be cleaned on demand within the periods.

#### 4. Performance and Operations

#### 4.1 Ultimate Vacuum

The ultimate vacuum is the maximum vacuum or lowest pressure the vacuum equipment typically reached after 3-5 days of uninterrupted pumping. This is a benchmark value which is used to assess the long term quality of the vacuum system. It is sometimes referred to as the *base vacuum or static vacuum*.

Generally, vacuums in the beam lines and chambers are well within the high vacuum range. Vacuums lower than this range will indicate a higher than normal gas load, a failing seal or a pump malfunction. On the accelerators the two main types of high vacuum pumps are turbomolecular and cryogenic pumps. For a system designed for high vacuum or better with no leaks and low outgassing then vacuums better than  $4 \times 10^{-6}$  Pa should be readily achieved.
The ultimate vacuum is improved by the correct choice of materials, seal types, high vacuum pumps used cleanliness and correct operation. All fittings must be designed or selected deliberately for high to ultrahigh vacuum use. Surface preparation and cleanliness will contribute to improved vacuums. A finger print can outgas for long periods of time. A bigger (higher speed, higher ultimate vacuum) vacuum pump does not mean a better vacuum!

### 4.2 Working Vacuum

The working vacuum may be lower than the ultimate vacuum due to sample cycling (changing), introduced gas loads from adjoining systems or outgassing samples. A system must be designed to ensure the high vacuum pumping system has the optimum pumping speed and ultimate vacuum. Once a vacuum system reaches the "working vacuum" little operator intervention is necessary to maintain it but there are some important points regarding ongoing monitoring to ensure a failure is not immanent.

For sections of beam lines that are holding a static vacuum a cursory glance at the local vacuum gauge will indicate the stability of the vacuum. Generally, the (vacuum) pressure will change slightly throughout a day for example due to diurnal changes in ambient temperature and perceived changes due to ionisation gauges "wandering" as they become dirty.

In sections of beam lines that are in the vicinity of operating beam lines or near end stations the vacuum will be continually changing. Factors that influence these changes include increased gas loads from samples outgassing or surfaces that have just be exposed to high humidity, outgassing due to temperature changes within the vacuum space (including bake outs), outgassing from ion beams striking plastics, etc. Gas molecules don't have a preference to whether they travel towards or away from a pump during molecular flow so the source of gas can come from just about anywhere in the open system. These factors must be considered when monitoring vacuums throughout an entire system.

If a vacuum system's pressure rises slowly over many days or weeks if it is not related to the cryopump loading then it may be a seal failing. In this case a helium leak test is recommended.

A fundamental flaw in many vacuum system designs is the neglect for monitoring the backing pressure between the backing pumps and turbo pumps. Pirani gauges which are best suited for this role are relatively cheap and are the best diagnostic tool for determining overall vacuum system performance. The backing pressure should be consistent with changes in the high vacuum pressure. By monitoring the backing pressure over time, the base vacuum will be realised relative to the high vacuum so a pending failure of the backing pump can be determined, hopefully, well before it happens.

Care must be taken to maintain systems that have the potential to degrade over time for example an oil vane roughing pump will back stream oil mist that may accumulate in the high vacuum side of the system over time. This will contribute to lower vacuums due to higher gas loads from the partial pressures of contaminants.

#### 4.3 Sustainable Operation

Now that the vacuum system is designed, manufactured and commissioned for routine service, operating procedures have to be defined and adhered to, to maintain reliable trouble-free service. There are 3 processes that summarise the continuous cycle of operating a vacuum system:

- Pumping down from atmospheric pressure
- Using the working vacuum
- Venting to atmospheric pressure

The accelerator beam lines are divided into stand alone vacuum systems but in essence function as one. Each system can impact on a neighbouring system if things go wrong. In the case of end stations which cycle up and down in pressure more than any other devices an operator must be 100% sure of the pump down sequence in order to avoid damaging, pumps, samples, high voltage devices, detectors, etc. It is possible to shut down the accelerators as well. Most sections of the accelerator vacuum systems are interlocked to isolate a pressure surge to small area. Beam line vacuum systems are design so that both the vacuum pump and the beam line are isolated via gate valves. This is a typical arrangement for endstations as well.

The following must be read remembering that all 3 processes work in a cyclic fashion in a continuous loop.

#### 4.4 **Operational Protocols**

Key vacuum systems operate continuously, 24 hours per day, seven days per week. There are several vacuum systems that are cycled on and off as usage demands. All vacuum systems have one or more high vacuum gauges, some of which are interlocked to make the systems fail-safe. Any slow leaks or major catastrophic failures will close valves and isolate the system. In the few cases where no interlocks are used, a catastrophic failure will result in the high vacuum pump shutting down.

- For a <u>turbo pump</u>, as the pressure rises the gas load on the pump will cause the pump to draw more current to maintain it's speed. It will eventually fail when the current trip level is reached in the controller.
- For a <u>cryopump</u>, as the pressure rises the inflowing gas will condense or freeze onto the in the pump until it begins to warm up. Eventually there will be a thermal trip activated o the compressor which will shut is down. In both cases it is fail-safe.

As part of the accelerator's maintenance system, daily monitoring of all vacuum pressures is carried out. Where a pressure is seen to rise by a few percent without a particular reason, further monitoring will occur and corrective action started. Instantaneous failures are dealt with immediately. All observations and whether any maintenance carried out is noted on the maintenance log.

Where a vacuum system is isolated for service or where it has failed the appropriate tags are placed on the power leads.

Unattended out of hour's operation notification is not normally used as vacuum systems run continuously.

#### 4.5 Pump down

#### General

Pump down time is when most mistakes are made and significant time lost. Before a pump down begins the operator must ensure that all seals that were disturbed had been cleaned and replaced if damaged. The system must then be sealed ready for pump down and the roughing-out valve on the rough-out port, closed.

If a dry pump is to be used, such as a scroll pump, for rough-out then the tube is connected to the pumping port. If an oil vane rotary pump is used then ensure the cold trap has been filled beforehand.

#### Rough-out

Start the roughing pump and allow it to reach base vacuum. This is your first indicator of whether you have a leak in the system being pumped and when you have reached the pumping limit of the

pump. Slowly open the pump out valve on the system. This is where mistakes are made and damaged caused. Relatively speaking the volume in most sections of beam lines and endstations is small and the bulk of the gas can be removed very quickly. The problem is that a reduction in pressure too quickly can cause mechanical shock to fragile parts that must adjust slowly in order to minimise stress such as thin windows on detectors and detectors or devices that contain carbon foils.

Operators must always open roughing out valves slowly then gradually increasing as the gas load reduces. The change in the sound of the loaded pump is a very good indicator. A good design for more sensitive pump outs is to use a metering valve in line with the rough-out port to act as a regulating orifice.

#### Cryogenic Vacuum Pumps

For systems using cryopumps as the primary high vacuum pump it is essential to bring the base pressure down to the limit of the roughing pump before changing over pumping to the high vacuum pump. This reduces the gas load to be trapped (captured) on the cryo pump and extends the service life of the cryopump. Cryopumps are widely used on ANTARES as the primary high vacuum pumps but their limitation is that the warm up as they trap more gas causing a rise in base pressure. That is, when a cryopump is reaching saturation the temperature on the surface increases. More energy is required to hold the molecules on the surface and since it is almost fixed by design the temperature increases. A cryopump's effective pumping speed depends on the freezing and boiling points of the gases being pump relative to the cryopump's temperature. As the temperature increases more molecules leave the surface (boil away) raising the pressure. Some molecules are re-trapped as they hit the surface of the arrays and loose kinetic energy but they have a short residency time and soon add to the rising pressure in the system. Eventually the base pressure in the system becomes unusable so the pump must be regenerated.

#### Turbomolecular Vacuum Pumps

The other type of high vacuum pump widely used in the accelerator area is a turbo pump. Unlike a cryo pump that traps or captures gas molecules a turbo pump transfers the gas by conducting it almost one-way out of the vacuum space. The lowest roughing pressure before changing to a high vacuum pump is not as crucial when a turbo pump is being used. In saying this, the bulk of the gas must be removed before changing from roughing out to high vacuum pumping otherwise the vanes on the turbo pump may be overly stressed due to the pressure surge. A typical changeover pressure range is 5 - 50 Pa.

The optimum changeover time to minimise pump down times can be estimated with the assistance of the formulas found within the document. Letting the roughing pressure reduce to the limit of the roughing pump before changing over to the turbo pump is an inefficient method of system pumping. A turbo pump is more efficient at removing gas molecules as the flow moves towards molecular flow. Changing pumping to a turbo pump just before molecular flow is reached will increase the pumping speed and base vacuum will be reached sooner.

#### Pumping Speed

Each type of gas will pump away at a different speed depending on the mean velocity of the molecule which is a function of its mass. A hydrogen molecule of 0.02 kg/mol and mean velocity of 1762 m/s will reach a pump much quicker than say a Xenon atom of 0.13 kg/mol with a mean velocity of 217 m/s.

#### **Limitations**

In rare circumstances roughing pumps may be left pumping high vacuum systems by mistake. It is unlikely to damage either pump but the ultimate vacuum will not be reached as the roughing pumps are ineffective at conducting away molecules in molecular flow. Water vapour and residual solvents are difficult to remove from a vacuum space with *normal* vacuum pumping. Most low vacuum backing pumps provide gas ballast which can speed up the removal of water vapour and solvents that may otherwise condense inside of the pump during the compression stage. By introducing a small amount of air near the outlet of the compression stage the small increase in pressure helps carry water vapour and solvents to the exhaust before they can condense.

Some vacuum systems have been designed to allow chambers and beam lines to be heated. Heating causes contaminants to be removed from surfaces at lower than normal vacuums. The higher the temperature the faster the removal will be. Care must be taken to ensure heating doesn't inadvertently cause plastics and elastomers used within the vacuum space to outgas. Most bakeable systems use ceramics and metals only.

It is important that where vacuum systems are permanently connected to high voltage devices for roughing out, the insulated (plastic) pump out lines between the roughing pump and high voltage device must be brought up to atmospheric pressure before high voltages are reapplied. Otherwise the low molecular density of gas in the tube may ionise causing burning of the tube or even spark damage that can puncture the tube causing a leak.

# 4.6 Venting

#### General

Venting can cause damage to vacuum components just like pumping out a system from atmospheric pressure too fast.

#### Venting Speed

As discussed earlier the volume of vacuum systems on the accelerators is measured in litres to a several tens of litres. A catastrophic loss of vacuum will be more of a brief audible event rather than a dangerous mechanical failure. Little energy is required to change these systems by 100 kPa. There are a few devices in the vacuum space that may be damaged due to pumping out too fast but none that are considered a danger to personnel.

Other than venting too fast the second point to consider is the introduction of water vapour if venting with air. Key vacuum systems on the accelerators are vented with either dry nitrogen or argon both of which are delivered through a closed reticulation system from gas bottles. The choice of gas is dependent on molecular contamination within the system for example nitrogen is not preferred to vent the 846 ion source as nitrogen is an element that is known to bond with other elements causing molecular ion beams.

#### Regulation of Flow

There are many low flow regulators and rotameters (tubular flow meters) available that provide adequate control over venting. A simple in-house gas regulating system which works well is a tee piece connected between the venting port and the venting gas supply line. The centre port of the tee piece faces upwards and a ping pong ball sits over the hole with a cage over it so that it cant be blown away. Under the ball is an 'o' ring seal. Before the venting valve is opened the venting gas is allowed to flow. The pressure of the gas lifts the ping pong ball of its seal. The gas flow is adjusted depending on the height of the ball above the seal. The ball can only travel a centimetre or so. Once the flow is set the venting valve is opened and the now regulated gas flows into the vacuum space and the balls drops onto the seal stopping air from entering the vacuum space. As the pressure reaches equilibrium the ball begins to rise off the seal until eventually it rises to the original set height indicating the vacuum space is now at atmospheric pressure.

#### Precautions

- Before venting a vacuum system it is wise to isolate all ports and beam lines that must remain under vacuum. To save stressing a beryllium window on an x-ray detector due to the changes in pressure the system should be designed so that the x-ray detector can be isolated with a gate valve. The same logic can be applied to all equipment that may be effected. It is also important to ensure all power supplies and high voltages that may be hazardous to personnel or equipment are isolated prior to venting. This can be done either by administrative protocols or using a vacuum switch such as programmable relays in a vacuum gauge controller.
- Some vacuum gauge controllers use the signal from a Pirani gauge measuring at the same location as a high vacuum gauge to switch off the high vacuum gauge when the pressure becomes too high. This saves the gauge from ionising air when the system is at atmospheric pressure which will significantly reduce its life time.
- Another problem with venting too quickly is the disturbance of dust. Accelerator tubes must be vented slowly to minimise equalisation stresses and also the disturbance of dust. In this case a metering valve is used to limit the vent flow rate.
- Venting can cause loud high frequency noise so care must be taken to dampen the noise to a comfortable level by controlling the air admittance rate, or use hearing protection.
- Most beam line vacuum pumping systems are interlocked to isolate either the beam line or pump or both. In the event where an accidental venting occurs the gate valves will close minimising the effect on surrounding systems.

# 5. Safety

#### 5.1 General

Catastrophic failure causing injuries are not common and no reference is known from overseas accelerator facilities. This is almost certainly due to the low pressures involved in vacuums and the size and nature of the scientific apparatus. There are of course many failures reported for industrial sized tanks and equipment.

The vacuum systems are essentially fail-safe from absolute vacuum to atmospheric pressure. The worst case has been a number of beam lines and measurement chambers developing leaks and thin windows that have ruptured. In each case there has been a gradual rise in pressure to atmospheric pressure.

However, a few rules do apply.

- All vacuum systems must have one or more vacuum gauges attached with the exception to some static chambers used for storing moisture sensitive items or radiation detectors.
- High vacuum gauges must be switched off before vacuum systems are vented to atmospheric pressure.
- Only nitrogen, argon or other inert gas to be used for venting. No flammable gases to be used such as hydrogen.
- Turbomolecular pumps must not be vented from full speed. See manufacturer's recommendations.
- All designs must be consistent or better than the best practises outlined in this document. Inferior equipment must be isolated and reported to the Leader of Accelerator Operations.

- Cryopumps must not be operated without a serviceable pop off valve.
- Cold traps must be warmed and blown dry at least every 6 months to minimise  $O_2$  concentrating (and for de-icing as necessary).

# 5.2 Interlocks

Nearly all vacuum systems on the accelerators are interlocked via a set of integrated relays in a vacuum gauge controller, to protect equipment. Generally, if a rise in pressure is detected above a particular set level then the interlock relays will activate to close corresponding gate and line valves. This is designed to minimise the number of vacuums that must be regenerated in a fault. The interlock system also protects accelerator tubes from exposure to moisture and in the event of a catastrophic failure of the accelerator tubes; the vacuum system will close the main accelerator gate valves to trap SF6 gas.

If an interlocked failed causing a gate valve to stay open in the event of a pressure rise then the next interlock should activate the next gate valve. If the next interlock fails it is possible that the two interlocks share the same vacuum gauge controller which is at fault. In that case the next interlock should activate. If no interlocks activated there would be no increased risk of injury to personnel but all vacuum systems will require regeneration or a restart after the interlocks were repaired and tested.

## 5.3 Fumes from exhausts

There are a number of oil vane pumps in use on the accelerators. In the past, the exhaust on the pumps has released oil mist into the atmosphere inside the buildings. Now, on nearly all oil vane pumps in continual service there are mist filters attached or the exhaust is plumbed into an extraction system that vents the exhaust fumes outside of the building.

### 5.4 Earthing

Some vacuum gauge controllers are prone to high voltage transient surge damage. These sometimes cause unnecessary isolation of systems requiring resetting. It is very important that all vacuum gauge controllers are well earthed.

# 6. Serviceability

#### 6.1 Access

Most beam lines with the exception to some measurement chambers are static volumes of vacuum requiring little operator intervention other than during preventative maintenance. Vacuum pumping systems do require a higher level of intervention during maintenance and operation. With these points in mind the designer must ensure that all controls and monitoring devices are localised at the working face of the beam line or chamber.

Due to beam lines being so high above floor level, working on them usually requires some form of ladder or steps. When designing beam lines, be sure to have pump out ports facing the passageway beside the beam line. The same applies to vacuum chambers and other large volume vessels.

Measurement chamber will need to have the access port accessible from a location near the control side of the chamber. A platform is recommended to allow easy reach and eay viewing into the ports.

#### 6.2 Maintenance

A well designed vacuum system will require routine maintenance including:

- Changing faulty seals cleaning seals that are regularly opened
- Cleaning vacuum gauges especially cold cathode type high vacuum gauges
- General cleaning inside of measurement chambers and equipment that is; regularly opened and touched, whenever it looks dirty, when the base vacuum degrades or when the surfaces are exposed to the atmosphere for long periods at a time (weeks).
- Servicing of vacuum pumps the group of the most routine maintenance tasks includes; cryopump regeneration, turbo pump bearings, scroll pump tip seals and oil vane pump oil changes to name a few
- Actuator service sliding shafts that penetrate into the vacuum space

#### 6.3 Cleaning

Cleaning is by far the most essential part of general maintenance of a vacuum system. Poor cleanliness will lead to poor vacuums and possibly sample contamination. It is a general philosophy that cleaning is done with solvents, wiped over and/or scrubbed and thoroughly dried.

#### **Solvents**

Typically ethanol, methanol and acetone are used. These solvents remove most greases and oils that are encountered. What ever solvent is chosen the most crucial point to remember is that it must not leave any residue.

For surface finishing see section on 'Surface Finishes'.

Chemical polishing is not commonly used in the accelerator area due to the size of the baths that are needed for large parts, the problem of cleaning or neutralising residual chemicals and not having a suitable facility for handling the types of chemicals used. Although once used electro-polishing is no longer encourages for the same reasons. If suitable facilities were available both chemical and electro-polishing are real options for very good cleans.

A general clean will consist of a number of different levels of cleaning. A single clean with the same solvent will not remove some oils and greases therefore a progressive approached is used using a variety of solvents and wiping materials. The following is a guide only of some such techniques.

Using a dry gas or filtered compressed air, most loose dust can be blown away. This is sometimes the best starting point for cleaning. A final blow down is also useful to ensure complete removal of lint.

The first clean will take away coarse particulates or chemicals. Ethanol dampened tissues if good for this level of cleaning. The ethanol helps remove some oils and greases and mixes with water to reduce the evaporation time. It is also good for mopping up dirt and dust. Scour pads (or 1200 wet or dry paper) moistened with ethanol is good for removing stubborn dirt. This must be continued until better than 95% of dirt, dust, oil and grease is removed. For stubborn dirt it may be necessary to use a petroleum based solvent followed by an ethanol rinse and wipe. Wiping is key to cleaning. The light mechanical brushing dislodges most dirt.

The second clean starts with ethanol but finishes with acetone. Only lint free wipes are used such as Kimwipes. At the end of this level of cleaning the equipment must look ready for use in the vacuum. That doesn't mean it is. It will more than likely have residues remaining on the surface.

Final clean. It is recommended that methanol is used for the final clean or another low residue solvent. The final clean must use lint free wipes dampened with methanol and lightly rubbed over the entire surface. An alternative final clean is to use the solvent bath and suspend the equipment in the hot vapour for a few minutes. Care must be taken with the bath as the hot vapour will degrease skin very quickly.

If a shiny (healthy looking) clean is desired then a metal polish such as Brasso can be used. Whenever Brasso is used the residue must be cleaned off with ethanol. Care must be taken not to get metal polish pastes into gaps that cannot be thoroughly cleaned out. Brasso is an enemy of vacuum, it traps water.

#### 6.4 leak detection

In the accelerator area, a leak is characterised by a flow of gas molecules into a space that is below atmospheric pressure. The pressure of the gas entering the space will restrict the vacuum system from reaching its design pressure which is typically in the high vacuum range. As a benchmark, if a vacuum is  $1 \times 10^{-5}$  Pa or more then disregarding the effects from outgassing, there may be a leak. In system designed for ultrahigh vacuum the benchmark will be in the order of  $1 \times 10^{-7}$  Pa.

Leak detection is usually only necessary for new chambers or equipment. It can be done offline or after the part has been mounted into a beam line. The most common method of leak detection is a helium leak test. Helium atoms being so small will find their way through openings that are too small for most other molecules. Generally, the smaller the leak, the less helium that can make its way along a leak path. The leak detector is usually a mass spectrometer tuned to have highest sensitivity for helium. The detector is connected directly to the device being tested. Any helium that makes its way through the leak will eventually be measured in the mass spectrometer. Residual Gas Analysers (RGA), although not specifically leak detectors, can be used for just that. Working on the same principle, mass spectrometry, the RGA can detect helium as well as other gases.

In some cases leaks can be too large for a helium leak test, that is, the amount of gas in the device being tested is too high diluting the helium to an ineffective percentage. Also, if the pressure is too high the leak detector cannot be opened to the device as the pressure gradient will be too high. Leak detectors work best from the higher end of the low vacuum range.

Often there are multiple leaks in systems. It may mean repairing large leaks in order to find small leaks. Where a leak is too large for the helium leak detector a few other methods can be used to locate leaks.

- Solvents have a very low viscosity and vaporise very quickly in air. Wetting surfaces with small amounts of acetone or ethanol can show small changes on Pirani and Penning gauges.
- Isolation of seals can, by process of elimination, restrict the testing area allowing large leaks to be isolated. For example, if a chamber with many ports and devices attached has a leak. It may be prudent to close all valves leading away from the chamber except for the leak detector valve then sequentially blank off the ports one at a time (using a sound flange and new seal) and test for a leak.

# 7. Soldering wires vs mechanical joints

It is preferable that wires in a vacuum space are joined together with a mechanical clamping type joint. The clamp must allow for gas to escape. Where a direct joint is necessary then soldering is permitted. Standard lead/zinc with resin core is okay provided that ALL flux is removed with ethanol. This may require some soaking in solvent with gentle agitation. Solder without resin core

can be used on stainless steel with a zinc chloride based eutectic flux, used sparingly. Ensure all flux is removed especially between the wire strands.

Be aware that in Faraday cups the continual flexing of wires can work harden the wire causing it to break. These are typically soldered joints. If soldering, be sure not to melt the insulation. It is highly recommended to replace normal plastic insulation with loose fitting PTFE (Teflon) spaghetti.

# 8. Flanges and seals

## 8.1 Common vacuum seals used in the accelerator area

A variety of standard vacuum seals are used on all vessels depending on the degree of vacuum to be achieved and the type of fitting that will be used. The types are widely used in the vacuum industry. ANTARES uses the Dependex style seals and flanges. This was developed for use with HVEC accelerators over 50 years ago. Other types in use include Conflat, ISO, and KF are used as well.

Туре	Vacuum level	Flange type	Seal type
KF	Low to High	Clamp (typically)	Viton 'o' ring, Al, PTFE
Edwards	Low	Threaded retaining nut	Viton 'o' ring
Dependex	High	Ring retained by spring clip	Viton 'o' ring supported by a ring
LF or ISO	High	Ring or clamps	Viton 'o' ring supported by a ring or in a groove
Conflat	Ultra high	Ring fixed or rotating	Copper ring, Square section 'o' ring
Specials	Various	Various	Aluminium or indium wire

Table 1. Common types of vacuum flanges and seals. Each type is available in multiple sizes

# 8.2 KF – Klein Flange

A common flange and seal used for general low vacuum applications due its ease of assembly and vacuum performance. It is common to have KF flanges and seals within the high vacuum side of a system. Vacuum performance can be enhanced by the use of aluminium or PTFE seals which directly replace the Viton 'o' ring.

Many general and specialised vacuum equipment is fitted with KF fittings as the range of adaptors from KF to other major types is readily available.





Fig 3. Assembly drawing for a typical KF flanges seal and a photo of the flange, seal and clamp

### 8.3 Edwards

This is no longer a preferred type that is used in the accelerator area however some equipment still uses this type.

One end of the fitting is shaped to retain an 'o' ring while the other end has an angled sealing surface that mates and crushes the 'o' ring to make a seal. They are connected by a threaded nut which when tightened squeezes the two halves together. It is normally for low vacuum applications.

#### 8.4 Dependex

This was designed for use on HVEC Van der Graaff accelerators and beam lines. It was introduced to ANSTO in the early 1960's when the 3MV Van de Graaff operated in B22. Here, 1", 2" and 4" Dependex was used. When ANSTO purchased the FN Tandem nearly all fittings used were 4" Dependex (100 mm nominal). It is suitable for high vacuum use and often mated with Conflat flanges in systems that operate near ultra high vacuum.

The Dependex seal consists of a supported 'o' ring sealing between two opposing  $5^{\circ}$  metal faces. The 'o' ring is supported around its inner diameter by a ring to ensure the 'o' ring aligns with the centre of the 2 faces. The inner ring also sets the maximum crush on the 'o' ring that is the ring fits into a step on both halves of the Dependex seal keeping them separated at a fixed distance. Another ring which fits on the outer diameter of the 'o' ring does little more than keep the 'o' ring clean. The rings are often called *egg rings* due to their resemblance.

The Dependex flanges are held in place on a tube, or other fitting that is designed to accept Dependex, by a large circlip that fits into an outer groove a few mm from the end of the sealing face.

The flanges are normally drilled such that the holes alternate between being tapped or clearance. 1" and 2" Dependex use <sup>1</sup>/<sub>4</sub>" UNC, cap (socket or Allen) head screws and 4" (100 mm) Dependex use 5/16" UNC, cap (socket or Allen) head screws. Only use stainless steel screws (of one length) and washers. Discard plated steel screws as they are identified during maintenance. Washers are recommended as most flanges are aluminium alloy.

To complete the Dependex assembly, the flanges must be placed over the tube ends and then retained in place with a circlip. Secondly, the 'o' ring is stretched onto an inner ring and then an outer ring is placed around the assembly. The two Dependex ends are brought close together then the seal assembly is placed between them. The inner ring is aligned with the step on one side then the other tube is aligned and than pushed onto the ring. At this point it is important to ensure the 'o' ring assembly stays in place until the bolts are tightened. Screws should be finger-tightened and the flange faces must be parallel. Tighten screws in an opposite pattern. Some old flanges will have worn threads so screws may only be turned with the aid of an Allen key.

#### Problems

There are a number of things that can go wrong with assembling Dependex fittings.

- There have been cases where inner egg rings have been made too wide so that when fitted between the tubes the 'o' ring is prevented from crushing adequately to form a vacuum seal.
- Another problem with the inner rings is where some Dependex seals have been designed with too shallow steps so that a standard inner ring becomes too wide. In this case special inner rings have been made which are narrower. Personnel servicing non-standard joints must take care to reuse the right sized inner ring. A label should be placed on all non-standard joints identifying them.

- Some steps for inner rings have also been found to be out of tolerance preventing standard size inner rings from locating in the step. In this case some operators have cut a small section from the inner rings so that they can be squeezed together. Cutting inner rings like this is only recommended to solve this type of problem.
- Some flanges have been design for special applications with slightly different PCD's. These have unfortunately bee mixed up with standard stocks. As personnel who identify them (sometimes after a frustrating encounter) should put them aside and label them accordingly.
- Old flanges that have been over-tightened may have stretched threads. They are identifiable by the difficulty in starting screws. Either re-tap the threads or discard the flange. There have been case were tight threads have mislead personnel into thinking they have tightened the Dependex joint only to find leaks.
- Circlips can stretch especially those in over or under sized slots. They are easily identified by there distorted shape. Only use circlips that are flat when not under tension. A discerning technician should discard rusty circlips that cannot be successfully cleaned.
- Care must be taken to make sure the two faces of the Dependex joint are parallel before the joint is closed and bolted together. Failing to do this may cause a leak in the joint. It is acceptable if a bellows is used between sections that have misalignments.
- Twisted 'o' rings can cause leaks although small. There have been cases where a twist has caused the 'o' ring to bulge towards the outer ring making a less than adequate seal.

# 8.5 Special Flanges

These consist of flanges that have been designed to fit application where "off-the-shelf" flanges and seals cannot be used. They are typically based on common designs but modified to suit the application.

#### Example

The Alphatross ion source uses two stainless steel flanges to hold the heater reservoir in place. Here the vacuum sealing surface is a flat surface about 8mm wide on each flange. The fixed flange is threaded in an imperial thread and the reservoir flange is open hole. The gasket used is aluminium wire which has been fused together to make a ring. The joint is similar in size to the wire. The wire is supported in place using about 3 to 4 strips of aluminium foil looped around the wire and sticky taped to the edges of the flange. The flange is tightened in a circular pattern to ensure maximum crush on the wire.

# Problems

- The wire is essentially unsupported so vacuum must not be applied until the wire is completely crushed.
- Bulbous joints in the aluminium wire dent the sealing faces on the flanges as they are tightened so they must not be used.

# 8.6 ISO or LF (Large Flange)

This is a European range of fittings designed for high vacuums and to suit most tube sizes used in the vacuum industry. There are two types of design which meet most light and heavy duty applications.

ISO-K flanges are a robust joint which are suitable for supporting larger and heavier items such as vacuum pumps and large gate valves. They are characterised by the outer clamping system which holds the two halves of the joint together.

ISO-F flanges are more of a traditional bolted flange type joint like Dependex. ISO-F and ISO-K can be joined together using a half clamp which fits onto a standard ISO-F flange. The half clamps then attach to the ISO-K flange.

Available in sizes from 63 mm nominal bore to 500 mm NB.



Fig 4. ISO family of Flanges

#### 8.7 Conflat CF

Conflat is designed as an all metal vacuum seal for ultra high vacuum applications. A copper gasket is sandwiched between two thick stainless steel flanges that have a knife edge machined into the sealing face. As the flanges are tightened together the knife edges "bite" into the copper to produce an all metal seal with very low outgassing.

The term "ConFlat" is a registered trademark of Varian, Inc., so "CF" is commonly used by other flange manufacturers. Conflat sealed systems can reach vacuums as high as  $1 \times 10^{-11}$  Pa.

The copper gasket locates into a partially recessed in a groove in each flange to provide alignment prior to tightening. It also stops the gasket from moving during bake out. Conflat can be baked up to  $450^{\circ}$ C.

Most Conflat flanges have imperial sized bolts. The original style of bolt had a multi-face head that a ring spanner would fit over. This is to allow more purchase on the head from many angles. It is not uncommon now to find hexagon and socket head screws in use. Be aware that the steel must be a high tensile grade to ensure optimum force can be used to crush the gasket. It is essential that the flange faces are parallel during and on final tightening. A torque wrench may be used to facilitate uniform tightening. It is highly recommended that a molybdenum grease or graphite loaded grease is used on the threads.

Conflat flanges are available in sizes from 10 mm nominal bore to 250 mm NB. Sometimes nominal bore is written DN in front of the size, eg DN63.

Square section Viton 'o' rings may be used with Conflat flanges however this compromises the ultra high vacuum design and ultra high vacuums may not be reached.

## Problems

- Copper gasket not aligned in groove and edge is partially clamped on the bolt hole face of the flange. This lead to partial seal and most likely, a leak. Most Conflat flanges have two small grooves a little more than a millimetre wide machined into the bolt face. These are for locating special clips that hold the gasket in place while the flanges are brought into place. Just prior to tightening they clips are removed. They are sometimes mistakenly called pressure relieving grooves.
- Copper gaskets must only be used once. Unless the copper hasn't been fully compressed after the first use there will not be enough material for the knife edge to reform a seal.
- Due to the forces required for a knife edge to indent a copper gasket the flanges are made of stainless steel and quite thick. Large flanges are quite heavy and care must be taken not to drop them.
- Where Conflat flange bolts have been used more than once the threads in the flange and screws may have stretched. In this case, it is recommended that the same screws are reused in the stretched threads (with a little molybdenum grease) as they will have the same profile. Sometimes using new screws in a stretched thread will cause binding. Re-tap the flange if necessary.
- Stretched threads are possible where nuts and bolts are used through open holes so ensure the nuts go onto the bolt the same way they were removed. Alternatively replace the nuts and bolts outright.
- For ultra high vacuum ensure the gaskets are handled with gloves. Copper reacts well with the sweat in a fingerprint leading to a long outgassing process.

# 9. Bellows

Bellows consist of two styles, edge welded for ease of extension or compression and corrugated style which is has minimal movement. Each style is made from a thin stainless steel that has been electron beam welded. The ends are usually finished in a standard flange to suit the application.

#### 9.1 Corrugated style

These are typically used for two purposes. Firstly, to allow sections of beam lines or equipment to be compressed to allow easy removal of sections without dragging the sealing surfaces against each other. Secondly, as bellows can distort and still maintain their structural integrity they are ideal to

join sections of beam line or equipment that have a deliberate mis-alignment. They can be purchased with almost any style of flange including specials. See example pictures.



Large bellows. ~300mm diameter. Stainless steel, wall thickness ~ 0.8 mm. This bellows is used to correct alignment offsets in the magnet box

Medium bellows. ~ 100 mm diameter. Stainless steel. wall thickness <0.5 mm. Used to allow sections of the beam line to be compressed to facilitate beam line component removal.



Small bellows. Electron beam edge welded stainless steel. Material thickness <0.25 mm. Used to isolate vibrations in the beam line.

Fig 5. Examples of bellows

#### 9.2 **Edge Welded Bellows**

These bellows are made up from very thin stainless steel discs stacked together and welded along their edges. The advantage of the design is the concertina style bellows that is very flexible allowing designs that have long extensions and short compressions. They have two main applications. Firstly, applications that requires a device to extend or contract into the vacuum space where an elastomeric seal on a sliding shaft is not adequate. Such as a sample manipulator or Faraday cup. Secondly, where a device such as a detector or sample positioner is sensitive to vibration and must be decoupled from the vibration source.

# 10. Valves

Along the axis of the beam lines gate valves are used to enable the isolation of sections for maintenance and also to minimise loss of vacuum in the whole accelerator if one section develops a leak. Most gate valves are electro-pneumatically operated and many are interlocked to local high vacuum gauges. The few that are manually operated are for maintenance isolations.

On ANTARES the electro-pneumatically operated valves are interlocked to close when the pressure, as measured near the gate valve, rises above 5 x  $10^{-5}$  Pa. This figure can vary depending on the location of the valve. See the Accelerator WIKI for values around the ANTARES. Once an interlock has tripped the switch that operates the gate valve is bypassed. To reset the valve:

- The vacuum must be within the high vacuum range as read on the corresponding vacuum gauge controller with a pressure better than the trip point.
- Once this level is reached the bypass button is held down momentarily.
- At this point the pressure will rise then fall as the gas load is pumped away.
- When the system pressure is less than the trip level the bypass switch can be let go.
- An indicator light near the switch will show its status.

#### Notes:

- Operators and technicians must be aware that gate valves must be orientated so that the gate always seals towards the vacuum pump. That is, they seal best in one direction only. Failure to orientate the gate the correct way may cause a vacuum leak when atmospheric pressure forces the gate off its seal.
- In some cases gate valves may remain sealed after they have been switched to open. This can be due to the withholding (sometimes called back pressure) pressure not being adequate enough to lift the gate off the seal.
- Gate valves, while mostly metal can become hot if a gate is closed in front of an ion beam. If left for long periods with high current beams the elastomer seals may warm up and begin to outgas.
- For roughing ports typically 25KF bellows sealed valves are used. On STAR these same valves are used to isolate vacuum gauges for cleaning.
- Where fine flow control is required for either roughing out or venting, metering valves are used. These are basically multi-turn needle valves for fine adjustment.
- Valves do require maintenance to keep the seals clean and in working order. After long periods of time the 'o' ring seals will deform and often will not recover their original shape.

There have been attempts to build fast acting gate valves for the purpose of isolating the accelerator tank's load of  $SF_6$  in the event of a breach in a tube. While it seemed like a good idea the force of the valve closing at high speed could damage the gate seal causing the valve to leak. Designers must use care to ensure that a standard high differential pressure rated gate valve isn't adequate before launching into these expense type valves.

# **11. External Interfaces**

#### **11.1 Electrical Feedthroughs**

a) Low Voltage, Low Current

There are few applications where voltages greater than 24Vac are used within the vacuum space. For these voltages at a few amps coaxial feedthroughs such as BNC, SHV, MHV, etc, are often used in place of more expensive dedicated commercial types. In one application 200Vdc is used for faraday Cup suppressors but current is only a few micro-amps at most.

Notes:

• Do not use multi-pin signal feedthroughs to carry voltages unless the pins used for voltage can be a few unused pins away from used signal pins.

- Feedthroughs can be purchased is almost any flange style but are usually restricted to small size flanges.
- Insulation must be used on all wires carrying voltage. It is highly recommended to use single or stranded wires (preferably nickel coated) with loose fitting PTFE (Teflon) spaghetti.

### b) Signal

Signal feedthroughs other than coaxial such as BNC, SMC, Microdot, etc are from one to multiple pins. It is recommended that connections to multi-pin feedthroughs are by the use of slide on pins. Many are either sliver of gold plated for good electrical contact. Avoid soldering unless care is used to remove all traces of flux and no pin-holes in the solder have been created

On coaxial feedthroughs the coaxial connector will be either both sides of the feedthrough and hermetically sealed or with the coaxial connector on the atmospheric side and a plain pin on the vacuum side, also hermetically sealed. For either type, where coaxial cable must continue into the vacuum space it is recommended to remove the outer plastic insulation and replace it with loose fitting PTFE spaghetti. This is also recommended for all single or stranded wires carrying signals.

## c) High Voltage, High Current

There is a wide range of high voltage and high current feedthroughs available in most flange types. High voltage feedthroughs usually have a larger insulator in ceramic and sometimes glass. These must be kept clean on the atmospheric side. Long term breakdown on high voltage feedthroughs can damage the insulator causing vacuum leaks.

For high current applications such as heaters, dedicated high current feedthroughs must be used. These are characterised by larger cross section wires to carry the higher currents. Like HV feedthroughs they must be kept clean. It is recommended to clean the insulators on signal feedthroughs periodically to remove any accumulation of dust.

#### d) Power

There are few feedthroughs designed for powering devices at mains voltages. It is highly recommended that experiments are designed not to have mains voltages in vacuum vessels or equipment due to safety concerns.

# **11.2** Mechanical Feedthroughs

#### a) Rotary

Rotary feedthroughs allow the transition of rotary movement into the vacuum space to, for example, operate shutters or position samples or detectors. They are widely available in many of the common flange styles. There are three common types both of which are very good in high vacuums.

The first type is a continuous shaft between the atmospheric side and vacuum side using rubber vacuum seals in the form of either supported 'o' rings, square or odd shaped rings (like common shaft seals), and flat rubber sheet seals. These are best for general rotary actuation rather than continually rotating. Okay in high vacuum but care must be taken to keep the seals clean and periodically replaced or whenever wear is noticed.

The second type is a ferro-fluidic type which also uses a continuous shaft between the atmospheric side and vacuum but the seal is made by encapsulating a dense fluid around the shaft. These types usually have bearings to keep the shaft centred and free moving. They are best for high revolutions in high vacuum applications.

The third type is magnetically coupled rotary feedthroughs. These are expensive. They provide a seal by magnetically coupling the shaft on the atmospheric side to the shaft on the vacuum side across a continuous sealing plate. If either shaft is removed the vacuum is not compromised. These are excellent for ultrahigh vacuums and lower. They are for moderate speed revolving applications and general actuations.

### **b**) Sliding

Usually these feedthroughs are a round shaft that can slide in and out of the vacuum as well as rotate. Sliding feedthroughs are used for positioning devices or where a linear actuation is necessary. The length can vary from a few millimetres to hundreds. In either case there is a shaft which penetrates from the atmospheric side to the vacuum side. The type of seals used varies like in the rotary seal. Sometimes dual seals are used to minimise leakage especially in high actuation rate applications. In some applications the void between the two seals is pumped increasing the reliance of the seal. These types are good in high vacuums. Notes:

- Care must be taken to ensure the shaft is lubricated with a suitable vacuum oil or grease. It is imperative that the oil or grease on the shaft is changed regularly to minimise dust build.
- There are bellows sealed sliding seals available. These types do not rotate and can only move in and out a few tens of centimetres at most. Very good in high to ultrahigh vacuums.
- Some sliding seals are able to move radially which will affect alignment. Where alignment is crucial a linear bearing type is recommended.

#### c) Wobbler

A wobbler feedthrough allows users to use a shaft as a lever or pick up. They are designed around a flexible rubber or bellows seal they allows a greater range of circular movement.

#### 11.3 Manipulators

These are a class of feedthroughs design for high to extremely high precision positioning of samples, detectors or other instruments. They can be purchased having all or either X, Y, Z and rotary movement. The X and Y axes can have various travel lengths however they are typically less than 100 mm each. The Z axis (vertical) can have greater lengths but is limited as with the X and Y axes by the physical limitations of the vacuum bellows.

#### 11.4 Actuators

'Actuators', combine sliding and rotary feedthroughs with pneumatic, electro-pneumatic or mechanical actuators to do the work.

In the accelerator area, actuators can be found on Faraday Cups to insert the cup into the beam line on a bellows sealed sliding feedthrough and inside of the ANTARES pressure vessel to move the stripper tube in and out of the beam axis.

#### 11.5 Load locks

Load locks are not common in the accelerator area but are ideal for moving samples into a high vacuum space without significant loss of vacuum pressure. A sample or device is mounted onto the end of the load lock shaft. It is inserted into a tube connected to a chamber but isolated from the chamber vacuum by an in-line gate valve. The tube is sealed and then evacuated to a vacuum pressure similar to the chamber. Once the pressures equalise the gate valve is opened and the shaft pushed into the vacuum space. From this point the sample or device can be removed from the shaft by other remote means.

# **12. Internal Devices**

There are many devices that are commonly used inside of vacuum systems. Not all are represented in this section however similar reasons are use to assess new and unusually devices for use in vacuum.

## 12.1 Lighting

Lighting is necessary in most sample measurement chambers where the operator must be able to see the sample in order to position it in front of the ion beam. In most cases a standard bare 12Vac halogen bulb is used with good results. Care must be taken to ensure the bulb can be dimmed as when running at maximum power the heat load can only be dissipated through the connecting wires. That is, there is little gas inside a vacuum to conduct head away from the bulb. Bright white LED's are also used with mixed success. They have less radial light output as compared with a bare halogen bulb. If the LED is setup correctly the light can be focussed towards the necessary target.

Preferably, wires must be stranded or solid wire with a loose Teflon sleeve but normal plastic coated hook up wire works in high vacuums. In some cases where the wires as not likely to come into contact with other components then they are used uninsulated. All wiring must be restrained so that it cannot move into the path of the ion beam. As a rule-of-thumb wires should be at least 20 mm from the beam.

A standard double BNC feedthrough can be used, that is, only the centre core connections are used. There are many off-the-shelf electrical feedthroughs available that will be more suited for this specific use.

## 12.2 Heating

Providing power to a heating stage on a sample (target) stick requires a high current feedthrough. If a feedthrough isn't provided from the supplier of the heating stage then many types can be found from larger vacuum suppliers. Ensure the feedthrough selected is designed to carry the maximum current that can be supplied and not the maximum current likely to be used. Like in the lighting section above, care must be taken to ensure wires are insulated and away from the beam path.

Heating stages rely on heat dissipation through the support for the stage which is usually made from stainless steel, a relatively poor conductor. When opening devices for handling be sure they are cool to touch first.

#### 12.3 Cooling

Cooling stages come in 2 distinctly different types. The first is the Peltier cooled stage which is all electric. Powering the stage can be managed as for lighting and heating. The second type is a liquid nitrogen cooled system. It uses an insulated tube to carry LN2 through a feedthrough into the vacuum space. A typical design is where LN2 is pumped through a coil attached to a heavy copper braid. The braid is then connected to the cooling stage where heat is conducted away from the mounting stage. Thermal isolation of the cooling stage from the holder can be Teflon or sapphire standoffs or similar materials.

These cooling devices must only be switched on when the vacuum is at the working level otherwise condensation and possibly ice can form on their surface becoming a large outgassing problem. Likewise ensure the stage is close to room temperature before opening up the vacuum system to atmospheric pressure. Always vent with a dry gas.

# **13. Pumping**

# 13.1 General

It is not the intention of this document to describe the fundamental operation of particular vacuum pumps. Adequate information is available from manufacturer's catalogues. The following is information on what type of systems are used in the accelerator area and the reasons why.

Vacuum pumps don't suck. They act essentially as one way valves in a system where gas is flowing towards them. This is discussed in Chapter 3 of this document. There effectiveness is there ability to stop recoiling gas molecules returning into the vacuum space such as a beam line or vacuum chamber.

When considering what type of pump to use for a vacuum system the following points must be considered.

- What is the application?
- Will it have a relatively high gas load, that is;
- Will it be pumping a static gas load or changing gas load?
- Will there be a high outgassing rate?
- Are there known hidden/trapped voids to be pumped?
- It is a clean or dirty device? Is it contaminated with grease and oil?
- What pumping speed will be needed to maintain a high vacuum?
- What is the volume of the system? What is the conductance of the system? Will a small pump be just as effective as a large pump?
- What gases will be pumped?
- What type of pumps and how many are being used in the vicinity?
- Will the pumping speed need to be regulated such as for pumping near foils or delicate samples, detectors, etc?
- Cycling time whether it will be required to regularly pump from low vacuums
- Will the pumps operate reliably in their chosen application?
- Are they compatible with other pumps used on the accelerators? Are they cost effective?

Another consideration is vibration. Pump vibration can be significant leading to micrphonic interference in detectors and vibration of samples being measured. Vibration can be reduced by selecting pumps with less inherent vibration like turbomolecular pumps or the more fickle ion pumps. If a pump is a source of vibration it can be decoupled from the detector or measurement chamber by the use of a flexible bellows. An fine edge welded bellows is recommended.

# **13.2 High Vacuum Pumps**

See section 4.5, Pump Down characteristics for cryogenic and turbomolecular vacuum pumps.

#### <u>Cryopumps</u>

For ANTARES the main beam line pumps in use are cryopumps. The types of pumps trap (or immobilise) gas molecules. They are used because they have a long lifetime between major

services, can be regenerated without removing them from service, have a high pumping speed for most gases and can maintain high vacuums for long periods in a static vacuum application.

#### Turbomolecular pumps

Where a vacuum system is cycled such as at a measurement chamber, then turbo pumps are used. Turbo pumps are transfer type pumps meaning, gas is transferred from the vacuum space into the atmosphere. A turbo pump can routinely recover a vacuum from low vacuum after a measurement chamber has been roughed out without needing regeneration as for a cryopump.

Turbo pumps are also more effective at pumping lighter molecules such as helium and hydrogen as compared with cryopumps. Using turbo pumps near systems that use these gases will reduce the effect of loading on cryopumps.

Most new turbo pumps can be serviced in-house. Some models purchased require special balancing of the bearings after they had been changed. In this case they are sent back to the manufacturer at great cost. It is important that models chosen can be maintained in-house.

#### Other high vacuum pumps

There have been few applications where titanium sublimation or ion pumps have been used in the accelerator area. Most have been stand alone UHV systems or cryostats for specialised applications. The practicality of cleaning and regenerating these gas trapping type vacuum pumps on systems requiring relatively frequent cycling is prohibitive.

Getter materials once used widely in electronics in tubes are becoming more used within larger static volumes such as beam lines and associated equipment. They can be coated onto surfaces to assist in maintaining uniform high vacuums in spaces that have poor pumping speeds.

#### **13.3** Low Vacuum, Roughing or backing pumps

#### Oil free pumps

These pumps are categorised by either being an oil type pump or dry (oil free) pump. Oil free vacuum systems are of choice for accelerator systems now that the technology has been proven to be reliable and cost effective. The majority of roughing and backing pumps now in use are scroll pumps as well. Piston pumps have been trialled with little success due to the short life of the seals. Some applications have used diaphragm pumps and multistage roots pumps but scroll pumps remain the most cost effective and easiest to maintain option. The Edwards type scroll was chosen over other brands as this type of scroll pump is designed with the bearings mounted outside of the vacuum which in other brands often caused the bearings to fail from lubricant loss.

#### Oil vane pumps

Low Vacuum, Roughing or backing pumps are all the same type of pump. Oil vane type pumps were widely used in the accelerator area until 2006 when a systematic change began on the STAR accelerator to replace oil vane pumps with oil free scroll pumps. Some oil vane pumps remain in use where some back streaming oil vapour is not considered a problem. If an oil vane pump is to be used for roughing out purposes, it is high recommended that a liquid nitrogen cold trap is used to trap vapours. It is the ultimate aim to have oil free pumping on all accelerators in the future.

# 14. Monitoring

#### 14.1 Vacuum Gauges

The standard type of high vacuum gauge in use is the cold cathode penning type gauge.

- On ANTARES the Pfeiffer brand is in use in two versions; the standard cold cathode type and full range type which has an integrated Pirani gauge.
- On STAR the most widely used brand is Leybold.

Generally, vacuums are measured as an indication only for process controlling and establishing basic quality to ensure pressures are within levels that are accepted for "normal" operation. This varies depending on location and use of the vacuum system for example a measurement chamber is cycled often and affects surrounding vacuum systems. It will more than likely be a much high pressure than surrounding static systems.

In most cases, absolute vacuums are not required to be measured. However on some AMS beam lines where thin windows are used it is important to have accurate measurement of pressures when cycling pressures in the gas detectors. If accuracy is required it must be remembered that different gases ionise at different pressures and so pressures readings from the same volume will be different depending on the dominant background gas. Most gauges have characteristics related to Nitrogen (air).

#### **14.2 Gauge Controllers**

On ANTARES the most widely used gauge controller is the Pfeiffer TPG300. This is an aging model and is being replaced in some key areas with the Pfeiffer Maxi-gauge which allows 6 heads to be connected at once.

The TPG300 has inputs for up to 4 heads whether Penning or Pirani. The limitation with the TPG300 is the electrical robustness when operated near the accelerator high voltage generator. Many failures have been attributed to high voltage transient surges. The TPG300 is now technologically aged and will ultimately be replaced outright. Various other models of TPG's are in use on ANTARES especially where single gauges are required. It is the TPG300 controllers on ANTARES that provides the interlocking capabilities.

The MAXI gauge is still being trialled as a replacement to the TPG300. A unit under test for many years at the HE end of the accelerator has already failed during an accelerator sparking event. Its real place may be away from the accelerator where the 6 heads allow consolidation of a whole beam line's vacuum monitoring system.

On STAR the common high vacuum gauge is the Leybold cold cathode type. It has been integrated into the STAR control system via cat 5 cable. They operate using a similar principle of measuring current flow across the plasma of the ionising gas. These gauges have been far more unreliable than the Pfeiffer equivalent. Unfortunately they are not interchangeable.

If venting with gas other than air or nitrogen be aware that the response on the Pirani gauges may show lower pressure than actual pressure.

#### 14.3 Bourdon Gauges

Bourdon gauges are normally used for indicating that pressure is below atmospheric. They are not used on accelerator systems as they have poor accuracy in the range of interest i.e., below  $1 \times 10^{-3}$  Pa.

# Chapter 3



# **Overview of Basic Vacuum Technology**

The ANSTO accelerators consist of 140 metres of beam lines under vacuum. Supporting these beam lines are 60 individual high vacuum pumping stations. There are over 70 isolation zones along the beam lines to allow isolation of a zone without interrupting the adjacent zones. This facilitates isolations to repair leaking sections, routine maintenance and cycling of pressure in zones to change samples, etc.

The following information are the general tools used by technicians and engineers to design reliable and high performance vacuum systems for the accelerator facilities.

# 1. Vacuum Overview

#### 1.1 What is vacuum?

A vacuum is defined as a diluted gas, or the corresponding state at which its pressure or density is lower than that of the ambient surrounding atmosphere.

Due to the Earth's gravity, atmospheric pressure varies with altitude. As a standard atmospheric pressure at sea level  $p_o = 101.325$  kPa. A Pascal is unit of vacuum relating to a force of newtons per metre squared.

An absolute vacuum of 0 kPa is not practically achievable so in real terms we can only approach zero for example on the ANSTO accelerators we have "operating" vacuums better than  $1 \times 10^{-4}$  Pa. In outer space the vacuum is less than  $10^{-12}$  Pa but not 0 Pa as there are some molecules of gas present which exert a very small partial pressure.

The pressure exerted from a vacuum lies between 0 to 100 kPa (see table below). In an ideal pumped vacuum system the pressure will continue to decease approaching 0 kPa. In accelerator systems the working ranges are medium to ultra high vacuums. In some special cases ion sources may operate in the low vacuum range but generally vacuum systems for the majority of accelerator systems are in the high vacuum range with some vacuum end stations close to ultra high vacuum.

## 1.2 Units used

The SI unit  $Pa = N/m^2$  or kg·m<sup>-1</sup>·s<sup>-2</sup>, will be used where any vacuum quantity is referenced. In the accelerator area the vacuum units used are in Pascals (Pa) and although the vacuum pressures are below atmospheric pressure "negative" pressures are not represented as -kPa but of a magnitude which approaches absolute zero using scientific notation, e.g. 1 x 10<sup>-6</sup> Pa.

#### **1.3** Vacuum categories

Vacuums used in accelerator applications vary in magnitude from low vacuum to ultra high vacuum. These categories describe the pressure ranges which in the vacuum world become references for vacuum quality in a given system. Low vacuum is managed differently from high and ultra high vacuums due to the physical limits of the system designs. Generally, a vacuum system must be designed with the desired ultimate vacuum in mind to ensure the most appropriate materials, seals, pumps and vacuum management are used.

The table below shows each of the ranges based on the European system. This is the closest to that used for the ANSTO accelerators. The shaded section represents the working ranges used throughout the accelerator for example, ion sources may have low to high vacuums, beam lines may have very high vacuums and some endstation vacuum chambers may use high to very high vacuums. This document describes the design factors and particular management controls for achieving vacuums in the various ranges.

Range	Pressure (Pa)
Atmospheric pressure	1.013 x 10 <sup>5</sup>
Low vacuum	$1 \times 10^5$ to $3 \times 10^3$
Medium vacuum	$3 \times 10^3$ to $1 \times 10^{-1}$
High vacuum	$1 \times 10^{-1}$ to 1 x $10^{-4}$
Very high vacuum	$1 \ge 10^{-4}$ to $1 \ge 10^{-7}$
Ultra high vacuum (UHV)	$1 \ge 10^{-7}$ to $1 \ge 10^{-10}$
Extremely high vacuum	< 1 x 10 <sup>-10</sup>
Outer Space	$< 1 \times 10^{-12}$
Perfect vacuum	0 Pa

Table 2. Reference pressure ranges (European scale).

A. Berman, Vacuum Engineering Calculations, Formulas, and Solved Exercises, Academic Press, Inc. (1992).

In the accelerator area:

Low Vacuum = atmospheric pressure to medium vacuum

High Vacuum = high vacuum to very high vacuum

Ultra high vacuum = all those equal to and higher than ultra high vacuum

# 2. Pressure Fundamentals

### 2.1 General

To understand the reason why vacuum systems must be carefully engineered to achieve high vacuums consideration must be given to how gases act in a closed chamber.

Any gas enclosed within a volume (a chamber or pipe work) is uniformly distributed or moving towards uniform distribution as in the case of accelerator tubes connected to stripper gas enclosures. The individual gas particles are constantly moving back and forth at high-speed within the volume; upon striking the vessel wall, they exert a force F on surface A due to pulse transmission. The pressure p that is exerted on the wall is defined as:

$$p = F/A$$

If the gas is made up of different types of gases, each of these gases will exert a pressure that corresponds to its concentration; this is called <u>partial pressure</u>. The sum of all partial pressures equals the total pressure. Air is a good example of this: In addition to its main constituents of nitrogen, oxygen and water vapour, air also contains many trace gases, the sum of which equal  $\sim$ 101.3 kPa, total pressure (atmospheric pressure). See table below.

In an accelerator system it would not be uncommon to have various carbon based compounds, sulphur hexafluoride and specific stripper gases present in trace amounts.

Gas	% by volume	Partial Pressure (Pa)		
Ar	0.92	934		
CO <sub>2</sub>	3.26 x 10 <sup>-2</sup>	33		
H <sub>2</sub>	4.9 x 10 <sup>-5</sup>	4.94 x 10-1		
He	5.16 x 10 <sup>-4</sup>	5.23 x 10-1		
Kr	1.1 x 10 <sup>-4</sup>	1.15 x 10-1		
$N_2$	77.17	78180		
Ne	1.8 x 10 <sup>-3</sup>	1.82		
O <sub>2</sub>	20.7	20970		
Xe	8.59 x 10 <sup>-6</sup>	8.7 x 10-3		
H <sub>2</sub> O	1.18	1200		
Other	remainder	remainder		
Total	100 %	101325 Pa		
Notes: H <sub>2</sub> O may vary depending on environment				
"Other" may include: $CH_4$ , $O_3$ , $N_2O$				

Table 3. Partial Pressures in AirThe Vacuum Technology Book – Pfeiffer Vacuum September 2008

#### 2.2 Ideal (General) gas equation

1 mole of any gas at STP occupies 22.414 litres. At a temperature 273.15 K (0 °C) and a pressure of 101,325 Pa (standard pressure) 1 mole of any gas contains 6.02 x  $10^{23}$  particles. This is referred to

as Avogadro's number. The mass of the gas thus enclosed is its molecular weight in grams. The ideal gas equation describes the state of a gas as a function of pressure, temperature and volume.

$$pV = nRT$$

Example:

For a chamber, Ø300 mm x 500 mm long, a volume of 0.035 m<sup>3</sup>, calculate the mass of gas in the chamber at a vacuum of 1 x  $10^{-6}$  Pa.

pV = nRT

n = pV/RT  $n = 1 \times 10^{-6} \times 0.035 / 8.31451 \times 293 = 1.43 \times 10^{-11} \text{ moles}$ Mass of remaining volume = moles x mass = 1.43 x 10<sup>-11</sup> x 29 (air) = 0.415 ng No. of molecules = moles x Avogadro's Number = 1.43 x 10<sup>-11</sup> x 6.02 x 10<sup>23</sup> = 8.6 x 10<sup>12</sup> Per cm<sup>3</sup> = 8.6 x 10<sup>12</sup> / 35000 cm<sup>3</sup> = 246 x 10<sup>6</sup> molecules per cm<sup>3</sup>

Where:

 $p = \text{pressure (Pa)}, V = \text{volume (m}^3), n = moles, R = \text{general gas constant} = 8.314510 \text{ kJ/(kmol K)}, T = \text{thermodynamic temperature (K)}, Avogadro's number = 6.02 x 10^{23}$ 

# **3.** Flow Fundamentals

#### **3.1** Mean free path $(\lambda)$

The mean free path is the mean path length that a molecule traverses between two successive impacts with other molecules. High vacuums provide a longer mean free path than low vacuums. In accelerator systems high vacuums are crucial as ions that collide with gas molecules in beam lines loose energy and are not transported to the point of measurement. In the accelerator tubes high mean free paths are crucial to ensure low energy loss in the ion beam and optimised ion beam transmission.

Gas (at 0°C and 1 atm)	λ (nm)
Air	68
Ar	62.6
Cl <sub>2</sub>	27.4
CO	58.6
CO <sub>2</sub>	39
$H_2$	110.6
Не	173.6
Kr	36
$N_2$	58.8
Ne	124
02	63.3
Xe	26

Table 4. Mean free Paths for various gases

Hirschfelder, Curtiss and Bird (1954) Molecular Theory of Gases and Liquids, Wiley, New York

Pressure (Pa)	Molecules / cm <sup>3</sup>	Molecules / m <sup>3</sup>	Mean free path
101325	$2.7 \times 10^{19}$	$2.7 \times 10^{25}$	68 nm
	<b>—</b> 11 <b>—</b> 31	1	

Table 5. Other characteristics of Air

#### 3.2 Mean Velocity (u) m/s

The residency time of various gases in a system (or the time taken to remove various gases) relates to their individual mean velocity. The following table shows mean velocities for some gases. It can be seen that very light gases travel significant faster than heavy gases. It should be noted that Hydrogen and Helium do not pump efficiently in turbo pumps or cryopumps but heavy gases generally do.

Gas	Molar Mass kg/mol	Mean Velocity m/s	Mach Number
Air	0.028966	463	1.4
Ar	0.039948	394	1.2
Cl <sub>2</sub>	0.0709	296	
СО	0.028011	471	
CO <sub>2</sub>	0.04401	376	1.1
$H_2$	0.002016	1762	5.3
He	0.00402	1246	3.7
Kr	0.0838	272	
$N_2$	0.02801	471	1.4
Ne	0.020179	555	
O <sub>2</sub>	0.03199	441	
Xe	0.1313	217	
H <sub>2</sub> O	0.01802	587	1.8

Table 6. Mean Velocity for gases at 20°C

The Vacuum Technology Book – Pfeiffer Vacuum September 2008

$$u = \sqrt{\frac{8.R.T}{\pi.M}}$$

Where: *M* = Molar mass (kg/mol)

For example: Mean velocity of Argon @  $20^{\circ}$ C u =SQRT (8 x 8.31451 x 293 / 0.039948 x 3.1416) u =394 m/s

#### 3.3 Types of flow

The different flow phases need to be understood in order to select the appropriate pumping system for a particular application. It may be that in most cases a chamber or other piece of equipment is

pumped at the highest flow rate achievable with a given pump but it could be the case for example where a chamber has delicate films inside and gas movement past the foils has the potential to tear them apart. In this case a low steady flow rate is needed.

The flow phases in a vacuum system characterise the gas molecule movement related to pumping speed in a vacuum system. Factors that determine flow include pressure differentials, mean free paths, gas type, geometry, dimensions of the system and temperature. There are many different nomenclatures used to represent flow phases but 3 types in particular are used in the accelerator area, Turbulent, Laminar (both in Viscous phase), and Molecular. Less emphasis is placed on the intermediate or transitional flow as once this state is reached it is assumed that the system is pumping well. At atmospheric pressure up to about 100 Pa, the mean free path of the gas molecules is very small. Therefore, the gas flow is limited by the viscosity of the gas being pumped so the type of flow is called Viscous.

In an *ideal* system, if a roughing pump has a pumping speed of say 22 m<sup>3</sup>/hr and there is negligible flow resistance between the pump and the chamber then a chamber of  $\emptyset$ 300 mm ID x 500 long (a volume of 0.35 m<sup>3</sup>) at atmospheric pressure, will take approximately 1 minute to remove the bulk of the gas. During this time the flow remains in the [Viscous] Laminar and/or Turbulent phase.

Where a system has a leak, depending on the magnitude of the leak it is possible that the pumping will plateau at either the Turbulent, Laminar or Molecular flow phase. With outgassing systems the less referred to transitional state may be reached and with systems designed to reach ultra high vacuums outgassing may be occurring well into the molecular flow state.

Pumping	Flow	Phase	Description	Type of vacuum	Flow rate Pa-l/sec
Pumping begins		Turbulent Flow	Initial flow experienced at start of pumping or in a low vacuum environment where the flow rate is high. Chaotic movement of molecules. Eddies and other non-parallel flows occur.	Low Vacuum	>1.01
	Viscous	Laminar Flow	Initial flow experienced at start of pumping or in a low vacuum environment where the flow rate is low. Gas molecules bouncing off other gas molecules (gas-gas) in the vacuum system. Short mean free path. Molecules streaming from system.	Medium Vacuum	10.1 to 1.01 x 10 <sup>-4</sup>
	Transit (Kn	ional Flow udsen)	Intermediate flow between laminar and molecular. Flow is dominated by both gas-gas and gas-wall collisions.	Medium to High Vacuum	1.01 x 10 <sup>-2</sup> to 1.01 x 10 <sup>-5</sup>
Base vacuum reached	Molec	ular Flow	Remaining gas molecules bounce off the walls (gas-wall) of the vacuum system and not each other. Long mean free paths. Flow rate very small.	High to Ultra-High Vacuum	<1.01 x 10 <sup>-5</sup>

Table 7. Vacuum flow phases

By evaluating the Knudsen (Kn) and Reynolds (Re) number, one can predict the various flow phases. These phases can be seen in the picture below which also shows how the gas molecules behave in the different flow phases moving through the opening d.

#### 3.4 Knudsen Number (Kn)

The Knudsen number is a dimensionless number defined as the ratio of the molecular mean free path length to a representative physical length scale. The Knudsen number in a viscous flow phase < 0.01 and molecule-molecule collisions dominate gas behaviour which behaves as a fluid. In molecular flow Kn > 1 and molecule-surface collisions dominate.

$$Kn = \frac{\lambda}{d}$$

 $\lambda$  = Mean free path

d = diameter of the opening that gas will pass through

#### 3.5 Reynolds Number (Re)

Reynolds number indicates whether the flow of a gas is absolutely steady (laminar flow) or on average steady but with small, unsteady changes (turbulent flow). The Reynolds number, Re, has no dimensions and is defined as the size of the flow.

Osborne Reynolds demonstrated in 1883 that the change from laminar to turbulent flow in a pipe occurs when the value of the Reynolds number exceeds 2,100. The exact value of Re for which the flow changes from laminar to turbulent depends on the geometry of the component, its surface roughness and other experimental factors. During evacuation of a vessel, turbulent flow normally occurs only for a short period of time at the beginning. It has been found to be approximately proportional to the root of the pressure gradient.

$$\operatorname{Re} = \left(\frac{\rho}{\eta}\right) d.v$$

 $\rho$  = gas density (kg/m<sup>3</sup>)  $\eta$  = viscosity (Pa.s) (Pascal-second) v = flow velocity (m/s) d = tube diameter (m)

Gas	Formula	Molecular weight	Gas Density [2] kg/m <sup>3</sup>	Viscosity η Pa.s
Air [1]	$N_2 + O_2$	29	1.1839	1.79 x 10 <sup>-5</sup>
Ammonia	NH <sub>3</sub>	17.031	0.7449	1.01 x 10 <sup>-5</sup>
Argon	Ar	39.948	1.723	2.24 x 10 <sup>-5</sup>
Carbon Dioxide	$CO_2$	44.01	1.9105	1.51 x 10 <sup>-5</sup>
Carbon Monoxide	CO	28.01	1.2082	1.74 x 10 <sup>-5</sup>
Chlorine	Cl <sub>2</sub>	70.906	3.1124	1.34 x 10 <sup>-5</sup>
Helium	He	4.02	0.171	1.99 x 10 <sup>-5</sup>
Hydrogen	$H_2$	2.016	0.0868	8.80 x 10 <sup>-5</sup>
Hydrochloric Acid	HCl	36.5	1.5844	1.46 x 10 <sup>-5</sup>
Hydrogen Sulphide	$H_2S$	34.076	1.4876	1.26 x 10 <sup>-5</sup>
iso-Butane	$C_4H_{10}$	58.12	2.3758	7.49 x 10 <sup>-5</sup>
Methane	$CH_4$	16.043	0.6556	1.11 x 10 <sup>-5</sup>
Nitrogen	$N_2$	28.02	1.2088	1.76 x 10 <sup>-5</sup>
Oxygen	$O_2$	32	1.381	2.02 x 10 <sup>-5</sup>
Propane	C <sub>3</sub> H <sub>8</sub>	44.09	1.8024	8.26 x 10 <sup>-5</sup>
Sulphur Hexafluoride	$SF_6$	146.5	6.27	$1.53 \times 10^{-5}$

Table 8. Gas density and viscosities

Unless otherwise indicated gases referenced from McGraw Hill Chemical Properties Handbook 1 atm 25°C. [1] Air STD Atmosphere at Sea Level: 1 atm 15°C [2] Gas Density Values Interpolated From 15°C Data using Charles Law

http://pipeng.com/index.php/gsts/itdmodflup002a/itddaflup00201

#### 3.6 Viscous Flow

#### Low vacuum, p = 100000 - 100 Pa, where $\lambda \ll d$

What characterises viscous flows, is frequent contact between gas molecules, yet less frequent contact with the walls of the vessel. The mean free path of the gas molecules is therefore very small and the gas flow is limited by the viscosity of the gas being pumped (Viscous Flow). In this case, the mean free path of the gas molecules is significantly shorter than the dimensions d of the vacuum equipment. For both Laminar and Turbulent Flow, Kn < 0.01. In addition, the term viscous flow is used if the product of pressure p and diameter d of the components through which gas is flowing is p.d  $\geq$  60 Pa.cm for air. Whether the flow is in the Laminar or Turbulent flows phases can be calculated using the Reynolds Number formula above.

#### 3.7 Knudsen flow

#### Medium vacuum, p = 100 - 0.1 Pa with $\lambda \le d$

If the Knudsen number is between 0.01 and 0.5, this is termed Knudsen flow. Because many process pressures are in the medium vacuum range, this type of flow occurs with corresponding frequency. Since this is a transitional flow, this range is transited relatively quickly when pumping down vacuum chambers and hence not usually considered in accelerator systems. This means that the influence of this conductivity on pump-down times is correspondingly low. A simple approximation for the Knudsen range can be obtained by adding the laminar and molecular conductivities. The

table in "Conductivities" below shows the conductivities of round, one meter long tubes of differing diameters in all three flow ranges.

### 3.8 Molecular flow High vacuum, $(p = 0.1 - 10^{-5} \text{ kPa})$ , where $\lambda > d$ and in UHV $(p < 10^{-5} \text{ kPa})$ , with $\lambda >> d$

At Knudsen numbers of Kn > 0.5 molecule-molecule collisions virtually no longer occur. What prevails is molecular flow. In this case, the product of pressure p and component diameter d is p x d  $\leq$  1.3 Pa.cm.



Fig 5. Molecular paths during different flow phases Re reference: http://www.engineersedge.com/fluid\_flow/pressure\_drop/pressure\_drop.htm

#### **3.9 Pump throughput qpV**

The concept of pump throughput is of major significance in practice and should not be confused with the pumping speed! The pump throughput is the quantity of gas moved by the pump over a unit of time, expressed in Pa.1.s<sup>-1</sup>. Conversely, the pumping speed is the capacity of the pump to remove a volume of gas within a specific unit of time, measured in  $m^3/h$  or 1/s.

The throughput value is important in determining the size of the backing pump in relationship to the size of a high vacuum pump with which it is connected in series in order to ensure that the backing pump will be able to take away the gas moved by the high vacuum pump.

The pumping capacity (throughput) for a pump is equal either to the mass (m) flow through the pump intake port in a specified time:

$$q_m = \frac{m}{t}$$

Or to the pV (quantity of gas) flow through the pump's intake port:

$$q_{pV} = \frac{pV}{t}$$

It is normally specified in Pa.l.s<sup>-1</sup>. Here p is the pressure on the intake side of the pump. If p and V are constant at the intake side of the pump, the throughput of this pump can be expressed with the simple equation:

 $q_{pV} = p.S$ 

Where:

S = pumping speed of the pump at intake pressure of p.

#### 3.10 Speed of vacuum pump S

The speed of a vacuum pump is defined as

$$S = \frac{q_{pV}}{p}$$

p = Pressure at the pump inlet

However, pumps are usually connected to vacuum chambers via hoses resulting in a lower effective pumping speed  $S_{eff}$  at the chamber. If  $p_1$  is the pressure at the pump and  $p_2$  is the pressure in the vacuum chamber then:

$$q_{pV} = S.p_1 = S_{eff}.p_2$$

for continuous flow. Combining this with  $q_{pV} = C(p_1 - p_2)$  (see Conductance below) results in an expression for S<sub>eff</sub>

$$S_{eff} = \frac{S.C}{S+C}$$

#### **3.11** Conductance C

In a vacuum system, the volume that is pumped by a vacuum pump exhibits a level of flow resistance (Z) due to chamber and pipe sizes and their geometries, and the various sized orifices and other in-stream devices. The terminology used to describe this effect is the reciprocal of flow resistance which is *conductance* (C) or the systems ability to conduct gas.

Conductance is expressed in either l/s or sometimes  $m^3/h$  and is usually for steady, continuous flow through one or more components in a vacuum system. It is affected by the geometry of the piping element and relative to the flow phase and capacity of the pump used. In the high and ultrahigh vacuum ranges (molecular flows), C is a constant which is independent of pressure. In the rough and medium-high vacuums (turbulent and laminar) it is, by contrast, dependent on pressure. As a consequence, the calculation of C for the piping (beam line) elements must be carried out separately for the individual pressure ranges.

Flow resistance (sec/litre) 
$$Z = \frac{P_1 - P_2}{q_{_{PV}}}$$

Conductance (litre/sec) therefore

 $C = \frac{1}{Z} = \frac{q_{_{PV}}}{P_1 - P_2} \qquad (continued over)$ 

$$q_{pV} = C(p_1 - p_2)$$

Where:  $q_{pV}$  = Throughput flow rate p = Pressure (Pa)

At the molecular level, the conductivity of a system is reduced due to the friction of both the walls of the system and other molecules. As discussed above in "Types of Flow", these two affects occur at different flow phases. The total effect of conductance is the sum of the various conductance elements. In the case where a valve, a trap and an elbow in series each having a different conductance, the sum is:

$$\frac{1}{C_{total}} = \frac{1}{C_{valve}} + \frac{1}{C_{trap}} + \frac{1}{C_{elbow}}$$

If the components with different conductances are connected in the flow path in parallel, for example, 2 different diameter tubes connecting a chamber to a pump then the equation will be:

$$C_{_{Total}} = C_{_{tube1}} + C_{_{tube2}}$$

For the design of vacuum systems used on the accelerators conductance is mostly calculated for systems in molecular flow.



*Fig 6. Diagram for estimating pipe conductance Pupp/Hartmann, Vakuumtechnik, Grundlagen und Anwendugen, Hanser Verlag* 

# 4. Limitations

The most fundamental problems with vacuum systems are leaks and outgassing. Leaks can be categorised as either real leaks where gas enters the vacuum system from outside of the vessel or leaks form trapped voids which is in effect an internal leak. Trapped voids can contain gas that will continue to outgas for long periods reducing the ultimate vacuum that can be reached in a vessel. Real leaks can be found with the aid of a helium leak detector whereas internal leaks from trapped voids may never be found.

Outgassing can be minimised through careful selection of materials used within the vacuum space. The appendices have helpful tables to estimate outgassing rates. Outgassing may be in the form of desorption, diffusion or vaporisation. Permeation is not outgassing as the source of gas originates from outside of the vacuum space.

Examples commonly used substances in the accelerator systems that outgas,:

- Teflon, PVC, Ertalyte
- Viton, neoprene
- Copper, aluminium, stainless steel, brass, tantalum, rubidium, caesium, lithium, zinc
- Vacuum pump oil, vacuum greases, vacuum epoxies

These are just a few but it demonstrates that all materials will outgas at some point when the temperature and vacuum pressure reach their individual vapour pressure.

Neither of the problems is resolved by increasing the pumping speed (capacity) of the vacuum pump. There will be a point where the rate of gas entering the system will equalise with the pumping speed of the pump. The rate may vary depending on the vapour pressure of the substance outgassing and the temperature of that substance.



Fig 7. Limitation of Pumping From: http://www.mse.ncsu.edu/WideBandgaps/classes/MSE%20751/Handouts/HO02\_Leakage.pdf



Fig 8. Unwanted gas source wheel

# 4.1 Contamination

Vacuum chambers must be clean in order to reach the desired pressure as quickly as possible when they are pumped down. Typical contaminants include oil and grease on screws and seals, process reaction products or condensed vapours, particularly water that is adsorbed on the walls of the vessel. Consequently, it is necessary to ensure that the components are clean when installing vacuum equipment. All components attached in the vacuum chamber must be clean and grease-free. All seals must also be installed dry. If high or ultra high vacuum is to be generated, clean gloves must be worn during the assembly process.

Examples of common contaminants in accelerator vacuum systems

- Rotary pump oil
- Water
- Plasticisers from various plastics
- General airborne dust
- Machining oil
- Residual gases from stripper gases, venting gases, ion source gases
- Sample breakdown

# 4.2 Condensation and vaporisation

All substances can occur in a liquid, solid or gaseous state. Their aggregate status is a function of pressure and temperature. Liquids are transformed into their gaseous state through vaporisation, solids through sublimation. The separation of liquids or solids out of the gaseous phase is termed condensation. Since normal room air contains approximately 10g of water vapour per m<sup>3</sup>, condensed water vapour is always present on all surfaces.

Adsorption on surfaces is especially pronounced due to the strong polarity of the water molecules. Natural fibres, in particular, such as paper, contain large quantities of water that escape during drying processes under vacuum. Cooled condensers are used to separate the water vapour in this connection. Even some metals (Cd, Zn, Mg) can vaporise in noticeable quantity at temperatures of several 100 °C. Consequently, use of these metals is avoided in plant construction.

#### 4.3 Desorption

In addition to water, other substances (oil) can be adsorbed on surfaces. Substances can also diffuse out of the metal walls, which can be evidenced in the residual gas. In the case of particularly rigorous requirements, stainless steel vessels can be baked out under vacuum, thus driving the majority of the volatile components out of the metal walls.

Gas molecules, (primarily water) are bound to the interior surfaces of the vacuum chamber through adsorption and absorption, and gradually desorb again under vacuum. The desorption rate of the metal and glass surfaces in the vacuum system produces a gas yield that is a function of time, however. A good approximation can be obtained by assuming that after a given point in time  $t > t_0$  to the reduction will occur on a linear basis over time.  $t_0$  is typically assumed to be one hour.



Fig 9. Desorption curves



Fig 10. Adsorption Curves From: http://www.mse.ncsu.edu/WideBandgaps/classes/MSE%20751/Handouts/HO02\_Leakage.pdf

#### 4.4 Diffusion with desorption

At operation below  $10^{-4}$  kPa, desorption of plastic surfaces, particularly the seals, assumes greater significance. Plastics mainly give off the gases that are dissolved in these plastics, which first must diffuse on the surface.

Following extended pump downtimes, desorption from plastics can therefore dominate the metal surfaces. Although the surface areas of the seals are relatively small; the decrease in desorption rate over time occurs more slowly in the case of metal surfaces. As an approximation it can be assumed that the reduction over time will occur at the square root of the time.

The gas produced from plastic surfaces can thus be described as:

Desorption from plastic material  $Q_{diff} = q_{diff} \cdot A \sqrt{t_o/t}$ 

Where Ad denotes the surface area of the plastics in the vacuum chamber and  $q_{diff}$  denotes the surface area-specific desorption rate for the respective plastic. At even lower pressures, similar effects also occur with metals, from which hydrogen and carbon escape in the form of CO and CO<sub>2</sub> and can be seen in the residual gas spectrum.

#### 4.5 **Permeation and leaks**

For a gas passing through small holes in a thin wall in the Knudsen Flow regime, the number of molecules that pass through a hole is proportional to the pressure of the gas and inversely proportional to its molecular weight.

To Convert to Leakage	Multiply Helium Leak Rate by:		
Rate of:	Laminar Flow	<b>Molecular Flow</b>	
Argon	0.88	0.316	
Air	1.08	0.374	
Nitrogen	1.12	0.374	
Water vapour	2.09	0.469	
Hydrogen	2.23	1.410	

Table 9. Conversation table for leak rates

Seals, and even metal walls, can be penetrated by small gas molecules, such as helium, through diffusion. Since this process is not a function of time, it results in a sustained increase in the desired ultimate pressure. The permeation gas flow is proportional to the pressure gradient  $p_0 / d$  (d = wall thickness,  $p_0 =$  atmospheric pressure = ambient pressure) and to the permeation constants for the various materials  $k_{perm}$ .

Permeation

 $k_{perm}$ .  $Q_{perm} = k_{perm}$ .  $A \cdot p_0/d$ 

Permeation first manifests itself at pressures below  $10^{-6}$  kPa.  $Q_1$ , denotes the leakage rate, i.e. a gas flow that enters the vacuum system through leaks at a volume of V. The leakage rate is defined as the pressure rise  $\Delta p$  over time  $\Delta t$ :

Leakage rate  $Q1 = (\Delta p \cdot V)/\Delta t$ 

If a vessel is continuously pumped out at a volume flow rate S, an equilibrium pressure  $p_{gl}$  will be produced. Throughput is equal to the leakage rate  $Q_l = S \cdot p_{gl}$ . A system is considered to be
adequately tight if the equilibrium pressure  $p_{gl}$  is approximately 10 % of the working pressure. If, for example, a working pressure of  $10^{-4}$  kPa is attained and the vacuum pump that is being used has a pumping speed of 100 I/s, the leakage rate should not be more than  $10^{-3}$  kPa I/s. This corresponds to a leak of approximately 20.20  $\mu$ m<sup>2</sup> in size. Leakage rates  $Q_l$  of less than  $10^{-6}$  kPa I/s can usually be easily attained in clean stainless steel vessels. The ultimate pressure achievable after a given period of time *t* primarily depends upon all of the effects described above and upon the pumping speed of the vacuum pump. The prerequisite is naturally that the ultimate pressure will be high relative to the base pressure of the vacuum pump.



Fig 11. Permeation of Different Gases From: http://www.mse.ncsu.edu/WideBandgaps/classes/MSE%20751/Handouts/HO02\_Leakage.pdf

# 4.6 Leaks

The source of leaks is as wide and varied as can be imagined. There is no such thing as a common leak however there are several types generally seen. The following lists types in rough order of prevalence:

- Dirty seals elastomer type seals with traces of dust, dirt and fibres on the surface
- Jarred seals ill fitted flanges where the seal lays across the sealing surface
- Vacuum pumping tubes where a fitting has been fitted without care and the pump cannot reach its full capability
- Distortion over tightened or misaligned flanges and fittings that "lift" seals off the sealing surface
- Stress cracks usually in places around flanges and fittings that have been incorrectly tightened
- Pressure dependant mostly related to accelerator tubes that develop leaky seals allowing insulation gas to enter when the pressure is greater than atmospheric pressure.

# 4.7 Bake-out

The following prerequisites must be satisfied in order to achieve lower pressures ( $<10^{-6}$  kPa):

- The base pressure of the vacuum pump should be a factor of 10 lower than the required ultimate pressure
- Stainless steel vacuum recipients and components must be used
- Metallic seals (CF flange connections) are required
- Leaks must be avoided and eliminated prior to activating the heater (use helium leak detectors!)
- Clean work is a must, i.e. all parts must be thoroughly cleaned and must be installed with grease-free gloves
- Pump and equipment must be baked out

Bake-out significantly increases desorption and diffusion rates, and this produces significantly shorter pumping times. Bake-out temperatures of up to 300°C are used. The instructions of the pump manufacturers relating to maximum bake-out temperatures and maximum permissible radiation levels in the pump flange must be observed.

Following installation the equipment is switched on, and after reaching a pressure of  $P < 10^{-3}$  kPa the heater is then switched on. During the heating process, all gauge heads must be operated and degassed at intervals of 10 hours. In the case of stainless steel vessels and the use of metallic seals, bake-out temperatures of 120°C and heating times of approximately 48 hours are sufficient for advancing into the pressure range of  $10^{-8}$  kPa. Bake-out should be continued until 100 times the expected ultimate pressure is attained. The heaters for the pump and vacuum chamber are then switched off. After cool-down, the desired ultimate pressure will probably be achieved. In connection with pressures  $P < 5 \times 10^{-8}$  kPa and large interior surface areas, it will be advantageous to use a titanium sublimation pump that pumps the hydrogen escaping from the metals at a high volume flow rate.

# 4.8 Residual gas spectrum

When leaks have been solved in a vacuum system and poor vacuum persists than a residual gas analysis (RGA) measurement should be made to determine the composition of the gas load. This will give vital information on where the vacuum problem may be. Interpreting the information will be trial and error as users will have to think about all possible sources of gases (and outgassing) that may be present. Also relative ratios of gases should be considered to make sense of the measurement. Care should be taken especially if making a measurement near a source such as a cryopump. Some gases that have been trapped on the cryopump may be liberating from the cold surface and will make up a part of the gas spectrum. Other places to take care are near the accelerator tubes and ion sources.

If developing an ultra high vacuum system it is important to know all sources of gas otherwise moving lower than  $1 \times 10^{-6}$  Pa will be an unnecessary challenge. For all other systems it is nice to know but not essential unless residual gases affect ion beam measurements. The dominate gases (and their masses) that may be seen include:

Gas	Mass	Gas	Mass
Water	18	Carbon Monoxide	28
fragment HO	17	Carbon Dioxide	44
Nitrogen	28	Argon	40
Oxygen	32	Sulphur Hexafluoride	146
Hydrogen	2	Helium	4

Table 10. Table of masses for common "in vacuum" gases

# **Appendix 1 – Structural Calculations for Scientific Vacuum Vessel Design**

Calculations for Vacuum Vessel wall and end plate thickness

The following formulae are from the Vacuum Society of Australia (VSA) training course notes which have been in circulation since the early 1980's. VSA continues to support these calculations for training vacuum technologists around Australia. The calculations provide conservative estimates of vacuum vessel wall thicknesses as compared with formulas used in ASME Section VIII – Division 1 UG-28 (Thickness of Shells and Tubes under External Pressure) and associated documents, which have been developed for larger industrial type vacuum vessels.

### Cylindrical Vessel – Stainless Steel @ 20°C

Thickness of plate in a given diameter (h):

$$\frac{D}{h} \le 105, \ \frac{h}{D} \ge \frac{1}{105}$$

Where: D = diameterh = thickness

Maximum length of vessel for a given diameter (L<sub>c</sub>):

$$\frac{L_c}{D} \le 11.5$$

Where: D = diameter $L_c = \text{length}$ 

# **End Plates – Stainless Steel**

Thickness of plate in a given diameter (h<sub>1</sub>):

$$\frac{D_1}{h_1} \le 89$$
 ,  $\frac{h_1}{D_1} \ge \frac{1}{89}$ 

Where:  $D_1$  = diameter  $h_1$  = thickness

Minimum thickness for given deflection at centre:

 $\frac{h_1}{\delta} \ge 3$ 

Where:  $\delta$  = deflection  $h_1$  = thickness For Hemispherical End (h<sub>2</sub>):

$$\frac{R}{h_2} \le 830 \text{ requires } \frac{h_2}{R} \ge \frac{1}{830}$$

Where: R =radius  $h_2 =$  thickness

**Example 1:** To construct Stainless Steel vacuum chamber at  $20^{\circ}$  C as shown – Length L = 50 cm = 500 mm, D = 40 cm = 400 mm, Maximum allowable deflection at centre  $\delta = 1$  mm. Find  $h, h_1, h_2$  to satisfy vacuum chamber strength requirements.

# For Cylinder

1. 
$$\frac{D}{h} \le 105$$
,  $\frac{h}{D} \ge \frac{1}{105}$   $h \ge 400/105$ ,  $h \ge 3.8 \text{ mm}$   
2.  $\frac{L_c}{D} \le 11.5$ ,  $L_c \le 11.5 \times 400$   $L_c \le 4600 \text{ mm}$  with  $L = 500 \text{ mm} \checkmark$ 

### **For End Plates**

1. 
$$\frac{D_1}{h_1} \le 89$$
,  $\frac{h_1}{D_1} \ge \frac{1}{89}$   $h_1 \ge 396.2/89$ ,  $h_1 \ge 4.45 \text{ mm}$   
2.  $\frac{h_1}{\delta} \ge 3$ , with  $\delta = 1 \text{ mm}$  then require  $h_1 \ge 3 \text{ mm}$ , however  $h_1 = 4.45 \text{ mm} \checkmark$ 

### For Hemispherical End

1. 
$$\frac{R}{h_2} \le 830$$
 requires  $\frac{h_2}{R} \ge \frac{1}{830}$   $h_2 \ge 198.1/830 \ge 0.24$ mm

Dimension of cylindrical, planar and hemispherical parts of vacuum enclosures

	At	Cylin	nders	End I	Plates	Hemispherical
Material	Temp (°C)	D/h	$L_c/D$	$D_1/h_1$	$h_l/\delta$	<i>R/h</i> <sub>2</sub>
Copper	20	84	10	52	15	600
Copper	500	58	8.5	-	-	-
Nickel	20	100	11	73	8	780
Nickel	500	90	10.5	-	-	-
Aluminium Alloy	20	70	9	37	57	470
Aluminium Alloy	500	62	8.7	-	-	-
Stainless Steel (304)	20	105	11.5	89	3	830
Stainless Steel (304)	500	89	10.5	-	-	-
Glass (hard)	20	70	9	16	117	470
Neoprene	20	2.5	1.7	10	0.2	30
Teflon	20	12	3.8	14	9	-
PVC (Tygon)	-	3.7	2.1	-	-	-
Perspex	-	_	-	30	-	-
Mica	-	_	-	58	15	-

Factor tables for thickness calculations of common materials used in high vacuum systems

 $\delta$  = maximum permissible deflection at centre of plate L<sub>c</sub> = critical length for cylinder = 1.11D(D/h)1/2

**Example 2:** Using the ASME Boiler and Pressure Vessel Code (the Code) Section VIII, Division 1 for the safe design pressures for a vacuum vessel is used to calculate the maximum external pressure acceptable for a given wall thickness. The following example uses the same vessel dimensions as in Example 1, with a wall thickness of 3.8 mm as calculated using the VSA method.

The calculations reference the Vacuum Vessel Cylindrical Shell Thickness using paragraphs UG-28, UG-27 of ASME Boiler and Pressure Vessel Code.

Vessel shell thickness t = 3.8 mm  $D_o = 400$  mm (outer diameter of shell)  $D_o/t = 105.26$  Since this ratio is greater than 10, follow UG-28(c)(1) L = 500 mm (length of stiffened shell)  $L/D_o = 1.25$ E = 28 x 10<sup>6</sup> (modulus of elasticity for 304 stainless steel)

From Table G in Sec II, Subpart 3 to determine Factor A

A = 1 x 10<sup>-3</sup> (Factor A, from Table G of Sec. II, Subpart 3)  $P_{ext} = \frac{2AE}{3(\frac{D_o}{t})}$ 

 $P_ext = 177.33$  psi (max allowable working external pressure for given *t*) = 1222 kPa

A wall thickness of t = 3.8 mm results in a maximum allowable working external pressure of P\_ext = 325 kPa, which is greater than the external pressure that the vacuum vessel will see.

**Example 3:** For comparison, calculate the maximum external pressure as in Example 2 but with a wall thickness of 2.5 mm.

 $D_{o} = 400 \text{ mm}$ t = 2.5  $D_{o}/t = 160$ L = 500 L/D\_{o} = 1.25 E = 28 x 10<sup>6</sup> (modulus of elasticity for 304 stainless steel)

From Table G in Sec II, Subpart 3 to determine Factor A

A =  $5.3 \times 10^{-4}$  (Factor A, from Table G of Sec. II, Subpart 3)

$$P\_ext = \frac{2AE}{3(\frac{D_o}{t})}$$

P\_ext = 61 psi (max allowable working external pressure for given t of 2.5 mm) = 420 kPa

Therefore a wall thickness of 2.5 mm has a P\_ext of 420 kPa.

**Example 4:** This is a worked example for a large vacuum vessel from ASME Boiler and Pressure Vessel Code (the Code) Section VIII, Division 1, paragraph UG-28. It is in imperial units.

Calculate the maximum allowed external working pressure following UG-28. The minimum required thickness takes into account the support of the stiffening rings. Note that the actual vessel shell thickness t = 0.375-inch

 $D_o = 38.0$  inch (965 mm) (outer diameter of shell) t = 0.261 inch (6.63 mm) (minimum required thickness of shell for external pressure of 14.5-psia)  $D_o/t = 145.594$  Since this ratio is greater than 10, follow UG-28(c)(1) L 235.7 inch (6057.5 mm) (length of stiffened shell)  $L/D_o = 6.203$ E =29x10<sup>6</sup> psi (modulus of elasticity for SA-516 Gr 60 carbon steel)

From Table G in Sec II, Subpart 3 to determine Factor A A =  $1.108 \times 10^{-4}$  (Factor A, from Table G of Sec. II, Subpart 3)

$$P\_ext = \frac{2AE}{3(\frac{D_o}{t})}$$

P\_ext = 14.709 psi (maximum allowable working external pressure for given t) = 101.35 kPa

A wall thickness of t = 0.261 inch results in a maximum allowable working external pressure of P\_ext = 14.7 psia, which is greater than the external pressure that the vacuum vessel will see. Since the actual wall thickness is 0.375 inch (9.5 mm), the vessel design is adequate for the working external pressure.



Factor A table for Stainless Steels

# **Appendix 2 – Low Outgassing Specialist Non Metal Materials**

# • Celazole® PBI (PolyBenzImidazole) http://www.boedeker.com/celazo\_p.htm

Celazole® is the highest temperature-capable plastic available. However, it is very brittle (almost ceramic-like) and quite difficult to machine. That said, is it frequently used for bushings, bearings, rollers, and spacers in extreme environments. Its outgassing values are listed as 2.50% TML, 0.00% CVCM, 0.40% WVR.

# • Vespel® (Polyimide)

http://www2.dupont.com/Vespel/en\_US/assets/downloads/vespel\_gen/E61500.pdf

DuPont Vespel® SP-1 is one of the most-used high-temperature plastic materials used in applications where high-purity and electrical properties are needed. Vespel is frequently used in ultra-clean semiconductor and chemical applications. It is also one of the most expensive materials sold, but is flight-approved for NASA, USAF and other aerospace agencies. Its NASA outgassing values are listed as 1.09% TML, 0.00% CVCM, 0.40% WVR.

# • Duratron® XP (Polyimide)

http://www.portplastics.com/download/pdf/plastics/highPerformance/highPerformance26.pdf

Duratron® XP is the first real alternative to Vespel ... it was developed specifically to replace Vespel in extreme applications at a slightly lower price. It contains less than 1% metallic impurities as measured using the ICP-MS test standard. Duratron XP is ideal for use in high-energy gas plasma etch and strip processes. Outgassing values for Duratron XP are 0.75% TML, 0.00% CVCM, 0.49% WVR.

# • Torlon® 4203 unfilled PAI (PolyAmide-Imide) http://www.boedeker.com/torlon-4203.htm

Unfilled Torlon® 4203 has high dielectric properties and low thermal expansion, and is much less expensive than some advanced polymers. Torlon 4203 is typically used for insulators, spacers, and mechanical parts up to 520°F. Its outgassing values are listed as 1.85% TML, 0.00% CVCM and 0.49% WVR.

# • Torlon® 5530 glass-filled PAI (PolyAmide-Imide) http://www.boedeker.com/torlon-5530.htm

Torlon 5530 (30% glass-filled) is typically used for applications where dimensional stability over a wide temperature range is needed, as with temperature test sockets, nests, and fixtures. Its outgassing values are listed as 0.58% TML, 0.00% CVCM (% WVR is not shown). NOTE: Torlon's moisture absorption is a bit high, so critical dimensional stability can be an issue.

# • Semitron® ESd 500HR (filled PTFE)

http://www.portplastics.com/download/pdf/plastics/staticControl/staticControl18.pdf

Semitron® ESd 500HR is antistatic/conductive PTFE. This material is relatively clean, readily machinable, dissipates static electricity reliably ... as a result it is used in test handling equipment, fixtures, and other applications where static generation may cause failures and/or errors in production environments. This material has low outgassing values of 0.04% TML, 0.00% CVCM and 0.01 % WVR. PTFE has good mechanical properties up to approximately 500°F.

# • Neoflon® PCTFE (PolyChloroTetraFluoroEthylene)

http://www.aftonplastics.com/materials/pdfs/neoflan\_pctfe.pdf

PCTFE exhibits high chemical resistance, low and high temperature capability, resistance to most chemicals (including strong acids and bases), low friction, electrical and thermal insulation, and "slipperiness". PCTFE has the lowest outgassing values of any thermoplastic material we sell ... 0.01% TML, 0.00% CVCM, 0.00% WVR.

• **PEEK (PolyEtherEtherKetone)** http://www.dotmar.com.au/ketron-peek-1000/ketron-peek-1000-polyetheretherketone.html

PEEK is pure, easily machinable, chemically resistant, stable, and also has relatively low outgassing values (0.31% TML, 0.00% CVCM, 0.06% WVR). PEEK has good mechanical properties, but will not take temperatures over  $350^{\circ}$ F, so it may not have the mechanical or thermal performance needed.

• **Techtron® PPS (PolyPhenylene Sulfide)** http://www.dotmar.com.au/techtron-hpv-pps/techtron-hpv-pps.html

Techtron® PPS is easily machined to close tolerance, has excellent mechanical, thermal and chemical stability and has one of the lowest outgassing values of any thermoplastic material we offer (0.04% TML, 0.00% CVCM ... % WVR is not shown). Techtron PPS is generally a bit less expensive than PEEK or Torlon, but again, will not take as high temperatures.

• Ultem® PEI (PolyEtherImide) http://www.boedeker.com/ultem\_p.htm

Ultem<sup>®</sup> has good dielectric properties and low thermal expansion, and is considerably less expensive than some other polymers. PEI is also clean and stable, but is not particularly resistant to chemicals or solvents ... it has outgassing values of 0.40% TML, 0.00% CVCM and 0.06 % WVR. PEI has good mechanical properties up to approximately 410°F.

• Semitron® ESd 410C (filled PEI) http://www.boedeker.com/sem410\_p.htm

Semitron® ESd 410C is antistatic/conductive PEI. This material is relatively clean, readily machinable, dissipates static electricity reliably ... as a result it is used in test handling equipment, fixtures, and other applications where static generation may cause failures and/or errors in production environments. This material has outgassing values of 0.46% TML, 0.00% CVCM and 0.17 % WVR. PEI has good mechanical properties up to approximately 340°F.

# • Ertalyte® PET-P (Polyethylene Terephthalate) http://www.boedeker.com/ertaly\_p.htm

Ertalyte® offers the dimensional stability of acetal with the wear resistance of nylon. Ertalyte® PET-Polyester is clean, chemically resistant, stable, and also has relatively low outgassing values (0.13% TML, 0.00% CVCM ... % WVR is not shown). PET-P is considerably less expensive than most of the other materials listed above, but may not have the mechanical or thermal performance needed for all applications.

# • Semitron® ESd 225 (filled acetal) http://www.boedeker.com/sem225\_p.htm

Semitron® ESd 225 is antistatic/conductive acetal. This material is relatively clean, readily machinable, dissipates static electricity reliably ... as a result it is used in test handling equipment, fixtures, and other applications where static generation may cause failures and/or errors in production environments. This material has low outgassing values of 1.00% TML, 0.05% CVCM and 0.60 % WVR. Acetal has good mechanical properties up to approximately 180°F.

# Appendix 3 – Materials for use in Vacuum

The choice of materials is limited for vacuum systems to a range that has little impact on vacuum production and ultimate base vacuums.

# 1.1 Metals

- <u>Stainless Steel</u> is used for the majority of vacuum system designs due to its:
  - o Durability
  - Surface finishing
  - Ability to withstand moderate temperatures for system bake out
  - Low magnetic susceptibility
  - Low thermal conductivity
  - Resistance to oxidisation
  - Low vapour pressure

Type 304 or 316 stainless steel is ideally suited for vacuum vessel construction because of its machining/welding characteristics, excellent corrosion resistance and overall cost effectiveness.

Not all stainless alloys are acceptable. Free-machining 303 steel contains sulphur, which tends to outgas. Alloys with good weldability using TIG or MIG welding are usually chosen.

- 304 or 316 stainless steel is a common choice of a stainless steel.
- $\circ~$  304L stainless steel, a low-carbon variant of 304 steel, is used for ultra-high vacuum systems.
- 347 stainless steel does not accept high polish.
- $\circ$  321 stainless steel is chosen when low magnetic permeability is needed.
- <u>Mild steel</u> is okay for low to moderate vacuums above 10<sup>-4</sup> Pa. Outgassing can be reduced with suitable plating such as nickel. It has high permeability to hydrogen and tendency to rust. Mild steel must not be used for any components or chambers used near the bending magnets
- <u>Aluminium alloys</u> are easily machined and have a low vapour pressure, unless the alloys contain high proportion of zinc. 6061 grade is good for general use. Components used in high vacuums or better must not be anodized, as the oxide layer can trap water vapour and outgas. Aluminium and its alloys have low strength at high temperatures, distort when being welded, and the copper-containing ones are poorly weldable. Aluminium wire rings can be used as cheap gaskets in demountable seals. Soft alloys must be used to ensure wire gaskets do not damage the seal faces. Aluminium has high thermal conductivity, good corrosion resistance, and low solubility of hydrogen. Loss of strength at high temperatures limits its use in bakeable applications, but aluminium is advantageous for large-size systems due to its lower weight and lower cost than stainless steel. Aluminium is not recommended for vacuum chambers.
- <u>Brass</u> is not acceptable for vacuum chambers however it may be suitable for some small beam line inserts for specific applications. Once used widely but copper can cause problems with neutron production if struck by proton beams of particular energies. Brass is for high thermal conduction applications in cooling baffles or sample mounts that may be heated. Although bare

brass has good corrosion resistance the zinc content may cause outgassing problems. This can be reduced by plating with nickel.

- <u>Nickel</u> is widely used in vacuum technology, e.g. as mechanical parts in vacuum tubes. It is relatively low-cost, can be spot welded, can be easily machined, has high melting point and is resistant to many corrosive fluids and atmospheres. Its potential drawback is its ferromagnetism, which restricts applications that would be influenced by magnetic fields.
- <u>Beryllium</u> is used primarily for x-ray windows.
- <u>Oxygen-free copper</u> is okay for high vacuum but it is difficult to outgas completely. Copper is insensitive to hydrogen and impermeable to hydrogen and helium, has low sensitivity to water vapour, but is attacked by mercury. Although, oxygen-free copper is widely used as it is easily machined and has good corrosion resistance. It is unsuitable for bakeable vacuum envelopes due to its tendency to oxidize and create scales. Conflat flange seals are made from copper. Its strength falls sharply above 200 °C. Its vapour pressure becomes significant at above 500 °C.
- <u>Indium</u> wire is used as a gasket in demountable seals. Not suitable for high temperature above  $\sim 100^{\circ}$ C applications.
- <u>Gold</u> wire is used as a gasket in demountable seals for ultra-high vacuum.
- <u>Tantalum</u> is acceptable in vacuum systems but it is difficult to work with due to its hardness. It is expensive so it is usually purchased in sheet form. Typically mounted as a shield where ion beams contact a surface as it has a low production of secondary electrons. Good for apertures and defining slit faces and anywhere that a metal edge is used to intersect the ion beam.
- <u>Zirconium</u> is corrosion-resistant. Like tantalum, it has low production of secondary electrons, so it is used as a shield of areas where reducing their production is important. It is used for neutron windows. It is expensive and rarekly used. Zirconium and zirconium hydride are used for gettering.

# **1.2 Plastics**

Plastics or ceramics are not to be used as the primary vacuum chamber structure without engineering advice from ANSTO or a specialist scientific vacuum instrument company. Plastic is very good for electrical insulators, bushes or light weight components are necessary in vacuums.

- <u>Polytetrafluoroethylene</u>, PTFE or Teflon as it is commonly known is suitable for use inside of vacuum systems from low to ultra high vacuums. Being soft PTFE can flow (creep) so for applications such as sample positioning systems or other systems requiring high stability then other plastics may need to be considered. PTFE has be far the highest dielectric strength, for extruded PTFE, 19.7 kV/mm 60 173 kV/mm for an insulating film. This compares with air which is around 1 kV/mm. It is self-lubricating, tolerant to fairly high temperatures, and has low outgassing. It is not suitable for barrier between vacuum and atmosphere, due to its permeability.
- <u>Ertalyte</u> offers the dimensional stability of acetal with the wear resistance of nylon. It is stable with relatively low outgassing values and has very low water absorption. It is very good for small mechanical loads requiring electrical isolation in vacuum systems.

- <u>Polyvinyl Chloride (PVC)</u> is acceptable in vacuum systems but not where heat is above 50°C. It is usually an amorphous thermoplastic material with excellent chemical resistance and dielectric properties, good tensile, flexural and mechanical strength, low moisture absorption, exceptional dimensional stability and good flammability characteristics.
- Other plastics can be used in vacuum systems but must have low vapour pressure. Care must be taken to ensure they are not used in applications that may require temperatures that will cause high outgassing rates.
- <u>Perspex</u> (acrylic plastic) is another plastic which has good dielectric properties but a high vapour pressure especially at raised temperatures making it unsuitable in vacuums however it can be used in small amounts in a well pumped high vacuum system.
- <u>Vespel</u>, a polyimide, is very expensive, but machines well, has good electrical insulator properties and is compatible with ultra-high vacuum. It does however absorb moisture and requires a longer pump down time. It also performs well in extremely low cryogenic temperatures. Good for bushes or where a mechanically stable plastic is required.
- <u>Nylon</u> is self-lubricating but has high outgassing rate and relatively high water absorption.
- <u>Polycarbonates and polystyrene</u> are good electrical insulators with moderate outgassing.
- <u>Mylar</u> is used to make thin windows that allow ion beams to penetrate from vacuum to positive pressures with little energy loss. For example gas detectors mounted onto beam lines for AMS have gas inside of the detector separated from the beam line which is under vacuum. Ion beams pass through the window as they move from the vacuum side to the positive pressure side. Other materials in this category include Kapton which is rated for higher temperatures.

# **1.3 Elastomers**

Some elastomers have sufficient vacuum properties and are used widely as vacuum seals in the form of 'o' rings.

- <u>Viton</u> is the standard seal used throughout the accelerator area as it is long lasting, low vapour pressure as compared with other elastomers and is bakeable to 200 °C.
- <u>Nitrile</u> rubber is used for vacuum seals. Does tend to break down over time. First signs of breakdown are small splits. If used for vacuum seals then they must be inspected regularly.
- <u>Natural</u> rubber is not typically used in vacuum systems other than inside of vacuum pumps usually on the low vacuum side of the system.
- <u>Silicone</u> rubber is not generally used as a seal in high vacuum environments. Silicone is soft when compared with other elastomers. It has a relatively low vapour pressure.

Further reading: Materials for high vacuum technology: an overview, S. Sgobba, CERN, Geneva, Switzerland. http://cdsweb.cern.ch/record/983744/files/p117.pdf

# 1.4 Ceramics and glass

• <u>Alumina</u> ceramic based ceramics in vacuum systems perform well provided the ceramic is not porous which can trap gas bubbles. Ceramics are readily available bonded onto metal flanges or mounting plates. Ceramic can also be baked if necessary to speed up the outgassing of surfaces.

Glass is similar to ceramics in this application. It is very low vapour pressure but difficult to work with. Glass viewports can be purchased already mounted in a metal flange.

- <u>Borosilicate glass</u> is often used for smaller assemblies and for viewports.
- <u>Porcelain</u> ceramics, when fully vitrified and therefore non-porous, are excellent insulators usable to 1500 °C. it is generally commercially available bonded to metal in electrical insulators and other feedthroughs.
- <u>Mica</u>, although it is neither ceramic or glass it best fits in this category. Mica has been used in vacuum systems for both electrical and thermal insulation but because Mica is a series of laminations it contains trapped air which is not suitable in high to ultra-high vacuums.
- <u>Macor</u> is a machinable glass made by Corning Incorporated with similar properties to ceramics. It has a low vapour pressure and thermally stable up to 1000°C making it bakeable. It is made from mica and borosilicate glass. It is by far the most flexible of the glass-ceramic options.

 Leakage Testing Handbook, Prepared for Liquid Propulsion Section, Jet Propulsion Laboratory, National Aeronautics and Space Administration, Pasadena, California
 Nondestructive Testing Handbook, Volume One, Leaktesting, American Society for Nondestructive Testing.
 Leakage Testing Handbook, Revised Edition, July 1969, General Electric.
 Fluid Flow in Small Passages, Mars Hablanian, J.W.Marr, Varian

# 1.5 Greases and oils

There are few acceptable greases and oils for use in vacuum systems. Users must ensure they purchase greases and oils specifically designed for use in vacuums. Typically they will have low vapour pressures, extremely low water absorption and easy to remove for cleaning.

- <u>Apiezon family</u> For further information http://www.apiezon.com/ . These have been used throughout the history of accelerators at ANSTO with excellent results. Wherever greases are used remember to use the minimalist amount.
- <u>Greases</u>
  - Apiezon H High Temperature Vacuum Grease, silicone and halogen free
  - Apiezon L Ultra high vacuum grease, silicone and halogen free
  - Apiezon M High vacuum grease, silicone and halogen free
  - Apiezon N Cryogenic high vacuum grease, silicone and halogen free
  - Apiezon T Medium temperature grease, silicone and halogen free
  - o Apiezon AP100 Ultra High Vacuum Lubricating Grease, silicone free
  - o Apiezon AP101 Anti seize Vacuum Grease, silicone free
- <u>Waxes</u>
  - Apiezon® Waxes and Compounds
  - Apiezon Wax W (Hard Vacuum Sealing)
  - Apiezon Wax W40 (Softer Vacuum Sealing)
  - Apiezon Wax W100 (Softest Vacuum Sealing)

- Apiezon Sealing Compound Q
- <u>Vacuum Oil</u> is used in few applications around the accelerator area. With a change to oil free pumps most oil consuming pumps are redundant. However these types of pumps may still in use in other applications. Oil vane roughing pumps and diffusion pumps were the largest consumers of oil. More recent brands of vane pumps were specifically tuned to oils of particular specifications so generic oils may not be compatible. Diff pumps are more forgiving and high quality generic synthetic oils may be used. The list below is a small sample of what is available and previously used.
  - $\circ\,$  Shell Vitrea 68 oil for roughing pumps. Check specification on pump to ensure compatibility with this mineral oil
  - Fomblin PFPE (Perfluorpolyether) oils designed for rotary pumps. Check specification on pump to ensure compatibility with this mineral oil
  - Apiezon® Diffusion Pump Fluids Apiezon AP 201 The Apiezon diffusion pump fluid is known for its exceptional higher degree of oxidation resistance.

# **Appendix 4 – Surface finishing**

In most cases a clean polished surface will yield the least outgassing as there are minimal surface traps for gas. Also, a polished surface is easier to clean and keep clean. Achieving a polished surface may not be practicable due to accessibility into tubes, chambers and small components but a combination of other methods may provide an adequate surface finish that will allow the desired vacuum level to be reached. The best method for a given surface will depend on the material, geometry and accessibility to its surfaces.

- <u>Polishing</u> A polished surface may be either smooth or uneven but the metal will lustre in either case. Polishing is a method of removing a layer of metal to expose clean metal underneath. This can be done with a mechanical buff or by hand with the aid of a polishing compound. Care must be taken to ensure all traces of the polishing compound are removed during final cleaning. All residues must be removed.
- <u>Grinding, sanding and other coarse to fine abrasive methods</u> These methods tear the surface dragging metal over metal. Some metal breaks away exposing clean metal underneath. The problem with this type of surface preparation is that the metal can fold over and not break away leading to voids of trapped gases. It is important that a fine grit is the last grit used to ensure larger metal drifts on the surface are torn away.
- <u>Grit blasting with grit or beads</u> This method propels grit or beads onto the metal surface causing fragments of metal to be removed with the impact. Like the abrasive techniques metal can be layered on metal (peening) if the grit is too coarse. Best to finish with a fine grit then a mechanical polish. Grit blasting can provide a more uniform surface finish and is very good for removing scale that may be present from rolling or extrusions.
- <u>Pickling</u> by far a very good way to clean welds and other heat induced decolourisations in the metal. It will remove a thin layer of metal, scale, oils, etc. Care must be taken to ensure any chemicals used are neutralised and thoroughly cleaned away. Residues cannot be tolerated. Makes general maintenance of the surface easier.
- <u>Passivating</u> a form of chemical polishing and surface protection. Can be stimulated with an electric current. Passivating is the spontaneous formation of a hard non-reactive surface film that inhibits further corrosion. It isn't a preferred method of surface preparation in vacuum systems as damaged surfaces can form micro cracks trapping water and other gases. For example, damage to anodised aluminium may allow aluminium oxide to form which can readily trap water. Passivation is not typically used for stainless steel as the surfaces are self healing, provided sufficient oxygen is available.

Further reading on surface passivation can be found at: http://www.euro-inox.org/pdf/map/Passivating\_Pickling\_EN.pdfGetter surfaces

• Nickel Plating – has been used on vacuum components to provide stability to the surface of normally oxidising metals such as steel, copper and brass. It is important to ensure that the nickel has uniform bonding to the parent metal. Cases have been observed where the plating has lifted creating voids in the vacuum space. Although nickel is resistant to corrosion it will form a thin layer if exposed to moist atmospheres. A periodic clean is recommended to minimise problems with corrosion.

Element	Melting Point °C	Critical Temp °C	Phase at Critical T
Aluminium	660	1124	Liquid
Americium	1176	896	Solid
Antimony	631	455	Solid
Arsenic	84	270	Liquid
Barium	729	574	Solid
Beryllium	1289	1102	Solid
Cadmium	321	226	Solid
Caesium	29	114	Liquid
Calcium	847	538	Solid
Californium	900	1402	Liquid
Cerium	798	1602	Liquid
Chromium	1863	1220	Solid
Cobalt	1492	1418	Solid
Copper	1084	1146	Liquid
Dysprosium	1412	1025	Solid
Erbium	1529	1139	Solid
Europium	822	547	Solid
Gadolinium	1312	1450	Liquid
Gallium	30	954	Liquid
Germanium	938	1260	Liquid
Gold	1064	1291	Liquid
Hafnium	2231	2255	Liquid
Holmium	1474	1146	Solid
indium	157	836	Liquid
Iridium	2447	1199	Solid
Iron	1538	1253	Solid
Lanthanum	918	1586	Liquid
Lead	328	622	Liquid
Lithium	181	465	Liquid
Lutetium	1663	1517	Solid
Magnesium	651	386	Solid
Manganese	1246	887	Solid
Mercury	-39	22	Liquid
Molybdenum	2623	2319	Solid
Neodymium	1021	1219	Liquid
Neptunium	639	1781	Liquid
Nickel	1455	1409	Solid
Niobium	2468	2501	Liquid
Osmium	3033	2721	Solid
Palladium	1555	1348	Solid
Platinum	1769	1800	Liquid
Plutonium	640	1506	Liquid
Potassium	64	165	Liquid
Praseodymium	931	1381	Liquid
Protactinium	1572	2636	Liquid
Rhenium	3186	2817	Solid
Rhodium	1963	1874	Solid
Rubidium	39	129	Liquid

# **Appendix 5 – Critical Vapour Pressures**

Substance	Vapour Pressure (Pa)	Temperature (°C)
Acetaldehyde	98.7	20
Acetone	24640	20
Butane	22	20
Carbonyl sulphide	1255	25
Ethanol	5.83	20
Ethylene glycol	0.5	20
Formaldehyde	435.7	20
Freon 113	37.9	20
Methanol	12800	20
Methyl isobutyl ketone	26.48	25
Nitrogen (N <sub>2</sub> )	63200	20
Oxygen (O <sub>2</sub> )	54200	20
Propane	2200	55
Propanol	2.4	20
Tungsten	0.1	3203
Water (H <sub>2</sub> O)	2.3	20
Xenon difluoride	0.6	25

# **Appendix 5 – Vapour Pressures continued**

Substance	Vapour Pressure (Pa)	Melting point (°C)
Apiezon Oil J	0.13 at 200°C 1.3 x 10 <sup>-4</sup> at 20°C	-
Apiezon Oil K	0.13 at 300°C 1.3 x 10 <sup>-7</sup> to 10 <sup>-8</sup> at 20°C	-
Apiezon Grease L	0.13 at 100°C 1.3 x 10 <sup>-7</sup> to 10 <sup>-9</sup> at 20°C	47
Apiezon Grease M	0.13 at 200°C 1.3 x 10 <sup>-5</sup> to 10 <sup>-6</sup> at 20°C	44
Apiezon Grease N	0.13 at 200°C 1.3 x 10 <sup>-6</sup> to 10 <sup>-7</sup> at 20°C	43
Apiezon Grease T	About 1.3 x 10 <sup>-6</sup> at 20°C	125
High Vacuum Grease Dow Corning	< 1 x 10 <sup>-4</sup> at 20°C	-
Nylon	~ 1 x 10 <sup>-3</sup> at 20°C	-
Teflon	< 1 x 10 <sup>-4</sup> at 20°C	-
Viton	1 x 10 <sup>-6</sup> at 20°C	-

# Appendix 6 – Outgassing tables for various materials Reference site: http://home.fnal.gov/~mlwong/outgas\_rev.htm

					i sampies.	
Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	(hours)	Test method	Reference	Year
Aluminium	None	1x10 <sup>-6</sup>	1h		Schamus (ref Markley, et al)	1999
Aluminium	Degassed	$1.7 \times 10^{-7}$	1h		Schmaus (ref Markley, et al)	1999
Aluminium	Degassed	2.7x10 <sup>-8</sup>	10h		Schmaus (ref Markley, et al)	1999
Aluminium 6061-T6	Baked 13.5h @ 300°C	1.4x10 <sup>-8</sup>	10h		Schmaus (ref Das)	1999
Aluminium	Cleaned	8x10 <sup>-9</sup>	10h		Schmaus (ref Blears, et al)	1999
Aluminium	Fresh, degreased w/ trichloroethylene & cleaned w/ ethyl alcohol	6.3x10 <sup>-9</sup>	1h	conductance	Elsey (ref Schram)	1975 (1963)
Aluminium	Degassed	$4.6 \times 10^{-9}$	100h		Schmaus (ref Markley, et al)	1999
Aluminium 6061-T6	Baked @ 200°C	4.5x10 <sup>-9</sup>	10h		Schmaus (ref Das)	1999
Aluminium	Degassed 24h, degreased w/ trichloroethylene & cleaned w/ ethyl alcohol	4.14x10 <sup>-9</sup>	1h	conductance	Elsey (ref Schram)	1975 (1963)
Aluminium 6061-T6	None	2.5x10 <sup>-9</sup>	10h		Schmaus (ref Das)	1999
Aluminium 5083-O	Bell jar, as received, room temp.	2.18x10 <sup>-9</sup>	10.3h	Rate-of-rise	Schrank, et al	1964
Aluminium 5083-O	Bell jar, as received, room temp.	1.27x10 <sup>-9</sup>	8h	Conductance	Schrank, et al	1964
Aluminium 5083-O	Bell jar, as received, room temp.	6.6x10 <sup>-10</sup>	24h	Conductance	Schrank, et al	1964
Aluminium 5083-O	Bell jar, as received, room temp. after baking @ 220°C	$4.6 \times 10^{-10}$	50h (baked @27-31h)	Conductance	Schrank, et al	1964
Aluminium 6061-T6	Baked 15h @ 300°C	$1.6 \times 10^{-10}$	10h		Schmaus (ref Das)	1999
Aluminium	Degassed 24h, degreased w/ trichloroethylene & cleaned w/ ethyl alcohol	3.06x10 <sup>-10</sup>	1h	conductance	Elsey (ref Schram)	1975 (1963)
Aluminium 5083-O	Bell jar, no. 4 finish (lapped), room temp.	$2.87 \times 10^{-10}$	6.0h	Rate-of-rise	Schrank, et al	1964
Aluminium	Fresh, degreased w/ trichloroethylene & cleaned w/ ethyl alcohol	6.0x10 <sup>-10</sup>	10h	conductance	Elsey (ref Schram)	1975 (1963)
Aluminium, type 1100	Cleaned w/ detergent, rinsed w/ acetone, pumped 24 hours	~10 <sup>-10</sup>	0	conductance	Young	1968
Aluminium	LEP vacuum chamber, chem clean, baked in situ @ 150°C	$2.3 \times 10^{-11}$	24h		Mathewson, et al	1988
Aluminium 6061-T4	Degreased in acetone w/ methanol rinse; baked 100°C	6x10 <sup>-12</sup>	24h	Rate-of-rise & conductance	Halama, Herrera	1976

Outgassing rates of aluminium Note the different methods of measurement and treatment of samples.

Aluminium 6061-T4	Degreased in acetone w/ methanol rinse, chem polished, baked 100°C	3x10 <sup>-12</sup>	24h	Rate-of-rise & conductance	Halama, Herrera	1976
Aluminium 6061-T4	Degreased in acetone w/ methanol rinse, chem polished, glow discharge in Ar, baked 100°C	5x10 <sup>-13</sup>	24h	Rate-of-rise & conductance	Halama, Herrera	1976
Aluminium, type 1100	Above plus baked 15h @ 250°C under vacuum	$4x10^{-13}$	24h @ room temp	conductance	Young	1968
Aluminium	PETRA vacuum chamber, glow discharge@145°C	$1 \times 10^{-13}$	Up to 200h	In situ glow discharge, conductance	Mathewson, et al	1977

# Outgassing rates of stainless steels, along with the surface treatment and pumping time.

Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	Time (hours)	Test method	Reference	Year
Stainless steel	None	6.4x10 <sup>-7</sup>	1h		Schamus (ref Markley, et al)	1999
Stainless steel	degreased	4x10 <sup>-7</sup>	1h		Schamus (ref Markley, et al)	1999
Stainless steel	None	2x10 <sup>-7</sup>	1h		Schamus (ref Blears, et al)	1999
Stainless steel	None	2x10 <sup>-8</sup>	10h		Schamus (ref Blears, et al)	1999
Stainless steel NS22S	Fresh	1.4x10 <sup>-8</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel ICN 472	fresh	1.3x10 <sup>-8</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel	None	1.2x10 <sup>-8</sup>	10h		Schamus (ref Das, et al)	1999
Stainless steel ICN 472	sanded	8.3x10 <sup>-9</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel NS22S	electro polished	4.3x10 <sup>-9</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel NS22S	mechanically polished	1.7x10 <sup>-9</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel	fresh	1.5x10 <sup>-9</sup>	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel ICN 472	Polished & vapor degreased	1.4x10 <sup>-9</sup>	10h		Schamus (ref Dayton, et al)	1999
Stainless steel	None	1.4x10 <sup>-9</sup>	10h		Schamus (ref Das, et al)	1999
Stainless steel NS22S	fresh	1.3x10 <sup>-9</sup>	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel ICN 472	sanded	1.0x10 <sup>-9</sup>	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel	None	7.6x10 <sup>-10</sup>	1h		Schamus (ref Das, et al)	1999
Stainless steel NS22S	mechanically polished	$4.6 \times 10^{-10}$	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel NS22S	electro polished	$4.3 \times 10^{-10}$	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel	Baked 24h @ 200°C	$1.5 \times 10^{-10}$	1h		Schamus (ref Das, et al)	1999

Stainless steel	None	$1.1 \mathrm{x} 10^{-10}$	100h		Schamus (ref Das, et al)	1999
Stainless steel 304	Degrease + water rinse	$4.0 \times 10^{-11}$	40h	Conductance	Strausser	1973
Stainless steel U15C	Baked 25h @ 300°C	$4.5 \times 10^{-12}$			Elsey (ref Calder, et al)	1975
Stainless steel 304	Degrease + water rinse, baked in vacuum 150°C for 12h	4.0x10 <sup>-12</sup>	5h after bakeout	Conductance	Strausser	1967
Stainless steel 304	Electropolished, baked 30h @ 250°C	$3.0 \times 10^{-12}$			Elsey (ref Young)	1975
Stainless steel U15C	Baked 45h @ 360°C	$2.6 \times 10^{-12}$			Elsey (ref Calder, et al)	1975
Stainless steel	Baked 24h @ 200°C	$9.3 \times 10^{-13}$	100h		Schamus (ref Das, et al)	1999
Stainless steel U15C	Baked 3h in vacuum @ 1000°C + 25h in situ @ 360°C	1.6x10 <sup>-14</sup>			Elsey (ref Calder, et al)	1975

Other cleaning methods although outgassing rates are not measured:

Material	Treatment	Results	Reference	Year
Aluminium	Quench a hot aluminium extrusion in an Ar- $O_2$ atmosphere	Dense, thin (~20Å thick) oxide layer	Sasaki, Y.T.	1990
Aluminium 6063	Clean with an alkaline detergent (Almeco 18)	Removes MgO, C, and Al <sub>2</sub> O <sub>3</sub>	Sasaki, Y.T.	1990
Aluminium	Vapour degreasing, pump for 120h before and after bake	Dominant residual gases: H <sub>2</sub> O, H <sub>2</sub> , CO, CO <sub>2</sub> , & CH <sub>4</sub> ; after baking @ 150°C and pumping, H <sub>2</sub> O & CH <sub>4</sub> gone, outgassing rates of other gases reduced 1 order of magnitude except H <sub>2</sub>	Mathewson, A.G., et al	1989
Aluminium	Vapour degreasing, then light alkaline etch (CERN basic procedure), pump for 120h before and after bake	Dominant residual gases: H <sub>2</sub> O, H <sub>2</sub> , CO, & CO <sub>2</sub> ; after baking @ 150°C and pumping, only H <sub>2</sub> exists	Mathewson A.G., et al	1989
Aluminium	Strong alkaline etching in NaOH, pump for 120h before and after bake	Dominant residual gases: H <sub>2</sub> O, H <sub>2</sub> , CO, CO <sub>2</sub> , & CH <sub>4</sub> ; after baking @ 150°C and pumping, only H <sub>2</sub> exists	Mathewson A.G., et al	1989
Aluminium	Vapour degreasing, Ar glow discharge @ 150°C	On initiation of discharge, large pressure increase (normal in Ar glow discharge) was too large for accurate measurement	Mathewson A.G., et al	1989
Aluminium	Vapour degreasing, then light alkaline etch (CERN basic procedure), Ar glow discharge @ 150°C	H <sub>2</sub> , CO dominant gases desorbing from surface during discharge cleaning	Mathewson A.G., et al	1989
Aluminium	Strong alkaline etching in NaOH, Ar glow discharge @ 150°C	H <sub>2</sub> , CO dominant gases desorbing from surface during discharge cleaning at order of magnitude less than light alkaline etch	Mathewson A.G., et al	1989
SS	H <sub>2</sub> glow discharge - 22°C	Dominant residual gases: $CH_4$ , $CO$ , & $C_2H_4$ ; water production insignificant; frequent arcing	Dylla, H.F.	1988

		on all exposed surface noted during initial discharge period		
SS	H <sub>2</sub> glow discharge – 150°C	Dominant residual gases: $CH_4$ , $CO$ , & $C_2H_4$ , and $H_2O$ ; frequent arcing on all exposed surface noted during initial discharge period	Dylla, H.F.	1988
SS	Ar glow discharge – ambient temp	Dominant residual gases that were significantly removed: $CO_2$ , $H_2$	Dylla, H.F.	1988
SS	Ar/O <sub>2</sub> glow discharge	Improved removal of CO and CO2; minor amts of implanted Ar that can be removed by baking @ 350°C	Dylla, H.F.	1988
	Ar/O <sub>2</sub> glow discharge 2h, exposed to air, then Ar/O <sub>2</sub> glow discharge 2h	Some degree of passivation: residual gases were removed with 1 <sup>st</sup> cleaning & did not show up during the 2 <sup>nd</sup> cleaning	Dylla, H.F.	1988
	O <sub>2</sub> glow discharge	Rapid removal of carbon, not of hydrocarbons (which can be baked out initially), increased surf. Oxidation, sputtering of the base metal, and possible enhanced outgassing of O <sub>2</sub> - containing molec.	Dylla, H.F.	1988

# Different outgassing measurements of epoxies

Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	Time (hours)	Test method	Reference	Year
NMA w/ solid alumina filler	Cured 300°F	$1.6 \times 10^{-7}$			Hanson, Patel	1970
Polyimide polymer	Thermosetting; measured @ 40°C	~1.1x10 <sup>-7</sup>	4h+9min	conductance	Kendall, Zabielski	1965
Polyimide polymer	Thermosetting; measured @ 155°C	~5x10 <sup>-8</sup>	4.5h increase temp + 6h	conductance	Kendall, Zabielski	1965
Silicone resin	Needs dicumyl peroxide catalyst; thermosetting, measured @ 35°C	~5x10 <sup>-8</sup>	4h	conductance	Kendall, Zabielski	1965
Araldite epoxies	Cured ~150°C for 15h	~10 <sup>-8</sup>	51h pumping	conductance	Barton, Govier	1965

# Kapton is a newer material and little data exists about its outgassing rate. Below are 3 references

Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	Time (hours)	Test method	Reference	Year
Kapton cable		1.0x10 <sup>-5</sup>	0h	conductance	Postma	1999
Kapton foil		$1.0 \times 10^{-7}$	40h		Ferro-Luzzi	1999
Polyimide	Bake @ 300°C	$4.0 \mathrm{x} 10^{-8}$	12h		Weston	1970
Kapton cable		$2.4 \times 10^{-8}$	168h	conductance	Postma	1999
Kapton cable		2.7x10 <sup>-9</sup>	336h	conductance	Postma	1999
Kapton cable		$6.5 \times 10^{-10}$	504h	conductance	Postma	1999

# Outgassing rates of several types of plastics

Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	Time (hours)	Test method	Reference	Year
Nylon		1.2x10 <sup>-5</sup>	1h		Elsey (ref	1975
					Power, et al)	
Nylon		6.0x10 <sup>-6</sup>	1h		Elsey (ref	1975

					Power, et al)	
PVC	24h @ 95% relative	8.5x10 <sup>-7</sup>	1h	conductance	Elsey (ref	1975
	humidity				Santler)	
PTFE	Fresh	$1.7 \mathrm{x} 10^{-7}$	1h	conductance	Elsey (ref	1975
					Santler)	
Teflon		6.5x10 <sup>-8</sup>	1h	conductance	Elsey (ref	1975
					Santler)	
PTFE	Fresh	3.3x10 <sup>-8</sup>	10h	conductance	Elsey (ref	1975
					Santler)	
Teflon		$2.5 \times 10^{-8}$	10h	conductance	Elsey (ref	1975
					Santler)	
PVC	24h @ 95% relative	$2.0 \times 10^{-8}$	10h	conductance	Elsey (ref	1975
	humidity				Santler)	
G-10		~10 <sup>-8</sup>			Beams	2001
					Division -	
					FNAL	

Reference to the following has been lost but it is believed to have originated in an early Vacuum Catalogue from the 1990's

 $K_1$  is the outgassing rate (air equivalent) after 1 hour pumping  $\alpha_1$  is the slope of the (log K - log t) curve at 1 hour

Material	K <sub>1</sub> Torr litre/sec cm <sup>2</sup> (x 10 <sup>-10</sup> )	α1	K <sub>10</sub> Torr litre/sec cm <sup>2</sup> (x 10 <sup>-10</sup> )	a <sub>10</sub>
Aluminium Alloy (fresh)	63	1	6.0	1
Aluminium Alloy (degassed 24 hours)	41.4	3.2	3.06	0.9
Aluminium Alloy (3 hours in air)	65.5	1.9	4.75	0.9
Aluminium Alloy (anodised -2µ pores)	2760	0.9	322	0.9
Aluminium Alloy (bright rolled)	-	-	75	1
Duralumin	1700	0.75	350	0.75
Brass (wave guide)	4000	2.0	100	1.2
Copper (fresh)	400	1	41.5	1
Copper (mechanical polish)	35	1	3.56	1
OFHC Copper (fresh)	118	1.3	12.6	1.3
OFHC Copper (mechanical polish)	19	1.1	1.63	1.1
Gold (fresh wire)	1580	2.1	5.1	1
Mild steel	5400	1	500	1
Mild steel (slightly rusty)	6000	3.1	130	1
Cr plated steel (fresh)	70.5	1	6.8	1
Cr plated steel (polished)	91	1	8.0	1
Ni plated steel (fresh)	42.4	0.9	4.84	0.9
Ni plated steel	27.6	1.1	2.33	1.1
Chemically Ni plated steel (fresh)	83	1	7.05	1
Chemically Ni plated steel (polished)	52.2	1	4.6	1
• • • • •				

Araldite (moulded)	116	0.8	35.2	0.8
Araldite D	800	0.8	220	0.78
Araldite D	190	0.3	125	0.5
Araldite F	150	0.5	73	0.5
Celluloid	860	0.5	430	0.5
(PTFE) (fresh)	16.6	0.8	3.31	0.9
Kel-F Oak ridge	4	0.57	1.7	0.53
Methyl methacrylate	420	0.9	140	0.57
Mylar V-200 (24 hr at 95% RH)	230	0.75	40	-
Nylon	1200	0.5	600	0.5
Pertinax	620	0.18	290	0.5
Perspex	72	0.44	27	0.44
Perspex	310	0.4	180	0.4
Polyamid	460	0.5	230	0.5
Polyester - glass laminate	250	0.84	80	0.81
Polyethylene	23	0.5	11.5	0.5
Polystyrene	2000	1.6	200	1.6
Polystyrol	56	0.6	12	0.61
Polyvinylcarbazol	160	0.5	80	0.5
PTFE	30	0.45	15	0.56
P.V.C. (24 hr at 95% RH)	85	1.00	2	-
Teflon	6.5	0.6	2.5	0.2
Terephenil (fresh)	62.2	0.5	16.8	0.5
Neoprene	3000	0.4	-	-
Viton	114	0.8	-	-
	1	1		

# **Appendix 7 – Local Plant and Safety Regulations**

Regular maintenance inspections are carried out on ANTARES and STAR accelerator vacuum equipment to ensure operational status, vacuum quality, vacuum pump performance, and the mechanical and electrical integrity of the systems.

Generally, the integrity of the vacuum systems can be measured as a direct function of the known static vacuum pressures in a given vacuum system. That is, a lowering of vacuum from the known average vacuum pressure in a given system will indicate either/and, a leak in a seal, a failing vacuum pumping system or a structural failure.

The following is an overview of the application of the ANSTO OHSE Plant and Safety Standard, AS 2309, to vacuum systems in the accelerator facilities. *Reference: http://docushare.ansto.gov.au/Get/File-32340/Plant\_Safety\_Standard.pdf* 

Risk	Hazard	Risk rating	Controls	Risk rating (with controls in place)
Catastrophic failure vacuum vessels	Implosion – scattering of material fragments and loose fittings becoming missiles	Very low	<ul> <li>Use of vacuum standards to design equipment for safe usage.</li> <li>Protective barriers around equipment and fittings that may be susceptible to knocking</li> </ul>	Very low
Catastrophic failure vacuum beam lines and associated equipment	• Implosion – scattering of material fragments and loose fittings becoming missiles	Very low	<ul> <li>Use of vacuum standards to design equipment for safe usage.</li> <li>Purchased equipment from reputable companies/designers only</li> </ul>	Very low
	• Electric shock from fault in high vacuum gauge	Very low	• Vacuum gauges using high voltages in key areas will automatically switch off at low vacuums/atmospheric pressure	Very low
	• Failure of welded joint	Very low	• System will leak to atmospheric pressure at a rate commensurate with the conductance of the failed joint. No control is deemed necessary.	Very low
Electric shock from a failed vacuum pump or powered fitting	Electric shock from frayed lead or failure of earth inside of equipment	Low	<ul> <li>All equipment is protected from short circuits by circuit breaker protection.</li> <li>Most circuits now have RCD protection.</li> <li>A program is in place to put all circuits onto RCD protection</li> </ul>	Very Low
Failure of support structures	Crush, fall, bump, trip hazards during and after failure	Low	<ul> <li>Use of vacuum standards to design equipment for safe usage</li> <li>Access to equipment at height via approved ladders or dedicated platforms</li> <li>Non acceptance of standing on beam line stands and mounts</li> </ul>	Very Low
breakages to	<ul> <li>Fragments of broken</li> </ul>	LOW	<ul> <li>Barriers around exposed equipment</li> </ul>	LOW

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fittings and equipment protruding from vessels and beam lines	<ul> <li>parts become missiles.</li> <li>Cuts to personnel bumping into fittings</li> <li>Secondary injuries including electric shock from exposed wiring</li> </ul>		<ul> <li>that is deemed to be hazardous</li> <li>Access control into accelerator areas restricted to accelerator personnel and visitors who are escorted</li> <li>See electric shock hazard controls above</li> </ul>	
Over pressure of vacuum systems during venting	Positive pressure in beam lines when venting with gases from high pressure storage	Very Low	<ul> <li>Pressure relief valves in use at systems where regular vacuum cycling is necessary</li> <li>Use of portable pressure relief fittings where local venting is required.</li> </ul>	Very Low
Implosion of glass view ports	Shards of glass scattering towards operators	Low	<ul> <li>Use purpose designed commercial vacuum windows</li> <li>Use small diameter (&lt;50mm x 6 mm thick) quartz windows on vacuum systems where ion beams can strike. Quartz is not known to crack through but to develop small surface anomalies</li> <li>Use double rubber seals to support windows to minimise mechanical stresses</li> </ul>	Low
Broken film windows	Sudden venting of vacuum to atmospheric pressure	Very Low	<ul> <li>Damage to equipment – high vacuum gauges must have auto shutdown at low vacuums</li> <li>Vacuum system isolated with gate valve to minimise loss of vacuum in adjoining systems</li> </ul>	Very Low
Breach of accelerator tubes with SF6	Vacuum systems pressurised with SF6 gas	Low	<ul> <li>Large gate valves either end of the accelerator vacuum tubes will shut if pressure rises to maintain SF6 within the vacuum vessels</li> <li>Some gas will enter the vacuum systems outside of the isolated section. The pressure will be relieved through blow off venting ports at each end of the vessel.</li> <li>Oxygen depletion alarms will sound if dangerous levels of SF6 are released. Personnel will be ordered to leave building through PA announcement</li> </ul>	Low

- 2) Risk control measures for eliminating or reducing the assessed risks *See table above*
- 3) Registration or notification of design of plant *Not deemed necessary. See AS 4343-2005*
- 4) Licensing of plant Not deemed necessary. See AS 4343-2005
- 5) Maintenance of plant

*Regular maintenance applied via Accelerator Maintenance system. See ENV-I-076-004 Vacuum Systems Maintenance manual* 

- 6) Plant operations and risk controls *See table above*
- 7) Requirements in relation to specific types of plant *Only vacuum system plant*
- 8) Training and supervision Vacuum technology training is provided in-house to world's best practices. Additional training has been provided by the Vacuum Society of Australia through their training courses.
- 9) Monitoring and evaluation Regular maintenance applied via Accelerator Maintenance system. See ENV-I-076-004 Vacuum Systems Maintenance manual

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# Vacuum Technology

# and

# Vacuum Design Handbook

# for

# **Accelerator Technicians**

Prepared by David Garton November 2011 Revision 0 Vacuum Technology and Vacuum Design Handbook for Accelerator Technicians

This handbook is a compilation of information gathered from over 50 years of direct hands-on experience to applicable information widely available from the vacuum technology industry. It seeks to address common and specific vacuum technology problems whilst clarifying the design standards and philosophies adopted for use in the ANSTO accelerator facilities. The author wishes to thank the reviewers and the many technicians from ANSTO that have contributed directly and indirectly to this booklet.

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# Chapter 1

# Vacuum design standards and good practises for the Accelerator Area

- \* This chapter must be read in conjunction with the detailed vacuum information in this document.
- \* Based on best practises developed at ANSTO and other local Australian accelerator laboratories in conjunction with Australian Standards and industry technical guides, referenced throughout this document.
- \* Where a standard is unavailable for a specific design, best practices are used from industry's best practises.
- \* *Refers to all vacuum chambers, beam lines and other enclosures used on the accelerators or stand alone.*

# 1. Basic design

- All vacuum vessel designs must be consistent with the design standards in Australian Standard AS 1210-2010 and maintain a Hazard Level of E as per the design conditions tabled in "Hazard Levels of Pressure Equipment" in Section 2, Hazard Levels of Pressure Equipment in Australian Standard AS 4343-2005.
  - The vacuum equipment designed by/for the accelerator area maintains a Hazard level of E. All pressure equipment with a pV  $\leq$  30, pressure (p in MPa) x volume (litres)  $\leq$  30 is classed as Hazard level of E. For vacuum equipment in Hazard Level E the derating value of 0.1 is applied, ie 0.1pV  $\leq$ 30. Note the amount of stored energy at this level is very small. For example a Ø 1000 mm chamber, 1000 mm high will have a pV for vacuum equal to 7.85.
  - AS 4343-2005, Section 2.1.2 Typical hazard levels, part (d), states for Hazard Level E, "This equipment is usually exempt from special regulatory control but is <u>covered</u> by general plant safety regulations". See Appendix 7.
- All designs of chambers excluding general beam lines and fittings should be checked for structural integrity using finite elements analysis. Sound designs with acceptable structural integrity will be released for manufacture. A copy of the design acceptance tests must be filed with the project file. The standard finite element analysis can be performed utilising Solidworks. (Modelling for irregular shaped chambers, calculations Appendix 1 where appropriate for regular shapes)
- Stainless steel (304 or 316) will be used to construct chambers, beam lines and general fittings unless it is a *special* requirement to use other metals. Do not choose substances that have high outgassing rates or can contaminate vacuum systems.
- Measurement chambers and other large volume chambers should be cylindrical unless the chamber is an enclosure between pole faces (eg magnet box) or is an odd shape to accommodate steering plates or devices. When non-cylindrical chambers are required deflection of chamber walls should be modelled and taken into consideration.
- Other chambers including magnet boxes, ESA and deflector coffins to have suitable rib strengthened rectangular or curved sides where it is not practicable to increase the wall thicknesses.
- Minimum wall thickness calculation methods can be found in Appendix 1. As a general rule care must be taken when using formulas for vacuum calculations as historically many units other than SI units have been used to form some equations. For example length in cm, and pressure in torr is common in old notes.

- Top and bottom flanges to have integrated structural support rim which becomes the flange and seal for chamber base and top (user) flanges.
- Ports must be a suitable length to ensure unobstructed insertion for screws into flanges. Suitable weld preparation must allow for adequate weld penetration. See Welds below.
- All internal edges must have a smooth radius. No sharp edges to be left after welding or surface finishing.
- All machined finishes other than vacuum seals to be equal to or better than 1.6 microns
- No sections within the vacuum space will have metal to metal contact where gas can be trapped unless it is at the edge of a seal joint. When this is necessary, pump out grooves must be incorporated in the design.
- Vacuum windows shall be made from toughened glass or quartz. No plastic to be used for windows unless the window is a flange on a chamber that is designed to withstand more than 100 kPa. The large plastic flange/windows should be made from >18 mm thick up to Ø200 and >25 mm thick for up to Ø400 in clear Perspex or polycarbonate.
- For ANTARES beam lines the preferred flanges are Dependex, nominal diameter 100 mm. See section 8 for detail on Dependex flanges.
- For STAR there is no preferred flange type. It must be selected depending on the desired base vacuum and interface to other adjoining flanges.
- As a guide, the MDC catalogue from Vacuum Products Corporation 2003 onwards, Building Blocks for Vacuum Science and Technology, provides vacuum tubing dimensions for most common sizes. The dimensions must be assumed to be minimum dimensions. Other tubing sizes should be calculated using the formulas in Appendix 1.
- Vacuum pumping ports leading to the vacuum pumps must be designed to optimise conductance.
- Pressure relief valves to be incorporated where a vacuum system can be vented to >10 kPa above atmospheric pressure.
- ASME state that the design of an external pressure vessel must consider Material type, diameter of chamber, unstiffened length, temperature and wall thickness

# 2. Welds

- TIG is preferred for vacuum sealing welds as minimal scale and flux is produced. Electron beam welding is suitable for thin walled materials. TIG produces high quality welds with or without filler rods to produce flat, smooth well penetrated welds. TIG comes into its own where the weld preparation allows for melting together of the parent materials without filler rods. MIG has a higher deposition of filler material so care must be taken to ensure welds are kept smooth and flat.
- No standard exists for vacuum welding of compact scientific vacuum equipment where pressures approaching zero are necessary. Equipment designed in the accelerator area and welded in the ANSTO workshops since 1990 has had penetrations of ≥1mm but ≤2mm on a butt weld and ≥2 mm on a fillet weld. Experience has demonstrated the adequacy of these figures. No failure reported.
- For thin walled materials ie < 1 mm, such as bellows, manufacturers' will use their industry standard. ANSTO does not weld thin wall materials.
- Weld preparation must be provided where possible to ensure even heat penetration.

- Electric arc with rods is not recommended due to flux trapping. This can cause pits which have long lasting outgassing periods.
- All welds that form a seal against atmospheric or other positive pressures must be made to the internal side (vacuum side) of the joint.
- Tack (stitch) welding is recommended on the external side only with less than 50% of the diameter covered in small intervals. This minimises the chance of trapped voids and facilitates He leak testing post welding.
- All welds must be free from pitting and scale. Must be cleaned to bare metal. If chemicals are used to clean welds care must be taken to remove all traces of chemicals.
- Some chambers such as magnet boxes will need to have some joints welded externally due to access problems on thin chambers. In this case, good penetration must be achieved to ensure minimal gas trapping points.

# 3. Surface finishes

- The inside of the chamber and ports must be 1.6 micron or better for mechanical finishes.
- Can be chemically cleaned (polished) but all traces of chemicals must be neutralised and removed prior to use in vacuum.
- All machining oil to be removed with an alcohol scrub then acetone rinse, minimum. The use of kerosene, turpentine or other oily solvents is not encouraged however where they are used the final clean must be with acetone followed by alcohol. Water based cutting fluids can be used as they are more readily removed therefore having less effect on outgassing.
- Nitrile gloves to be used when assembling cleaned components. Other un-powdered gloves may be used but only those that do not react with the solvents being used.
- Care must be given to cleaning and handling surfaces that will be exposed to vacuum. Gloves should be used to minimise the transfer of dirt and oils from the skin to vacuum surfaces.
- A suitably clean area should be used for the preparation of vacuum systems and all tolls used should also be cleaned (degreased).
- Stainless steel can be cleaned in nitric acid to remove scale that remains from rolling or wire cutting. It is may also be used to prepare stainless steel components for use in ultra-high vacuum systems.

# 4. Flanges

- Flanges must be standard flanges selected from the group referenced this document for compatibility reasons. Exceptions may be made but all 'o' ring seals must be to Australian Standard AS 2842-1986. External flange rings not in contact with the vacuum space may be aluminium alloy. Stainless flanges are not economical or necessary unless the seal requires forces to crush metal seals such as copper ConFlat, aluminium or indium wire.
- Minimum thicknesses for top and bottom flanges to be calculated using the standard in Appendix 1 Structural Calculations for Scientific Vacuum Vessel Design.
- All other flanges to have threads consistent with their design standard.
- ANTARES Primary flanges will be Dependex unless interface is necessary to accommodate other standard flanging.
- For Dependex, screws to be imperial sizes consistent with UNC series.

• STAR utilises many of the major vacuum flange/seal designs. The most appropriate design must be chosen that meets the specific application.

# 5. Seals

- 'O' ring sizes and sealing groove dimensions must comply with the Australian Standard AS 2842-1986. Adjustments to groove sizes can be made where specific 'crush' is necessary for special applications.
- Viton rubber 'o' rings to be used unless a special requirement for an alternate elastomer is identified.
- Standard flange and seal sizes to be used unless an application prohibits their use.
- It is recommended that vacuum sealing surfaces are cleaned just before a seal is made to minimise the possibility of dust and hair settling on sealing surfaces.
- Metal seals may be used in specifically designed sealing surfaces including, aluminium and indium wires. They are very good as ultra-high vacuum seals.

# 6. Screws

- All screws for flanges other than Conflat will be made from stainless steel in either 304 or 316 grades.
- Screws used in Conflat flanges will be made from A2-70 or A4-70.
- All screws to be socket head unless access necessitates hexagon or other special head. Slotted or Philips head must not be used.
- Stainless steel flat washers to be used under all screws where practicable. Do not use lock or star washers on standard flanges.
- In the vacuum space, no screws to be used in holes unless the holes are through holes and can be pumped from both sides. Screws can be used in blind holes if they have a minimum Ø1 mm hole drilled through the centre to allow trapped gas/air to escape.
- Unless a standard flange uses non metric thread, all screw threads must be within the metric series. Unless there is a specific need, avoid using intermediate metric sizes, that is, 0.5 mm steps such as M1.5 mm, M2.5 mm etc.
- Anti-seizing compounds can be used on threads but extreme care must be taken to ensure they do not enter the vacuum space. Graphite powder can be used as a lubricant inside the vacuum space. It can be applied using ethanol to wet the surface to be lubricated. Allow solvent to evaporate prior to pumping.

# 7. Feedthroughs

- Sliding seals should have dual 'o' ring seals with a gland formed seal at least at one end. The shaft finish must be 0.8 micron or better.
- All sliding shafts must have retainers to stop them being pulled through into the vacuum space.
- All coax feedthroughs to be glass or ceramic hermetically sealed. No plastic.
### 8. Lubricants, vacuum greases and epoxies

- Only vacuum specific grease and oils to be used in vacuum systems.
- Silicone based or particular Apiezon family lubricants design for vacuum use, are recommended on sliding seals. Thin smears only.
- No vacuum greases should be used unless there is a small leak through a damaged sealing surface and all other options are not feasible. If grease must be used it must be designed for high vacuum use and only a very thin smear to be used.
- Do not use grease to fill suspected leaks in welds or fittings.
- Where a vacuum epoxy must be used the surface must be well prepared to ensure appropriate bonding. Care must be taken to ensure the epoxy seal doesn't create a trapped void of air which may become an outgassing problem. 'Torr-Seal' is commonly used due to its relatively low vapour pressure, and thermally stable to 120°C. (this is a Varian product)
- Vacuum oils are not recommended for general lubrication as they can creep on surfaces spreading contamination and increasing the outgassing surface.
- See Appendix 3 section 1.5.

#### 9. Mounting

- The load of the chamber assembly must rest on a standard accelerator beam line table ANSTO, HVEC, HVEE or NEC or a purpose built stand that minimises vibration.
- Vibration decoupling will be achieved by supporting the chamber separately from the vacuum pump and using an edge welded bellows between the chamber and pump.
- For ANTARES beam line vacuum equipment the beam axis alignment is nominally 1750 mm from floor height.
- For STAR beam line vacuum equipment the beam axis alignment is nominally 1193 mm from floor height.

#### 10. Access

- The working face of the chamber must face an area that is easily and comfortably accessible. The floor space immediately in front of the working face should be raised using steps to facilitate access.
- Heavy or awkward shaped vacuum equipment must be designed to be lifted with the aid of over cranes for example lifting lugs.

#### 11. Testing

- Vacuum equipment must be tested prior to routine use to minimise time spent chasing leaks in completed systems.
- Vacuum vessels must be helium leak tested to ensure all seals are better than  $1 \ge 10^{-10}$  Pa.m<sup>3</sup>/s ( $1 \ge 10^{-9}$  mbar.l/s). As a standard, during a leak test there should be no deviation from this baseline level for a leak-tight system. A record of the test is to be noted on the engineering drawing showing, date, maximum leak rate, leak location/s, leak detector used and operator name.

- For all other vacuum equipment in-service testing may be necessary to allow complete operation of various feedthroughs and positioners.
- Caution must be taken before positive pressure testing vacuum equipment as the equipment may be irreversibly damaged. Warning Do not use water testing on any vacuum equipment.
- A Residual Gas Analyser (RGA) can be used to give more detailed information about the gas composition inside a vacuum volume. An RGA can be used as a substitute for a helium leak detector with the benefit of providing more information about outgassing, or virtual leaks inside the vacuum volume. The pressure of the vacuum system must be  $< 1 \times 10^{-2}$  Pa to prevent damage to the filament inside the instrument. Instruction manuals must be checked before using the RGA head to ensure safe and effective use.

### 12. Operation and Continued Monitoring

- All chambers should have a high vacuum gauge sensing within the chamber space. This includes all adjoining compartments that may be isolated by a valve.
- The chamber must have valves attached that allow complete vacuum isolation from the beam lines and other equipment.
- To protect high vacuum gauges against the effects of operating in low vacuum, high vacuum gauges should be automatically isolated if the vacuum pressure rises above  $1 \times 10^{-2}$  Pa.
- High vacuum gauges must be cleaned at minimum every 3 years or whenever a gauge develops instability as compared with adjacent gauge readings.
- Systems indicating higher than normally observed pressure must be isolated and repaired. A helium leak test is recommended if the leak location is not clear.
- To avoid undesired contamination, vacuum chambers should be vented with dry nitrogen or argon instead of air. This minimises water vapour and oil from depositing on the vessel walls.

# Chapter 2

# Design Criteria, Information and Philosophies for Vacuum Systems used in the Accelerator Area

#### Overview

In an accelerator system ion beams are transported from ion sources through to end stations (or detectors) through vacuum space maintained inside of beam tubes (lines). The relatively low number of gas molecules in the vacuum ensures a higher mean free path for the accelerated ions and hence less loss of ions through collision which is crucial especially for AMS measurements when counting a very small number of ions. A high vacuum also minimises the formation of plasmas in an electric field at high voltages which can avalanche into spark breakdown damaging sensitive equipment.

The vacuum systems used in the accelerator area are designed to be fail-safe to protect people and equipment. A very high proportion of problems with vacuum systems relate to, seals leaking on first use, being incorrectly mounted or failing due to aging 'o' rings. Other problems, however less frequent, include mechanical damage to sealing surfaces or fatigued parts such as bellows. The problems are all characterised by a poor vacuum or a previously steady vacuum that begins degrading slowly as observed during the operation of the vacuum system over its life time. The remaining problems are due to poor welds or vacuum fittings breaking. Poor welds are usually found during the initial helium leak test. They are remedied by either removing and re-welding the section or using a commercial vacuum epoxy. The choice is dependent on whether the weld is on a structural component or just a seal. Broken vacuum fittings are replaced or repaired.

Within the accelerator area. physicists work directly with technicians who apply broad knowledge and experience in vacuum technology to develop their concepts for use in the accelerator environment. This mix has been successful for 50 years. However not all designs are completed in the accelerator area. There are cases where commercial projects call for a more production based design or designs of chambers with complex noncylindrical shapes. These may go to the ANSTO drawing office for completion or a specialist vacuum design company.



Fig 1. ANSTO designed and developed beam lines on the STAR accelerator in building 22

It is not possible and often not necessary to standardise the design requirements for all components used in the accelerator area so the following information is intended to help designers of vacuum equipment to comply with accelerator specific and Australian standards. As of 2011, there are no known commercial workshops in Australia who specialise in the production of vacuum vessels. Local representatives from vacuum suppliers can provide access to overseas specialist workshops.

# 1. Terminology

**ANTARES** – Australian National Tandem for Applied RESearch. The HVEC FN Tandem accelerator in B53.

Backing – To pump on the exhaust (back) of a high vacuum pump

Beam line – The evacuated tubes that carry the ion beams from the ion sources to the end stations

**Cryopump** – Cryogenic vacuum pump. Uses a helium cooling interface to the vacuum space to freeze gases.

**Dependex** – A type of flange which is the standard flange used on ANTARES and ANSTO built beam lines.

**Fore line** – The vacuum line, tube, pipe, whatever between the high vacuum pump and the backing pump

High vacuum – See details in Chapter 3, para 1.3

ISO - International Standards Organisation

KF – Klein (small) Flange

LF – Large Flange

Low Vacuum – See details in Chapter 3, para 1.3

Outgassing - the liberation of gaseous vapours from the surfaces within a vacuum space

Pa – Unit of pressure, Pascal. 1 Pa = 1 N/m<sup>2</sup> = 0.01 mBar = 7.5 mTorr = 1.45 x 10<sup>-4</sup> PSI

**Pump down = Pump out** – evacuating gas from a vacuum space

**Roughing or Roughing-out** – Usually used to describe the initial transfer of gas from atmospheric pressure to low vacuum.

STAR – Small Tandem for Accelerator Research. The HVEE Tandetron accelerator in B22.

Turbo – an abbreviation for turbomolecular vacuum pump

**Ultra-high vacuum –** See details in Chapter 3, para 1.3

Venting – the equalisation of negative pressure to atmospheric pressure in vacuum equipment

Vessel – generic for a vacuum chamber where an instrument is mounted or interfaced into the vacuum space.

## 2. Standard Units

### 2.1 General

Although metric standard is adopted across all ANSTO accelerator facilities it does not mean that all equipment is metric as the US built FN Tandem is mostly imperial, conversely, the Dutch built HVEE Tandetron is all metric. There is a combination of metric and imperial devices used throughout the facilities but designers must ensure that where practicable all new designs are metric.

### 2.2 Threads

New equipment for use in the accelerator area, where practicable will be designed with metric standard threads.

• Contrary to the standard, on ANTARES there is a combination of threads in use. The accelerator high voltage generator and parts that have originated from the United States are

mostly imperial. Typically, UNC and some UNF. Most beam lines and endstations are metric but all Dependex and most Conflat flanges are imperial.

• ON STAR all threads are metric. ANSTO designed and built beam lines are metric except for the Dependex flanges which are UNC.

Care must be taken to identify threads and use the correct screws. Where practicable, metric threads are to be used other than on Dependex flanging.

### 2.3 Pressure

All pressure indicating devices shall be in the SI unit, Pascal. Historically, Torr was used but this was replaced by Pa in the late 1980's. Most international accelerator labs use mbar. The conversion is as simple as multiplying mbar by 100 to get Pa. Pascal refers to a physical force of newtons per metre squared.

# 3. General Design Philosophies

There are many varieties of vacuum chambers and equipment in use in the accelerator area. Most have been designed and made at ANSTO. Examples include vacuum equipment measurement chambers where samples are measured, beam line transport components, ion source components or sensitive detectors, to name a few. Each has its own unique characteristics and vacuum design considerations.

In order to maintain a suitable mean free path for ion beams whether being transmitted from a source or scattered from a sample into a detection system, high to ultra-high vacuums are necessary. In a high vacuum the density of gas is low enough to minimise electron flow from surfaces at high voltages to surfaces at lesser potentials. It is therefore prudent to ensure designs of vacuum equipment that directly couple to the accelerator are designed for a minimum of high vacuum operation.

In every case the designers must ask the following questions:

- What shape? How big? Where will it fit? How will it be mounted? What level of vacuum?
- Will it be ridged enough and have low enough distortion to mount ion optical components?
- Is the design flexible enough to enable small scope changes in the future? Is it designed with consideration for reuse in another application when the project finishes?
- Does the design use vacuum technology best practises?
- Does it meet Australian and local standards where applicable?
- Is it a safe design?
- Is it achievable with resources available?
- Is it cost effective. Can we modify existing equipment to achieve a similar result?
- Does it have low environmental impact?

### 3.1 The Shape

The first consideration is to define the actual use of the vacuum equipment relative to the experiment. Where possible cylindrical shapes should be considered first as they are stronger under vacuum and consequently being round, ports can be welded into the chamber aligned radially to the centre which ideally aligns instruments including detectors and cameras with the intersection point of the ion beam and the sample being measured.

Non-cylindrical chambers must be carefully designed to included adequate strengthen ribs to minimise distortion and hence misalignment of instruments.



Fig 2. A few examples of vacuum vessels used in the accelerator area

## 3.2 The size

The size is dictated by the space needed inside of the vessel or equipment in order to meet the operational demands. In the case of measurement chambers too big may mean problems with the distance between samples and detection systems and too small may mean manual handling of samples may be inhibited. Large chambers may have to be certified as per AS 4343-2005, Hazard Levels of Pressure Equipment in Australian Standard.

### 3.3 Location

It will be an agreement between the accelerator user groups as to where equipment and beam lines will be located. This agreement will endeavour to minimise conflicts in beam line space, usage, accessibility and resourcing.

### 3.4 Mounting

In general equipment must be secured onto the floor unless it is designed as mobile. An accelerator compliant beam line table should be used with standard beam line supports. Vibration must be identified during the design stage and decoupling stages provided.

Insertion bellows must be provided in long sections of beam lines to enable compression of the beam lines to facilitate dismantling and to ensure minimal strain on vacuum joints.

Designs must consider trip, bump and fall hazards. Structures that support platforms or heavy equipment that may otherwise fall must not be reliant on the structure supporting the vacuum chambers and beam lines. For example, ladders, walkways, etc.

- For ANTARES beam line vacuum equipment the beam axis alignment is nominally 1750 mm from floor height.
- For STAR beam line vacuum equipment the beam axis alignment is nominally 1193 mm from floor height.

### 3.5 Flexible design

It is prudent to ensure where practicable designs ensure some redundancy for feedthroughs, access ports and internal space. A major problem with early chambers was the lack of space allowed for inserting samples by hand or manually manipulating internal equipment and wiring. It is easier to design flexibility into the chamber or equipment at the design stage rather than having to dismantle it and have it reworked in the workshops later.

### 3.6 Can it be made?

When designing any equipment seek advice from a qualified machinist. Often designs include machining that may be very expensive or not possible. Early in the design phase ensure that the resources are available to service the vacuum equipment. It is recommended to consider the following early in the design stage:

- Power
- Cooling water
- Air cooling
- Signal lines
- Control lines
- Add-on electronic and mechanical instrumentation

### 3.7 Safe design

There are no cases that the author has been able to find of scientific vacuum chambers or equipment, like those used in accelerator facilities, failing and causing injury. Vacuum pressure is relatively low, -100 kPa, or about half the equivalent magnitude in a car tyre.

For safe designs the structure must meet the minimum dimensions as calculated in Appendix 1 - Structural Calculations for Scientific Vacuum Vessel Design.

Care must be taken with the design of the following:

- Glass view ports (most glass breakages are in the form of a crack which leak to atmospheric pressure quickly without the spread of shards)
- No internal sharp edges; in the case where a person must insert their hands blindly to access components.
- Venting limits pressure relief for positive pressure?
- Evacuation rate regulation; especially in the case where very thin material windows separate different pressure systems within a single vacuum space for example beryllium windows on x-ray detectors.
- Equipment mounts/stands; where an operator must climb up high to operate devices on the system
- If oil type vacuum pumps are used then mist filters must be fitted to the exhausts.

## **3.8** Cost effective design

All *bells and whistles* may not be necessary even after considering some design redundancy for future changes. Where possible, designs should consider integrating components that are available off the shelf. Designing what has already been designed by someone else is wasting time and effort.

It may be cheaper to modify an existing chamber for use rather than start from the beginning. Many chambers used around the accelerator area have successfully had one or more lives.

### **3.9** Design by numbers

This is a matter of ensuring structural integrity and being able to prove it. Likewise, designing the vacuum pumping system requires calculating the most appropriate size pump rather than going for the "biggest"!

There are formulas in Chapter 3 that allow must crucial calculations to be made from wall thicknesses to conductance. Good enough is not acceptable. Replication of previously designed equipment is acceptable.

### **3.10** Australian Standards

Compliance is mandatory however there are few standards that are relevant to scientific vacuum vessels and equipment. The industry has developed its own standards for flange and seal designs which can be seen in the catalogues of most leading manufacturers of vacuum equipment. It is the responsibility of the designer to ensure compliance with all relevant Australian Standards. Chapter 1 discusses what standards are included.

### 3.11 Environmental impact

Most vacuum systems used in the accelerator area today are designed to be oil free. That is, they do not use oils for lubrication of vacuum seals. There is however some oil vane and diffusion pumps still in use but the oils are responsibly disposed of through waste management at ANSTO. Some old diffusion pumps had been used with mercury as the pumping agent. Personnel must be cautious of using old diffusion pumps and their associated backing pumps as there may be residual mercury on surfaces and in the backing pump oil.

Solvents used for cleaning are used in small amounts and usually for wiping rather than immersing. Where baths are needed the solvents are reused when possible. The solvent bath used for degreasing, self recycles the solvent by design however there is a small loss of solvent over time into the atmosphere.

Where possible solvents used are selected with the environmental impact in mind.

### 3.12 Manufacture

If ANSTO cannot provide the level of manufacture quality in-house then scientific vacuum equipment is best manufactured by companies that specialise in high tolerance machining and welding. A general metal fabricator will not suffice.

Attention must be paid to considering standard sized tooling during manufacture. Some companies will charge for the purchase of tooling that they consider to be non-standard.

It is imperative that the drawings for manufacture state all tolerances and qualities for the finished product.

On all design drawings, where the angle of the welded ports is crucial, ensure angular tolerances are clear and achievable. If necessary, specify the use of mandrels to hold ports in place during welding.

Some success has been made outsourcing the manufacture through vacuum companies to overseas manufacturers who specialise in high quality small runs.

### 3.13 Testing

Testing the vacuum equipment or chamber is essential to establish compliance with the design and the all important ultimate base vacuum. Helium leak testing is the standard method used for chambers and most equipment however on some equipment in-service testing may be the only option. This is where add on vacuum equipment doesn't change the original base vacuum of the system it is connected to.

### 3.14 Inspection

All parts should be cleaned prior to inspection. This doesn't need to be the final clean but enough to ensure all welds and surfaces can be clearly inspected. Welds must be checked for pits and alignment along the weld lines and seals must be checked for surface finishes. All crucial dimensional must be checked. It is recommended that any non-complying findings are photographed and documented in the project file. All non-complying parts should be reworked rather than *patched up*.

#### 3.15 Certification

As most vacuum vessels and equipment in the accelerator area fall within Hazard Level E of AS 4343-2005, certification is a local process. For large measurement vacuum vessels, results from the inspection and leak testing will be recorded on the engineering drawing and registered in the local accelerator register and/or ANSTO drawing register as appropriate.

#### 3.16 Documentation

For all large measurement chambers requiring any level of design calculations, design documents must be kept with project files.

#### 3.17 Maintaining

Vacuum chambers and beam lines do not require high levels of maintenance other than a general periodic clean and occasional seal replacement. Vacuum fittings and devices will have their own level of maintenance so access to these components must not be difficult.

The design must be easy to maintain especially moving parts, sliding or rotating seal and other parts in the vacuum space that may wear. Also vacuum pumping systems that may be mounted under or near the vacuum chambers. Generally any part that will require any level of routine maintenance must be accessible without the need for a major disassembly.

Vacuum pumping systems other than oil free systems, connected onto beam lines and chambers must include suitable oil traps between the backing and high vacuum pumps and where mist filters on all exhausts.

Some high vacuum gauges will require periodic cleaning. On ANTARES high vacuum cold cathode gauges are scheduled for cleaning every 12-18 months or max 3 years for difficult to service or less crucial instrument locations. Otherwise gauges will be cleaned on demand within the periods.

## 4. Performance and Operations

#### 4.1 Ultimate Vacuum

The ultimate vacuum is the maximum vacuum or lowest pressure the vacuum equipment typically reached after 3-5 days of uninterrupted pumping. This is a benchmark value which is used to assess the long term quality of the vacuum system. It is sometimes referred to as the *base vacuum or static vacuum*.

Generally, vacuums in the beam lines and chambers are well within the high vacuum range. Vacuums lower than this range will indicate a higher than normal gas load, a failing seal or a pump malfunction. On the accelerators the two main types of high vacuum pumps are turbomolecular and cryogenic pumps. For a system designed for high vacuum or better with no leaks and low outgassing then vacuums better than  $4 \times 10^{-6}$  Pa should be readily achieved.

The ultimate vacuum is improved by the correct choice of materials, seal types, high vacuum pumps used cleanliness and correct operation. All fittings must be designed or selected deliberately for high to ultrahigh vacuum use. Surface preparation and cleanliness will contribute to improved vacuums. A finger print can outgas for long periods of time. A bigger (higher speed, higher ultimate vacuum) vacuum pump does not mean a better vacuum!

### 4.2 Working Vacuum

The working vacuum may be lower than the ultimate vacuum due to sample cycling (changing), introduced gas loads from adjoining systems or outgassing samples. A system must be designed to ensure the high vacuum pumping system has the optimum pumping speed and ultimate vacuum. Once a vacuum system reaches the "working vacuum" little operator intervention is necessary to maintain it but there are some important points regarding ongoing monitoring to ensure a failure is not immanent.

For sections of beam lines that are holding a static vacuum a cursory glance at the local vacuum gauge will indicate the stability of the vacuum. Generally, the (vacuum) pressure will change slightly throughout a day for example due to diurnal changes in ambient temperature and perceived changes due to ionisation gauges "wandering" as they become dirty.

In sections of beam lines that are in the vicinity of operating beam lines or near end stations the vacuum will be continually changing. Factors that influence these changes include increased gas loads from samples outgassing or surfaces that have just be exposed to high humidity, outgassing due to temperature changes within the vacuum space (including bake outs), outgassing from ion beams striking plastics, etc. Gas molecules don't have a preference to whether they travel towards or away from a pump during molecular flow so the source of gas can come from just about anywhere in the open system. These factors must be considered when monitoring vacuums throughout an entire system.

If a vacuum system's pressure rises slowly over many days or weeks if it is not related to the cryopump loading then it may be a seal failing. In this case a helium leak test is recommended.

A fundamental flaw in many vacuum system designs is the neglect for monitoring the backing pressure between the backing pumps and turbo pumps. Pirani gauges which are best suited for this role are relatively cheap and are the best diagnostic tool for determining overall vacuum system performance. The backing pressure should be consistent with changes in the high vacuum pressure. By monitoring the backing pressure over time, the base vacuum will be realised relative to the high vacuum so a pending failure of the backing pump can be determined, hopefully, well before it happens.

Care must be taken to maintain systems that have the potential to degrade over time for example an oil vane roughing pump will back stream oil mist that may accumulate in the high vacuum side of the system over time. This will contribute to lower vacuums due to higher gas loads from the partial pressures of contaminants.

### 4.3 Sustainable Operation

Now that the vacuum system is designed, manufactured and commissioned for routine service, operating procedures have to be defined and adhered to, to maintain reliable trouble-free service. There are 3 processes that summarise the continuous cycle of operating a vacuum system:

- Pumping down from atmospheric pressure
- Using the working vacuum
- Venting to atmospheric pressure

The accelerator beam lines are divided into stand alone vacuum systems but in essence function as one. Each system can impact on a neighbouring system if things go wrong. In the case of end stations which cycle up and down in pressure more than any other devices an operator must be 100% sure of the pump down sequence in order to avoid damaging, pumps, samples, high voltage devices, detectors, etc. It is possible to shut down the accelerators as well. Most sections of the accelerator vacuum systems are interlocked to isolate a pressure surge to small area. Beam line vacuum systems are design so that both the vacuum pump and the beam line are isolated via gate valves. This is a typical arrangement for endstations as well.

The following must be read remembering that all 3 processes work in a cyclic fashion in a continuous loop.

### 4.4 **Operational Protocols**

Key vacuum systems operate continuously, 24 hours per day, seven days per week. There are several vacuum systems that are cycled on and off as usage demands. All vacuum systems have one or more high vacuum gauges, some of which are interlocked to make the systems fail-safe. Any slow leaks or major catastrophic failures will close valves and isolate the system. In the few cases where no interlocks are used, a catastrophic failure will result in the high vacuum pump shutting down.

- For a <u>turbo pump</u>, as the pressure rises the gas load on the pump will cause the pump to draw more current to maintain it's speed. It will eventually fail when the current trip level is reached in the controller.
- For a <u>cryopump</u>, as the pressure rises the inflowing gas will condense or freeze onto the in the pump until it begins to warm up. Eventually there will be a thermal trip activated o the compressor which will shut is down. In both cases it is fail-safe.

As part of the accelerator's maintenance system, daily monitoring of all vacuum pressures is carried out. Where a pressure is seen to rise by a few percent without a particular reason, further monitoring will occur and corrective action started. Instantaneous failures are dealt with immediately. All observations and whether any maintenance carried out is noted on the maintenance log.

Where a vacuum system is isolated for service or where it has failed the appropriate tags are placed on the power leads.

Unattended out of hour's operation notification is not normally used as vacuum systems run continuously.

#### 4.5 Pump down

#### General

Pump down time is when most mistakes are made and significant time lost. Before a pump down begins the operator must ensure that all seals that were disturbed had been cleaned and replaced if damaged. The system must then be sealed ready for pump down and the roughing-out valve on the rough-out port, closed.

If a dry pump is to be used, such as a scroll pump, for rough-out then the tube is connected to the pumping port. If an oil vane rotary pump is used then ensure the cold trap has been filled beforehand.

#### Rough-out

Start the roughing pump and allow it to reach base vacuum. This is your first indicator of whether you have a leak in the system being pumped and when you have reached the pumping limit of the

pump. Slowly open the pump out valve on the system. This is where mistakes are made and damaged caused. Relatively speaking the volume in most sections of beam lines and endstations is small and the bulk of the gas can be removed very quickly. The problem is that a reduction in pressure too quickly can cause mechanical shock to fragile parts that must adjust slowly in order to minimise stress such as thin windows on detectors and detectors or devices that contain carbon foils.

Operators must always open roughing out valves slowly then gradually increasing as the gas load reduces. The change in the sound of the loaded pump is a very good indicator. A good design for more sensitive pump outs is to use a metering valve in line with the rough-out port to act as a regulating orifice.

#### Cryogenic Vacuum Pumps

For systems using cryopumps as the primary high vacuum pump it is essential to bring the base pressure down to the limit of the roughing pump before changing over pumping to the high vacuum pump. This reduces the gas load to be trapped (captured) on the cryo pump and extends the service life of the cryopump. Cryopumps are widely used on ANTARES as the primary high vacuum pumps but their limitation is that the warm up as they trap more gas causing a rise in base pressure. That is, when a cryopump is reaching saturation the temperature on the surface increases. More energy is required to hold the molecules on the surface and since it is almost fixed by design the temperature increases. A cryopump's effective pumping speed depends on the freezing and boiling points of the gases being pump relative to the cryopump's temperature. As the temperature increases more molecules leave the surface (boil away) raising the pressure. Some molecules are re-trapped as they hit the surface of the arrays and loose kinetic energy but they have a short residency time and soon add to the rising pressure in the system. Eventually the base pressure in the system becomes unusable so the pump must be regenerated.

#### Turbomolecular Vacuum Pumps

The other type of high vacuum pump widely used in the accelerator area is a turbo pump. Unlike a cryo pump that traps or captures gas molecules a turbo pump transfers the gas by conducting it almost one-way out of the vacuum space. The lowest roughing pressure before changing to a high vacuum pump is not as crucial when a turbo pump is being used. In saying this, the bulk of the gas must be removed before changing from roughing out to high vacuum pumping otherwise the vanes on the turbo pump may be overly stressed due to the pressure surge. A typical changeover pressure range is 5 - 50 Pa.

The optimum changeover time to minimise pump down times can be estimated with the assistance of the formulas found within the document. Letting the roughing pressure reduce to the limit of the roughing pump before changing over to the turbo pump is an inefficient method of system pumping. A turbo pump is more efficient at removing gas molecules as the flow moves towards molecular flow. Changing pumping to a turbo pump just before molecular flow is reached will increase the pumping speed and base vacuum will be reached sooner.

### Pumping Speed

Each type of gas will pump away at a different speed depending on the mean velocity of the molecule which is a function of its mass. A hydrogen molecule of 0.02 kg/mol and mean velocity of 1762 m/s will reach a pump much quicker than say a Xenon atom of 0.13 kg/mol with a mean velocity of 217 m/s.

### **Limitations**

In rare circumstances roughing pumps may be left pumping high vacuum systems by mistake. It is unlikely to damage either pump but the ultimate vacuum will not be reached as the roughing pumps are ineffective at conducting away molecules in molecular flow. Water vapour and residual solvents are difficult to remove from a vacuum space with *normal* vacuum pumping. Most low vacuum backing pumps provide gas ballast which can speed up the removal of water vapour and solvents that may otherwise condense inside of the pump during the compression stage. By introducing a small amount of air near the outlet of the compression stage the small increase in pressure helps carry water vapour and solvents to the exhaust before they can condense.

Some vacuum systems have been designed to allow chambers and beam lines to be heated. Heating causes contaminants to be removed from surfaces at lower than normal vacuums. The higher the temperature the faster the removal will be. Care must be taken to ensure heating doesn't inadvertently cause plastics and elastomers used within the vacuum space to outgas. Most bakeable systems use ceramics and metals only.

It is important that where vacuum systems are permanently connected to high voltage devices for roughing out, the insulated (plastic) pump out lines between the roughing pump and high voltage device must be brought up to atmospheric pressure before high voltages are reapplied. Otherwise the low molecular density of gas in the tube may ionise causing burning of the tube or even spark damage that can puncture the tube causing a leak.

## 4.6 Venting

### General

Venting can cause damage to vacuum components just like pumping out a system from atmospheric pressure too fast.

#### Venting Speed

As discussed earlier the volume of vacuum systems on the accelerators is measured in litres to a several tens of litres. A catastrophic loss of vacuum will be more of a brief audible event rather than a dangerous mechanical failure. Little energy is required to change these systems by 100 kPa. There are a few devices in the vacuum space that may be damaged due to pumping out too fast but none that are considered a danger to personnel.

Other than venting too fast the second point to consider is the introduction of water vapour if venting with air. Key vacuum systems on the accelerators are vented with either dry nitrogen or argon both of which are delivered through a closed reticulation system from gas bottles. The choice of gas is dependent on molecular contamination within the system for example nitrogen is not preferred to vent the 846 ion source as nitrogen is an element that is known to bond with other elements causing molecular ion beams.

#### Regulation of Flow

There are many low flow regulators and rotameters (tubular flow meters) available that provide adequate control over venting. A simple in-house gas regulating system which works well is a tee piece connected between the venting port and the venting gas supply line. The centre port of the tee piece faces upwards and a ping pong ball sits over the hole with a cage over it so that it cant be blown away. Under the ball is an 'o' ring seal. Before the venting valve is opened the venting gas is allowed to flow. The pressure of the gas lifts the ping pong ball of its seal. The gas flow is adjusted depending on the height of the ball above the seal. The ball can only travel a centimetre or so. Once the flow is set the venting valve is opened and the now regulated gas flows into the vacuum space and the balls drops onto the seal stopping air from entering the vacuum space. As the pressure reaches equilibrium the ball begins to rise off the seal until eventually it rises to the original set height indicating the vacuum space is now at atmospheric pressure.

#### Precautions

- Before venting a vacuum system it is wise to isolate all ports and beam lines that must remain under vacuum. To save stressing a beryllium window on an x-ray detector due to the changes in pressure the system should be designed so that the x-ray detector can be isolated with a gate valve. The same logic can be applied to all equipment that may be effected. It is also important to ensure all power supplies and high voltages that may be hazardous to personnel or equipment are isolated prior to venting. This can be done either by administrative protocols or using a vacuum switch such as programmable relays in a vacuum gauge controller.
- Some vacuum gauge controllers use the signal from a Pirani gauge measuring at the same location as a high vacuum gauge to switch off the high vacuum gauge when the pressure becomes too high. This saves the gauge from ionising air when the system is at atmospheric pressure which will significantly reduce its life time.
- Another problem with venting too quickly is the disturbance of dust. Accelerator tubes must be vented slowly to minimise equalisation stresses and also the disturbance of dust. In this case a metering valve is used to limit the vent flow rate.
- Venting can cause loud high frequency noise so care must be taken to dampen the noise to a comfortable level by controlling the air admittance rate, or use hearing protection.
- Most beam line vacuum pumping systems are interlocked to isolate either the beam line or pump or both. In the event where an accidental venting occurs the gate valves will close minimising the effect on surrounding systems.

# 5. Safety

### 5.1 General

Catastrophic failure causing injuries are not common and no reference is known from overseas accelerator facilities. This is almost certainly due to the low pressures involved in vacuums and the size and nature of the scientific apparatus. There are of course many failures reported for industrial sized tanks and equipment.

The vacuum systems are essentially fail-safe from absolute vacuum to atmospheric pressure. The worst case has been a number of beam lines and measurement chambers developing leaks and thin windows that have ruptured. In each case there has been a gradual rise in pressure to atmospheric pressure.

However, a few rules do apply.

- All vacuum systems must have one or more vacuum gauges attached with the exception to some static chambers used for storing moisture sensitive items or radiation detectors.
- High vacuum gauges must be switched off before vacuum systems are vented to atmospheric pressure.
- Only nitrogen, argon or other inert gas to be used for venting. No flammable gases to be used such as hydrogen.
- Turbomolecular pumps must not be vented from full speed. See manufacturer's recommendations.
- All designs must be consistent or better than the best practises outlined in this document. Inferior equipment must be isolated and reported to the Leader of Accelerator Operations.

- Cryopumps must not be operated without a serviceable pop off valve.
- Cold traps must be warmed and blown dry at least every 6 months to minimise  $O_2$  concentrating (and for de-icing as necessary).

## 5.2 Interlocks

Nearly all vacuum systems on the accelerators are interlocked via a set of integrated relays in a vacuum gauge controller, to protect equipment. Generally, if a rise in pressure is detected above a particular set level then the interlock relays will activate to close corresponding gate and line valves. This is designed to minimise the number of vacuums that must be regenerated in a fault. The interlock system also protects accelerator tubes from exposure to moisture and in the event of a catastrophic failure of the accelerator tubes; the vacuum system will close the main accelerator gate valves to trap SF6 gas.

If an interlocked failed causing a gate valve to stay open in the event of a pressure rise then the next interlock should activate the next gate valve. If the next interlock fails it is possible that the two interlocks share the same vacuum gauge controller which is at fault. In that case the next interlock should activate. If no interlocks activated there would be no increased risk of injury to personnel but all vacuum systems will require regeneration or a restart after the interlocks were repaired and tested.

### 5.3 Fumes from exhausts

There are a number of oil vane pumps in use on the accelerators. In the past, the exhaust on the pumps has released oil mist into the atmosphere inside the buildings. Now, on nearly all oil vane pumps in continual service there are mist filters attached or the exhaust is plumbed into an extraction system that vents the exhaust fumes outside of the building.

### 5.4 Earthing

Some vacuum gauge controllers are prone to high voltage transient surge damage. These sometimes cause unnecessary isolation of systems requiring resetting. It is very important that all vacuum gauge controllers are well earthed.

## 6. Serviceability

### 6.1 Access

Most beam lines with the exception to some measurement chambers are static volumes of vacuum requiring little operator intervention other than during preventative maintenance. Vacuum pumping systems do require a higher level of intervention during maintenance and operation. With these points in mind the designer must ensure that all controls and monitoring devices are localised at the working face of the beam line or chamber.

Due to beam lines being so high above floor level, working on them usually requires some form of ladder or steps. When designing beam lines, be sure to have pump out ports facing the passageway beside the beam line. The same applies to vacuum chambers and other large volume vessels.

Measurement chamber will need to have the access port accessible from a location near the control side of the chamber. A platform is recommended to allow easy reach and eay viewing into the ports.

### 6.2 Maintenance

A well designed vacuum system will require routine maintenance including:

- Changing faulty seals cleaning seals that are regularly opened
- Cleaning vacuum gauges especially cold cathode type high vacuum gauges
- General cleaning inside of measurement chambers and equipment that is; regularly opened and touched, whenever it looks dirty, when the base vacuum degrades or when the surfaces are exposed to the atmosphere for long periods at a time (weeks).
- Servicing of vacuum pumps the group of the most routine maintenance tasks includes; cryopump regeneration, turbo pump bearings, scroll pump tip seals and oil vane pump oil changes to name a few
- Actuator service sliding shafts that penetrate into the vacuum space

### 6.3 Cleaning

Cleaning is by far the most essential part of general maintenance of a vacuum system. Poor cleanliness will lead to poor vacuums and possibly sample contamination. It is a general philosophy that cleaning is done with solvents, wiped over and/or scrubbed and thoroughly dried.

### **Solvents**

Typically ethanol, methanol and acetone are used. These solvents remove most greases and oils that are encountered. What ever solvent is chosen the most crucial point to remember is that it must not leave any residue.

For surface finishing see section on 'Surface Finishes'.

Chemical polishing is not commonly used in the accelerator area due to the size of the baths that are needed for large parts, the problem of cleaning or neutralising residual chemicals and not having a suitable facility for handling the types of chemicals used. Although once used electro-polishing is no longer encourages for the same reasons. If suitable facilities were available both chemical and electro-polishing are real options for very good cleans.

A general clean will consist of a number of different levels of cleaning. A single clean with the same solvent will not remove some oils and greases therefore a progressive approached is used using a variety of solvents and wiping materials. The following is a guide only of some such techniques.

Using a dry gas or filtered compressed air, most loose dust can be blown away. This is sometimes the best starting point for cleaning. A final blow down is also useful to ensure complete removal of lint.

The first clean will take away coarse particulates or chemicals. Ethanol dampened tissues if good for this level of cleaning. The ethanol helps remove some oils and greases and mixes with water to reduce the evaporation time. It is also good for mopping up dirt and dust. Scour pads (or 1200 wet or dry paper) moistened with ethanol is good for removing stubborn dirt. This must be continued until better than 95% of dirt, dust, oil and grease is removed. For stubborn dirt it may be necessary to use a petroleum based solvent followed by an ethanol rinse and wipe. Wiping is key to cleaning. The light mechanical brushing dislodges most dirt.

The second clean starts with ethanol but finishes with acetone. Only lint free wipes are used such as Kimwipes. At the end of this level of cleaning the equipment must look ready for use in the vacuum. That doesn't mean it is. It will more than likely have residues remaining on the surface.

Final clean. It is recommended that methanol is used for the final clean or another low residue solvent. The final clean must use lint free wipes dampened with methanol and lightly rubbed over the entire surface. An alternative final clean is to use the solvent bath and suspend the equipment in the hot vapour for a few minutes. Care must be taken with the bath as the hot vapour will degrease skin very quickly.

If a shiny (healthy looking) clean is desired then a metal polish such as Brasso can be used. Whenever Brasso is used the residue must be cleaned off with ethanol. Care must be taken not to get metal polish pastes into gaps that cannot be thoroughly cleaned out. Brasso is an enemy of vacuum, it traps water.

### 6.4 leak detection

In the accelerator area, a leak is characterised by a flow of gas molecules into a space that is below atmospheric pressure. The pressure of the gas entering the space will restrict the vacuum system from reaching its design pressure which is typically in the high vacuum range. As a benchmark, if a vacuum is  $1 \times 10^{-5}$  Pa or more then disregarding the effects from outgassing, there may be a leak. In system designed for ultrahigh vacuum the benchmark will be in the order of  $1 \times 10^{-7}$  Pa.

Leak detection is usually only necessary for new chambers or equipment. It can be done offline or after the part has been mounted into a beam line. The most common method of leak detection is a helium leak test. Helium atoms being so small will find their way through openings that are too small for most other molecules. Generally, the smaller the leak, the less helium that can make its way along a leak path. The leak detector is usually a mass spectrometer tuned to have highest sensitivity for helium. The detector is connected directly to the device being tested. Any helium that makes its way through the leak will eventually be measured in the mass spectrometer. Residual Gas Analysers (RGA), although not specifically leak detectors, can be used for just that. Working on the same principle, mass spectrometry, the RGA can detect helium as well as other gases.

In some cases leaks can be too large for a helium leak test, that is, the amount of gas in the device being tested is too high diluting the helium to an ineffective percentage. Also, if the pressure is too high the leak detector cannot be opened to the device as the pressure gradient will be too high. Leak detectors work best from the higher end of the low vacuum range.

Often there are multiple leaks in systems. It may mean repairing large leaks in order to find small leaks. Where a leak is too large for the helium leak detector a few other methods can be used to locate leaks.

- Solvents have a very low viscosity and vaporise very quickly in air. Wetting surfaces with small amounts of acetone or ethanol can show small changes on Pirani and Penning gauges.
- Isolation of seals can, by process of elimination, restrict the testing area allowing large leaks to be isolated. For example, if a chamber with many ports and devices attached has a leak. It may be prudent to close all valves leading away from the chamber except for the leak detector valve then sequentially blank off the ports one at a time (using a sound flange and new seal) and test for a leak.

## 7. Soldering wires vs mechanical joints

It is preferable that wires in a vacuum space are joined together with a mechanical clamping type joint. The clamp must allow for gas to escape. Where a direct joint is necessary then soldering is permitted. Standard lead/zinc with resin core is okay provided that ALL flux is removed with ethanol. This may require some soaking in solvent with gentle agitation. Solder without resin core

can be used on stainless steel with a zinc chloride based eutectic flux, used sparingly. Ensure all flux is removed especially between the wire strands.

Be aware that in Faraday cups the continual flexing of wires can work harden the wire causing it to break. These are typically soldered joints. If soldering, be sure not to melt the insulation. It is highly recommended to replace normal plastic insulation with loose fitting PTFE (Teflon) spaghetti.

# 8. Flanges and seals

### 8.1 Common vacuum seals used in the accelerator area

A variety of standard vacuum seals are used on all vessels depending on the degree of vacuum to be achieved and the type of fitting that will be used. The types are widely used in the vacuum industry. ANTARES uses the Dependex style seals and flanges. This was developed for use with HVEC accelerators over 50 years ago. Other types in use include Conflat, ISO, and KF are used as well.

Туре	Vacuum level	Flange type	Seal type
KF	Low to High	Clamp (typically)	Viton 'o' ring, Al, PTFE
Edwards	Low	Threaded retaining nut	Viton 'o' ring
Dependex	High	Ring retained by spring clip	Viton 'o' ring supported by a ring
LF or ISO	High	Ring or clamps	Viton 'o' ring supported by a ring or in a groove
Conflat	Ultra high	Ring fixed or rotating	Copper ring, Square section 'o' ring
Specials	Various	Various	Aluminium or indium wire

Table 1. Common types of vacuum flanges and seals. Each type is available in multiple sizes

## 8.2 KF – Klein Flange

A common flange and seal used for general low vacuum applications due its ease of assembly and vacuum performance. It is common to have KF flanges and seals within the high vacuum side of a system. Vacuum performance can be enhanced by the use of aluminium or PTFE seals which directly replace the Viton 'o' ring.

Many general and specialised vacuum equipment is fitted with KF fittings as the range of adaptors from KF to other major types is readily available.





Fig 3. Assembly drawing for a typical KF flanges seal and a photo of the flange, seal and clamp

### 8.3 Edwards

This is no longer a preferred type that is used in the accelerator area however some equipment still uses this type.

One end of the fitting is shaped to retain an 'o' ring while the other end has an angled sealing surface that mates and crushes the 'o' ring to make a seal. They are connected by a threaded nut which when tightened squeezes the two halves together. It is normally for low vacuum applications.

### 8.4 Dependex

This was designed for use on HVEC Van der Graaff accelerators and beam lines. It was introduced to ANSTO in the early 1960's when the 3MV Van de Graaff operated in B22. Here, 1", 2" and 4" Dependex was used. When ANSTO purchased the FN Tandem nearly all fittings used were 4" Dependex (100 mm nominal). It is suitable for high vacuum use and often mated with Conflat flanges in systems that operate near ultra high vacuum.

The Dependex seal consists of a supported 'o' ring sealing between two opposing  $5^{\circ}$  metal faces. The 'o' ring is supported around its inner diameter by a ring to ensure the 'o' ring aligns with the centre of the 2 faces. The inner ring also sets the maximum crush on the 'o' ring that is the ring fits into a step on both halves of the Dependex seal keeping them separated at a fixed distance. Another ring which fits on the outer diameter of the 'o' ring does little more than keep the 'o' ring clean. The rings are often called *egg rings* due to their resemblance.

The Dependex flanges are held in place on a tube, or other fitting that is designed to accept Dependex, by a large circlip that fits into an outer groove a few mm from the end of the sealing face.

The flanges are normally drilled such that the holes alternate between being tapped or clearance. 1" and 2" Dependex use <sup>1</sup>/<sub>4</sub>" UNC, cap (socket or Allen) head screws and 4" (100 mm) Dependex use 5/16" UNC, cap (socket or Allen) head screws. Only use stainless steel screws (of one length) and washers. Discard plated steel screws as they are identified during maintenance. Washers are recommended as most flanges are aluminium alloy.

To complete the Dependex assembly, the flanges must be placed over the tube ends and then retained in place with a circlip. Secondly, the 'o' ring is stretched onto an inner ring and then an outer ring is placed around the assembly. The two Dependex ends are brought close together then the seal assembly is placed between them. The inner ring is aligned with the step on one side then the other tube is aligned and than pushed onto the ring. At this point it is important to ensure the 'o' ring assembly stays in place until the bolts are tightened. Screws should be finger-tightened and the flange faces must be parallel. Tighten screws in an opposite pattern. Some old flanges will have worn threads so screws may only be turned with the aid of an Allen key.

#### Problems

There are a number of things that can go wrong with assembling Dependex fittings.

- There have been cases where inner egg rings have been made too wide so that when fitted between the tubes the 'o' ring is prevented from crushing adequately to form a vacuum seal.
- Another problem with the inner rings is where some Dependex seals have been designed with too shallow steps so that a standard inner ring becomes too wide. In this case special inner rings have been made which are narrower. Personnel servicing non-standard joints must take care to reuse the right sized inner ring. A label should be placed on all non-standard joints identifying them.

- Some steps for inner rings have also been found to be out of tolerance preventing standard size inner rings from locating in the step. In this case some operators have cut a small section from the inner rings so that they can be squeezed together. Cutting inner rings like this is only recommended to solve this type of problem.
- Some flanges have been design for special applications with slightly different PCD's. These have unfortunately bee mixed up with standard stocks. As personnel who identify them (sometimes after a frustrating encounter) should put them aside and label them accordingly.
- Old flanges that have been over-tightened may have stretched threads. They are identifiable by the difficulty in starting screws. Either re-tap the threads or discard the flange. There have been case were tight threads have mislead personnel into thinking they have tightened the Dependex joint only to find leaks.
- Circlips can stretch especially those in over or under sized slots. They are easily identified by there distorted shape. Only use circlips that are flat when not under tension. A discerning technician should discard rusty circlips that cannot be successfully cleaned.
- Care must be taken to make sure the two faces of the Dependex joint are parallel before the joint is closed and bolted together. Failing to do this may cause a leak in the joint. It is acceptable if a bellows is used between sections that have misalignments.
- Twisted 'o' rings can cause leaks although small. There have been cases where a twist has caused the 'o' ring to bulge towards the outer ring making a less than adequate seal.

## 8.5 Special Flanges

These consist of flanges that have been designed to fit application where "off-the-shelf" flanges and seals cannot be used. They are typically based on common designs but modified to suit the application.

### Example

The Alphatross ion source uses two stainless steel flanges to hold the heater reservoir in place. Here the vacuum sealing surface is a flat surface about 8mm wide on each flange. The fixed flange is threaded in an imperial thread and the reservoir flange is open hole. The gasket used is aluminium wire which has been fused together to make a ring. The joint is similar in size to the wire. The wire is supported in place using about 3 to 4 strips of aluminium foil looped around the wire and sticky taped to the edges of the flange. The flange is tightened in a circular pattern to ensure maximum crush on the wire.

## Problems

- The wire is essentially unsupported so vacuum must not be applied until the wire is completely crushed.
- Bulbous joints in the aluminium wire dent the sealing faces on the flanges as they are tightened so they must not be used.

## 8.6 ISO or LF (Large Flange)

This is a European range of fittings designed for high vacuums and to suit most tube sizes used in the vacuum industry. There are two types of design which meet most light and heavy duty applications.

ISO-K flanges are a robust joint which are suitable for supporting larger and heavier items such as vacuum pumps and large gate valves. They are characterised by the outer clamping system which holds the two halves of the joint together.

ISO-F flanges are more of a traditional bolted flange type joint like Dependex. ISO-F and ISO-K can be joined together using a half clamp which fits onto a standard ISO-F flange. The half clamps then attach to the ISO-K flange.

Available in sizes from 63 mm nominal bore to 500 mm NB.



Fig 4. ISO family of Flanges

### 8.7 Conflat CF

Conflat is designed as an all metal vacuum seal for ultra high vacuum applications. A copper gasket is sandwiched between two thick stainless steel flanges that have a knife edge machined into the sealing face. As the flanges are tightened together the knife edges "bite" into the copper to produce an all metal seal with very low outgassing.

The term "ConFlat" is a registered trademark of Varian, Inc., so "CF" is commonly used by other flange manufacturers. Conflat sealed systems can reach vacuums as high as  $1 \times 10^{-11}$  Pa.

The copper gasket locates into a partially recessed in a groove in each flange to provide alignment prior to tightening. It also stops the gasket from moving during bake out. Conflat can be baked up to  $450^{\circ}$ C.

Most Conflat flanges have imperial sized bolts. The original style of bolt had a multi-face head that a ring spanner would fit over. This is to allow more purchase on the head from many angles. It is not uncommon now to find hexagon and socket head screws in use. Be aware that the steel must be a high tensile grade to ensure optimum force can be used to crush the gasket. It is essential that the flange faces are parallel during and on final tightening. A torque wrench may be used to facilitate uniform tightening. It is highly recommended that a molybdenum grease or graphite loaded grease is used on the threads.

Conflat flanges are available in sizes from 10 mm nominal bore to 250 mm NB. Sometimes nominal bore is written DN in front of the size, eg DN63.

Square section Viton 'o' rings may be used with Conflat flanges however this compromises the ultra high vacuum design and ultra high vacuums may not be reached.

### Problems

- Copper gasket not aligned in groove and edge is partially clamped on the bolt hole face of the flange. This lead to partial seal and most likely, a leak. Most Conflat flanges have two small grooves a little more than a millimetre wide machined into the bolt face. These are for locating special clips that hold the gasket in place while the flanges are brought into place. Just prior to tightening they clips are removed. They are sometimes mistakenly called pressure relieving grooves.
- Copper gaskets must only be used once. Unless the copper hasn't been fully compressed after the first use there will not be enough material for the knife edge to reform a seal.
- Due to the forces required for a knife edge to indent a copper gasket the flanges are made of stainless steel and quite thick. Large flanges are quite heavy and care must be taken not to drop them.
- Where Conflat flange bolts have been used more than once the threads in the flange and screws may have stretched. In this case, it is recommended that the same screws are reused in the stretched threads (with a little molybdenum grease) as they will have the same profile. Sometimes using new screws in a stretched thread will cause binding. Re-tap the flange if necessary.
- Stretched threads are possible where nuts and bolts are used through open holes so ensure the nuts go onto the bolt the same way they were removed. Alternatively replace the nuts and bolts outright.
- For ultra high vacuum ensure the gaskets are handled with gloves. Copper reacts well with the sweat in a fingerprint leading to a long outgassing process.

# 9. Bellows

Bellows consist of two styles, edge welded for ease of extension or compression and corrugated style which is has minimal movement. Each style is made from a thin stainless steel that has been electron beam welded. The ends are usually finished in a standard flange to suit the application.

### 9.1 Corrugated style

These are typically used for two purposes. Firstly, to allow sections of beam lines or equipment to be compressed to allow easy removal of sections without dragging the sealing surfaces against each other. Secondly, as bellows can distort and still maintain their structural integrity they are ideal to

join sections of beam line or equipment that have a deliberate mis-alignment. They can be purchased with almost any style of flange including specials. See example pictures.



Large bellows. ~300mm diameter. Stainless steel, wall thickness ~ 0.8 mm. This bellows is used to correct alignment offsets in the magnet box

Medium bellows. ~ 100 mm diameter. Stainless steel. wall thickness <0.5 mm. Used to allow sections of the beam line to be compressed to facilitate beam line component removal.



Small bellows. Electron beam edge welded stainless steel. Material thickness <0.25 mm. Used to isolate vibrations in the beam line.

Fig 5. Examples of bellows

#### 9.2 **Edge Welded Bellows**

These bellows are made up from very thin stainless steel discs stacked together and welded along their edges. The advantage of the design is the concertina style bellows that is very flexible allowing designs that have long extensions and short compressions. They have two main applications. Firstly, applications that requires a device to extend or contract into the vacuum space where an elastomeric seal on a sliding shaft is not adequate. Such as a sample manipulator or Faraday cup. Secondly, where a device such as a detector or sample positioner is sensitive to vibration and must be decoupled from the vibration source.

## 10. Valves

Along the axis of the beam lines gate valves are used to enable the isolation of sections for maintenance and also to minimise loss of vacuum in the whole accelerator if one section develops a leak. Most gate valves are electro-pneumatically operated and many are interlocked to local high vacuum gauges. The few that are manually operated are for maintenance isolations.

On ANTARES the electro-pneumatically operated valves are interlocked to close when the pressure, as measured near the gate valve, rises above 5 x  $10^{-5}$  Pa. This figure can vary depending on the location of the valve. See the Accelerator WIKI for values around the ANTARES. Once an interlock has tripped the switch that operates the gate valve is bypassed. To reset the valve:

- The vacuum must be within the high vacuum range as read on the corresponding vacuum gauge controller with a pressure better than the trip point.
- Once this level is reached the bypass button is held down momentarily.
- At this point the pressure will rise then fall as the gas load is pumped away.
- When the system pressure is less than the trip level the bypass switch can be let go.
- An indicator light near the switch will show its status.

#### Notes:

- Operators and technicians must be aware that gate valves must be orientated so that the gate always seals towards the vacuum pump. That is, they seal best in one direction only. Failure to orientate the gate the correct way may cause a vacuum leak when atmospheric pressure forces the gate off its seal.
- In some cases gate valves may remain sealed after they have been switched to open. This can be due to the withholding (sometimes called back pressure) pressure not being adequate enough to lift the gate off the seal.
- Gate valves, while mostly metal can become hot if a gate is closed in front of an ion beam. If left for long periods with high current beams the elastomer seals may warm up and begin to outgas.
- For roughing ports typically 25KF bellows sealed valves are used. On STAR these same valves are used to isolate vacuum gauges for cleaning.
- Where fine flow control is required for either roughing out or venting, metering valves are used. These are basically multi-turn needle valves for fine adjustment.
- Valves do require maintenance to keep the seals clean and in working order. After long periods of time the 'o' ring seals will deform and often will not recover their original shape.

There have been attempts to build fast acting gate valves for the purpose of isolating the accelerator tank's load of  $SF_6$  in the event of a breach in a tube. While it seemed like a good idea the force of the valve closing at high speed could damage the gate seal causing the valve to leak. Designers must use care to ensure that a standard high differential pressure rated gate valve isn't adequate before launching into these expense type valves.

# **11. External Interfaces**

### **11.1 Electrical Feedthroughs**

a) Low Voltage, Low Current

There are few applications where voltages greater than 24Vac are used within the vacuum space. For these voltages at a few amps coaxial feedthroughs such as BNC, SHV, MHV, etc, are often used in place of more expensive dedicated commercial types. In one application 200Vdc is used for faraday Cup suppressors but current is only a few micro-amps at most.

Notes:

• Do not use multi-pin signal feedthroughs to carry voltages unless the pins used for voltage can be a few unused pins away from used signal pins.

- Feedthroughs can be purchased is almost any flange style but are usually restricted to small size flanges.
- Insulation must be used on all wires carrying voltage. It is highly recommended to use single or stranded wires (preferably nickel coated) with loose fitting PTFE (Teflon) spaghetti.

### b) Signal

Signal feedthroughs other than coaxial such as BNC, SMC, Microdot, etc are from one to multiple pins. It is recommended that connections to multi-pin feedthroughs are by the use of slide on pins. Many are either sliver of gold plated for good electrical contact. Avoid soldering unless care is used to remove all traces of flux and no pin-holes in the solder have been created

On coaxial feedthroughs the coaxial connector will be either both sides of the feedthrough and hermetically sealed or with the coaxial connector on the atmospheric side and a plain pin on the vacuum side, also hermetically sealed. For either type, where coaxial cable must continue into the vacuum space it is recommended to remove the outer plastic insulation and replace it with loose fitting PTFE spaghetti. This is also recommended for all single or stranded wires carrying signals.

### c) High Voltage, High Current

There is a wide range of high voltage and high current feedthroughs available in most flange types. High voltage feedthroughs usually have a larger insulator in ceramic and sometimes glass. These must be kept clean on the atmospheric side. Long term breakdown on high voltage feedthroughs can damage the insulator causing vacuum leaks.

For high current applications such as heaters, dedicated high current feedthroughs must be used. These are characterised by larger cross section wires to carry the higher currents. Like HV feedthroughs they must be kept clean. It is recommended to clean the insulators on signal feedthroughs periodically to remove any accumulation of dust.

### d) Power

There are few feedthroughs designed for powering devices at mains voltages. It is highly recommended that experiments are designed not to have mains voltages in vacuum vessels or equipment due to safety concerns.

## **11.2** Mechanical Feedthroughs

### a) Rotary

Rotary feedthroughs allow the transition of rotary movement into the vacuum space to, for example, operate shutters or position samples or detectors. They are widely available in many of the common flange styles. There are three common types both of which are very good in high vacuums.

The first type is a continuous shaft between the atmospheric side and vacuum side using rubber vacuum seals in the form of either supported 'o' rings, square or odd shaped rings (like common shaft seals), and flat rubber sheet seals. These are best for general rotary actuation rather than continually rotating. Okay in high vacuum but care must be taken to keep the seals clean and periodically replaced or whenever wear is noticed.

The second type is a ferro-fluidic type which also uses a continuous shaft between the atmospheric side and vacuum but the seal is made by encapsulating a dense fluid around the shaft. These types usually have bearings to keep the shaft centred and free moving. They are best for high revolutions in high vacuum applications.

The third type is magnetically coupled rotary feedthroughs. These are expensive. They provide a seal by magnetically coupling the shaft on the atmospheric side to the shaft on the vacuum side across a continuous sealing plate. If either shaft is removed the vacuum is not compromised. These are excellent for ultrahigh vacuums and lower. They are for moderate speed revolving applications and general actuations.

### **b**) Sliding

Usually these feedthroughs are a round shaft that can slide in and out of the vacuum as well as rotate. Sliding feedthroughs are used for positioning devices or where a linear actuation is necessary. The length can vary from a few millimetres to hundreds. In either case there is a shaft which penetrates from the atmospheric side to the vacuum side. The type of seals used varies like in the rotary seal. Sometimes dual seals are used to minimise leakage especially in high actuation rate applications. In some applications the void between the two seals is pumped increasing the reliance of the seal. These types are good in high vacuums. Notes:

- Care must be taken to ensure the shaft is lubricated with a suitable vacuum oil or grease. It is imperative that the oil or grease on the shaft is changed regularly to minimise dust build.
- There are bellows sealed sliding seals available. These types do not rotate and can only move in and out a few tens of centimetres at most. Very good in high to ultrahigh vacuums.
- Some sliding seals are able to move radially which will affect alignment. Where alignment is crucial a linear bearing type is recommended.

### c) Wobbler

A wobbler feedthrough allows users to use a shaft as a lever or pick up. They are designed around a flexible rubber or bellows seal they allows a greater range of circular movement.

### **11.3 Manipulators**

These are a class of feedthroughs design for high to extremely high precision positioning of samples, detectors or other instruments. They can be purchased having all or either X, Y, Z and rotary movement. The X and Y axes can have various travel lengths however they are typically less than 100 mm each. The Z axis (vertical) can have greater lengths but is limited as with the X and Y axes by the physical limitations of the vacuum bellows.

### 11.4 Actuators

'Actuators', combine sliding and rotary feedthroughs with pneumatic, electro-pneumatic or mechanical actuators to do the work.

In the accelerator area, actuators can be found on Faraday Cups to insert the cup into the beam line on a bellows sealed sliding feedthrough and inside of the ANTARES pressure vessel to move the stripper tube in and out of the beam axis.

### 11.5 Load locks

Load locks are not common in the accelerator area but are ideal for moving samples into a high vacuum space without significant loss of vacuum pressure. A sample or device is mounted onto the end of the load lock shaft. It is inserted into a tube connected to a chamber but isolated from the chamber vacuum by an in-line gate valve. The tube is sealed and then evacuated to a vacuum pressure similar to the chamber. Once the pressures equalise the gate valve is opened and the shaft pushed into the vacuum space. From this point the sample or device can be removed from the shaft by other remote means.

# **12. Internal Devices**

There are many devices that are commonly used inside of vacuum systems. Not all are represented in this section however similar reasons are use to assess new and unusually devices for use in vacuum.

### 12.1 Lighting

Lighting is necessary in most sample measurement chambers where the operator must be able to see the sample in order to position it in front of the ion beam. In most cases a standard bare 12Vac halogen bulb is used with good results. Care must be taken to ensure the bulb can be dimmed as when running at maximum power the heat load can only be dissipated through the connecting wires. That is, there is little gas inside a vacuum to conduct head away from the bulb. Bright white LED's are also used with mixed success. They have less radial light output as compared with a bare halogen bulb. If the LED is setup correctly the light can be focussed towards the necessary target.

Preferably, wires must be stranded or solid wire with a loose Teflon sleeve but normal plastic coated hook up wire works in high vacuums. In some cases where the wires as not likely to come into contact with other components then they are used uninsulated. All wiring must be restrained so that it cannot move into the path of the ion beam. As a rule-of-thumb wires should be at least 20 mm from the beam.

A standard double BNC feedthrough can be used, that is, only the centre core connections are used. There are many off-the-shelf electrical feedthroughs available that will be more suited for this specific use.

### 12.2 Heating

Providing power to a heating stage on a sample (target) stick requires a high current feedthrough. If a feedthrough isn't provided from the supplier of the heating stage then many types can be found from larger vacuum suppliers. Ensure the feedthrough selected is designed to carry the maximum current that can be supplied and not the maximum current likely to be used. Like in the lighting section above, care must be taken to ensure wires are insulated and away from the beam path.

Heating stages rely on heat dissipation through the support for the stage which is usually made from stainless steel, a relatively poor conductor. When opening devices for handling be sure they are cool to touch first.

### 12.3 Cooling

Cooling stages come in 2 distinctly different types. The first is the Peltier cooled stage which is all electric. Powering the stage can be managed as for lighting and heating. The second type is a liquid nitrogen cooled system. It uses an insulated tube to carry LN2 through a feedthrough into the vacuum space. A typical design is where LN2 is pumped through a coil attached to a heavy copper braid. The braid is then connected to the cooling stage where heat is conducted away from the mounting stage. Thermal isolation of the cooling stage from the holder can be Teflon or sapphire standoffs or similar materials.

These cooling devices must only be switched on when the vacuum is at the working level otherwise condensation and possibly ice can form on their surface becoming a large outgassing problem. Likewise ensure the stage is close to room temperature before opening up the vacuum system to atmospheric pressure. Always vent with a dry gas.

# **13. Pumping**

## 13.1 General

It is not the intention of this document to describe the fundamental operation of particular vacuum pumps. Adequate information is available from manufacturer's catalogues. The following is information on what type of systems are used in the accelerator area and the reasons why.

Vacuum pumps don't suck. They act essentially as one way valves in a system where gas is flowing towards them. This is discussed in Chapter 3 of this document. There effectiveness is there ability to stop recoiling gas molecules returning into the vacuum space such as a beam line or vacuum chamber.

When considering what type of pump to use for a vacuum system the following points must be considered.

- What is the application?
- Will it have a relatively high gas load, that is;
- Will it be pumping a static gas load or changing gas load?
- Will there be a high outgassing rate?
- Are there known hidden/trapped voids to be pumped?
- It is a clean or dirty device? Is it contaminated with grease and oil?
- What pumping speed will be needed to maintain a high vacuum?
- What is the volume of the system? What is the conductance of the system? Will a small pump be just as effective as a large pump?
- What gases will be pumped?
- What type of pumps and how many are being used in the vicinity?
- Will the pumping speed need to be regulated such as for pumping near foils or delicate samples, detectors, etc?
- Cycling time whether it will be required to regularly pump from low vacuums
- Will the pumps operate reliably in their chosen application?
- Are they compatible with other pumps used on the accelerators? Are they cost effective?

Another consideration is vibration. Pump vibration can be significant leading to micrphonic interference in detectors and vibration of samples being measured. Vibration can be reduced by selecting pumps with less inherent vibration like turbomolecular pumps or the more fickle ion pumps. If a pump is a source of vibration it can be decoupled from the detector or measurement chamber by the use of a flexible bellows. An fine edge welded bellows is recommended.

## **13.2 High Vacuum Pumps**

See section 4.5, Pump Down characteristics for cryogenic and turbomolecular vacuum pumps.

### <u>Cryopumps</u>

For ANTARES the main beam line pumps in use are cryopumps. The types of pumps trap (or immobilise) gas molecules. They are used because they have a long lifetime between major

services, can be regenerated without removing them from service, have a high pumping speed for most gases and can maintain high vacuums for long periods in a static vacuum application.

#### Turbomolecular pumps

Where a vacuum system is cycled such as at a measurement chamber, then turbo pumps are used. Turbo pumps are transfer type pumps meaning, gas is transferred from the vacuum space into the atmosphere. A turbo pump can routinely recover a vacuum from low vacuum after a measurement chamber has been roughed out without needing regeneration as for a cryopump.

Turbo pumps are also more effective at pumping lighter molecules such as helium and hydrogen as compared with cryopumps. Using turbo pumps near systems that use these gases will reduce the effect of loading on cryopumps.

Most new turbo pumps can be serviced in-house. Some models purchased require special balancing of the bearings after they had been changed. In this case they are sent back to the manufacturer at great cost. It is important that models chosen can be maintained in-house.

#### Other high vacuum pumps

There have been few applications where titanium sublimation or ion pumps have been used in the accelerator area. Most have been stand alone UHV systems or cryostats for specialised applications. The practicality of cleaning and regenerating these gas trapping type vacuum pumps on systems requiring relatively frequent cycling is prohibitive.

Getter materials once used widely in electronics in tubes are becoming more used within larger static volumes such as beam lines and associated equipment. They can be coated onto surfaces to assist in maintaining uniform high vacuums in spaces that have poor pumping speeds.

### **13.3** Low Vacuum, Roughing or backing pumps

#### Oil free pumps

These pumps are categorised by either being an oil type pump or dry (oil free) pump. Oil free vacuum systems are of choice for accelerator systems now that the technology has been proven to be reliable and cost effective. The majority of roughing and backing pumps now in use are scroll pumps as well. Piston pumps have been trialled with little success due to the short life of the seals. Some applications have used diaphragm pumps and multistage roots pumps but scroll pumps remain the most cost effective and easiest to maintain option. The Edwards type scroll was chosen over other brands as this type of scroll pump is designed with the bearings mounted outside of the vacuum which in other brands often caused the bearings to fail from lubricant loss.

#### Oil vane pumps

Low Vacuum, Roughing or backing pumps are all the same type of pump. Oil vane type pumps were widely used in the accelerator area until 2006 when a systematic change began on the STAR accelerator to replace oil vane pumps with oil free scroll pumps. Some oil vane pumps remain in use where some back streaming oil vapour is not considered a problem. If an oil vane pump is to be used for roughing out purposes, it is high recommended that a liquid nitrogen cold trap is used to trap vapours. It is the ultimate aim to have oil free pumping on all accelerators in the future.

# 14. Monitoring

### 14.1 Vacuum Gauges

The standard type of high vacuum gauge in use is the cold cathode penning type gauge.

- On ANTARES the Pfeiffer brand is in use in two versions; the standard cold cathode type and full range type which has an integrated Pirani gauge.
- On STAR the most widely used brand is Leybold.

Generally, vacuums are measured as an indication only for process controlling and establishing basic quality to ensure pressures are within levels that are accepted for "normal" operation. This varies depending on location and use of the vacuum system for example a measurement chamber is cycled often and affects surrounding vacuum systems. It will more than likely be a much high pressure than surrounding static systems.

In most cases, absolute vacuums are not required to be measured. However on some AMS beam lines where thin windows are used it is important to have accurate measurement of pressures when cycling pressures in the gas detectors. If accuracy is required it must be remembered that different gases ionise at different pressures and so pressures readings from the same volume will be different depending on the dominant background gas. Most gauges have characteristics related to Nitrogen (air).

### **14.2 Gauge Controllers**

On ANTARES the most widely used gauge controller is the Pfeiffer TPG300. This is an aging model and is being replaced in some key areas with the Pfeiffer Maxi-gauge which allows 6 heads to be connected at once.

The TPG300 has inputs for up to 4 heads whether Penning or Pirani. The limitation with the TPG300 is the electrical robustness when operated near the accelerator high voltage generator. Many failures have been attributed to high voltage transient surges. The TPG300 is now technologically aged and will ultimately be replaced outright. Various other models of TPG's are in use on ANTARES especially where single gauges are required. It is the TPG300 controllers on ANTARES that provides the interlocking capabilities.

The MAXI gauge is still being trialled as a replacement to the TPG300. A unit under test for many years at the HE end of the accelerator has already failed during an accelerator sparking event. Its real place may be away from the accelerator where the 6 heads allow consolidation of a whole beam line's vacuum monitoring system.

On STAR the common high vacuum gauge is the Leybold cold cathode type. It has been integrated into the STAR control system via cat 5 cable. They operate using a similar principle of measuring current flow across the plasma of the ionising gas. These gauges have been far more unreliable than the Pfeiffer equivalent. Unfortunately they are not interchangeable.

If venting with gas other than air or nitrogen be aware that the response on the Pirani gauges may show lower pressure than actual pressure.

### 14.3 Bourdon Gauges

Bourdon gauges are normally used for indicating that pressure is below atmospheric. They are not used on accelerator systems as they have poor accuracy in the range of interest i.e., below  $1 \times 10^{-3}$  Pa.

# Chapter 3



# **Overview of Basic Vacuum Technology**

The ANSTO accelerators consist of 140 metres of beam lines under vacuum. Supporting these beam lines are 60 individual high vacuum pumping stations. There are over 70 isolation zones along the beam lines to allow isolation of a zone without interrupting the adjacent zones. This facilitates isolations to repair leaking sections, routine maintenance and cycling of pressure in zones to change samples, etc.

The following information are the general tools used by technicians and engineers to design reliable and high performance vacuum systems for the accelerator facilities.

# 1. Vacuum Overview

### 1.1 What is vacuum?

A vacuum is defined as a diluted gas, or the corresponding state at which its pressure or density is lower than that of the ambient surrounding atmosphere.

Due to the Earth's gravity, atmospheric pressure varies with altitude. As a standard atmospheric pressure at sea level  $p_o = 101.325$  kPa. A Pascal is unit of vacuum relating to a force of newtons per metre squared.

An absolute vacuum of 0 kPa is not practically achievable so in real terms we can only approach zero for example on the ANSTO accelerators we have "operating" vacuums better than  $1 \times 10^{-4}$  Pa. In outer space the vacuum is less than  $10^{-12}$  Pa but not 0 Pa as there are some molecules of gas present which exert a very small partial pressure.

The pressure exerted from a vacuum lies between 0 to 100 kPa (see table below). In an ideal pumped vacuum system the pressure will continue to decease approaching 0 kPa. In accelerator systems the working ranges are medium to ultra high vacuums. In some special cases ion sources may operate in the low vacuum range but generally vacuum systems for the majority of accelerator systems are in the high vacuum range with some vacuum end stations close to ultra high vacuum.

### 1.2 Units used

The SI unit  $Pa = N/m^2$  or kg·m<sup>-1</sup>·s<sup>-2</sup>, will be used where any vacuum quantity is referenced. In the accelerator area the vacuum units used are in Pascals (Pa) and although the vacuum pressures are below atmospheric pressure "negative" pressures are not represented as -kPa but of a magnitude which approaches absolute zero using scientific notation, e.g. 1 x 10<sup>-6</sup> Pa.

#### **1.3** Vacuum categories

Vacuums used in accelerator applications vary in magnitude from low vacuum to ultra high vacuum. These categories describe the pressure ranges which in the vacuum world become references for vacuum quality in a given system. Low vacuum is managed differently from high and ultra high vacuums due to the physical limits of the system designs. Generally, a vacuum system must be designed with the desired ultimate vacuum in mind to ensure the most appropriate materials, seals, pumps and vacuum management are used.

The table below shows each of the ranges based on the European system. This is the closest to that used for the ANSTO accelerators. The shaded section represents the working ranges used throughout the accelerator for example, ion sources may have low to high vacuums, beam lines may have very high vacuums and some endstation vacuum chambers may use high to very high vacuums. This document describes the design factors and particular management controls for achieving vacuums in the various ranges.

Range	Pressure (Pa)
Atmospheric pressure	1.013 x 10 <sup>5</sup>
Low vacuum	$1 \times 10^5$ to $3 \times 10^3$
Medium vacuum	$3 \times 10^3$ to $1 \times 10^{-1}$
High vacuum	$1 \times 10^{-1}$ to 1 x $10^{-4}$
Very high vacuum	$1 \ge 10^{-4}$ to $1 \ge 10^{-7}$
Ultra high vacuum (UHV)	$1 \ge 10^{-7}$ to $1 \ge 10^{-10}$
Extremely high vacuum	$< 1 \times 10^{-10}$
Outer Space	$< 1 \times 10^{-12}$
Perfect vacuum	0 Pa

Table 2. Reference pressure ranges (European scale).

A. Berman, Vacuum Engineering Calculations, Formulas, and Solved Exercises, Academic Press, Inc. (1992).

In the accelerator area:

Low Vacuum = atmospheric pressure to medium vacuum

High Vacuum = high vacuum to very high vacuum

Ultra high vacuum = all those equal to and higher than ultra high vacuum

# 2. Pressure Fundamentals

### 2.1 General

To understand the reason why vacuum systems must be carefully engineered to achieve high vacuums consideration must be given to how gases act in a closed chamber.

Any gas enclosed within a volume (a chamber or pipe work) is uniformly distributed or moving towards uniform distribution as in the case of accelerator tubes connected to stripper gas enclosures. The individual gas particles are constantly moving back and forth at high-speed within the volume; upon striking the vessel wall, they exert a force F on surface A due to pulse transmission. The pressure p that is exerted on the wall is defined as:

$$p = F/A$$

If the gas is made up of different types of gases, each of these gases will exert a pressure that corresponds to its concentration; this is called <u>partial pressure</u>. The sum of all partial pressures equals the total pressure. Air is a good example of this: In addition to its main constituents of nitrogen, oxygen and water vapour, air also contains many trace gases, the sum of which equal  $\sim$ 101.3 kPa, total pressure (atmospheric pressure). See table below.

In an accelerator system it would not be uncommon to have various carbon based compounds, sulphur hexafluoride and specific stripper gases present in trace amounts.

Gas	% by volume	Partial Pressure (Pa)		
Ar	0.92	934		
CO <sub>2</sub>	3.26 x 10 <sup>-2</sup>	33		
H <sub>2</sub>	4.9 x 10 <sup>-5</sup>	4.94 x 10-1		
He	5.16 x 10 <sup>-4</sup>	5.23 x 10-1		
Kr	1.1 x 10 <sup>-4</sup>	1.15 x 10-1		
$N_2$	77.17	78180		
Ne	1.8 x 10 <sup>-3</sup>	1.82		
O <sub>2</sub>	20.7	20970		
Xe	8.59 x 10 <sup>-6</sup>	8.7 x 10-3		
H <sub>2</sub> O	1.18	1200		
Other	remainder	remainder		
Total	100 %	101325 Pa		
Notes: H <sub>2</sub> O may vary depending on environment				
"Other" may include: $CH_4$ , $O_3$ , $N_2O$				

Table 3. Partial Pressures in AirThe Vacuum Technology Book – Pfeiffer Vacuum September 2008

### 2.2 Ideal (General) gas equation

1 mole of any gas at STP occupies 22.414 litres. At a temperature 273.15 K (0 °C) and a pressure of 101,325 Pa (standard pressure) 1 mole of any gas contains 6.02 x  $10^{23}$  particles. This is referred to

as Avogadro's number. The mass of the gas thus enclosed is its molecular weight in grams. The ideal gas equation describes the state of a gas as a function of pressure, temperature and volume.

$$pV = nRT$$

Example:

For a chamber, Ø300 mm x 500 mm long, a volume of 0.035 m<sup>3</sup>, calculate the mass of gas in the chamber at a vacuum of 1 x  $10^{-6}$  Pa.

pV = nRT

n = pV/RT  $n = 1 \times 10^{-6} \times 0.035 / 8.31451 \times 293 = 1.43 \times 10^{-11} \text{ moles}$ Mass of remaining volume = moles x mass = 1.43 x 10<sup>-11</sup> x 29 (air) = 0.415 ng No. of molecules = moles x Avogadro's Number = 1.43 x 10<sup>-11</sup> x 6.02 x 10<sup>23</sup> = 8.6 x 10<sup>12</sup> Per cm<sup>3</sup> = 8.6 x 10<sup>12</sup> / 35000 cm<sup>3</sup> = 246 x 10<sup>6</sup> molecules per cm<sup>3</sup>

Where:

 $p = \text{pressure (Pa)}, V = \text{volume (m}^3), n = moles, R = \text{general gas constant} = 8.314510 \text{ kJ/(kmol K)}, T = \text{thermodynamic temperature (K)}, Avogadro's number = 6.02 x 10^{23}$ 

## **3.** Flow Fundamentals

#### **3.1** Mean free path $(\lambda)$

The mean free path is the mean path length that a molecule traverses between two successive impacts with other molecules. High vacuums provide a longer mean free path than low vacuums. In accelerator systems high vacuums are crucial as ions that collide with gas molecules in beam lines loose energy and are not transported to the point of measurement. In the accelerator tubes high mean free paths are crucial to ensure low energy loss in the ion beam and optimised ion beam transmission.

Gas (at 0°C and 1 atm)	λ (nm)
Air	68
Ar	62.6
Cl <sub>2</sub>	27.4
CO	58.6
CO <sub>2</sub>	39
$H_2$	110.6
Не	173.6
Kr	36
$N_2$	58.8
Ne	124
02	63.3
Xe	26

Table 4. Mean free Paths for various gases

Hirschfelder, Curtiss and Bird (1954) Molecular Theory of Gases and Liquids, Wiley, New York

Pressure (Pa)	Molecules / cm <sup>3</sup>	Molecules / m <sup>3</sup>	Mean free path	
101325	$2.7 \times 10^{19}$	$2.7 \times 10^{25}$	68 nm	
	<b>—</b> 11 <b>—</b> 31	1		

Table 5. Other characteristics of Air

### 3.2 Mean Velocity (u) m/s

The residency time of various gases in a system (or the time taken to remove various gases) relates to their individual mean velocity. The following table shows mean velocities for some gases. It can be seen that very light gases travel significant faster than heavy gases. It should be noted that Hydrogen and Helium do not pump efficiently in turbo pumps or cryopumps but heavy gases generally do.

Gas	Molar Mass kg/mol	Mean Velocity m/s	Mach Number
Air	0.028966	463	1.4
Ar	0.039948	394	1.2
Cl <sub>2</sub>	0.0709	296	
СО	0.028011	471	
CO <sub>2</sub>	0.04401	376	1.1
$H_2$	0.002016	1762	5.3
He	0.00402	1246	3.7
Kr	0.0838	272	
$N_2$	0.02801	471	1.4
Ne	0.020179	555	
O <sub>2</sub>	0.03199	441	
Xe	0.1313	217	
H <sub>2</sub> O	0.01802	587	1.8

Table 6. Mean Velocity for gases at 20°C

The Vacuum Technology Book – Pfeiffer Vacuum September 2008

$$u = \sqrt{\frac{8.R.T}{\pi.M}}$$

Where: *M* = Molar mass (kg/mol)

For example: Mean velocity of Argon @  $20^{\circ}$ C u =SQRT (8 x 8.31451 x 293 / 0.039948 x 3.1416) u =394 m/s

#### 3.3 Types of flow

The different flow phases need to be understood in order to select the appropriate pumping system for a particular application. It may be that in most cases a chamber or other piece of equipment is

pumped at the highest flow rate achievable with a given pump but it could be the case for example where a chamber has delicate films inside and gas movement past the foils has the potential to tear them apart. In this case a low steady flow rate is needed.

The flow phases in a vacuum system characterise the gas molecule movement related to pumping speed in a vacuum system. Factors that determine flow include pressure differentials, mean free paths, gas type, geometry, dimensions of the system and temperature. There are many different nomenclatures used to represent flow phases but 3 types in particular are used in the accelerator area, Turbulent, Laminar (both in Viscous phase), and Molecular. Less emphasis is placed on the intermediate or transitional flow as once this state is reached it is assumed that the system is pumping well. At atmospheric pressure up to about 100 Pa, the mean free path of the gas molecules is very small. Therefore, the gas flow is limited by the viscosity of the gas being pumped so the type of flow is called Viscous.

In an *ideal* system, if a roughing pump has a pumping speed of say 22 m<sup>3</sup>/hr and there is negligible flow resistance between the pump and the chamber then a chamber of  $\emptyset$ 300 mm ID x 500 long (a volume of 0.35 m<sup>3</sup>) at atmospheric pressure, will take approximately 1 minute to remove the bulk of the gas. During this time the flow remains in the [Viscous] Laminar and/or Turbulent phase.

Where a system has a leak, depending on the magnitude of the leak it is possible that the pumping will plateau at either the Turbulent, Laminar or Molecular flow phase. With outgassing systems the less referred to transitional state may be reached and with systems designed to reach ultra high vacuums outgassing may be occurring well into the molecular flow state.

Pumping	Flow Phase		Description	Type of vacuum	Flow rate Pa-l/sec
Pumping begins		Turbulent Flow	Initial flow experienced at start of pumping or in a low vacuum environment where the flow rate is high. Chaotic movement of molecules. Eddies and other non-parallel flows occur.	Low Vacuum	>1.01
	Viscous	Laminar Flow	Initial flow experienced at start of pumping or in a low vacuum environment where the flow rate is low. Gas molecules bouncing off other gas molecules (gas-gas) in the vacuum system. Short mean free path. Molecules streaming from system.	Medium Vacuum	10.1 to 1.01 x 10 <sup>-4</sup>
	Transitional Flow (Knudsen)	Intermediate flow between laminar and molecular. Flow is dominated by both gas-gas and gas-wall collisions.	Medium to High Vacuum	1.01 x 10 <sup>-2</sup> to 1.01 x 10 <sup>-5</sup>	
Base vacuum reached	Molecular Flow		Remaining gas molecules bounce off the walls (gas-wall) of the vacuum system and not each other. Long mean free paths. Flow rate very small.	High to Ultra-High Vacuum	<1.01 x 10 <sup>-5</sup>

Table 7. Vacuum flow phases
By evaluating the Knudsen (Kn) and Reynolds (Re) number, one can predict the various flow phases. These phases can be seen in the picture below which also shows how the gas molecules behave in the different flow phases moving through the opening d.

#### 3.4 Knudsen Number (Kn)

The Knudsen number is a dimensionless number defined as the ratio of the molecular mean free path length to a representative physical length scale. The Knudsen number in a viscous flow phase < 0.01 and molecule-molecule collisions dominate gas behaviour which behaves as a fluid. In molecular flow Kn > 1 and molecule-surface collisions dominate.

$$Kn = \frac{\lambda}{d}$$

 $\lambda$  = Mean free path

d = diameter of the opening that gas will pass through

#### 3.5 Reynolds Number (Re)

Reynolds number indicates whether the flow of a gas is absolutely steady (laminar flow) or on average steady but with small, unsteady changes (turbulent flow). The Reynolds number, Re, has no dimensions and is defined as the size of the flow.

Osborne Reynolds demonstrated in 1883 that the change from laminar to turbulent flow in a pipe occurs when the value of the Reynolds number exceeds 2,100. The exact value of Re for which the flow changes from laminar to turbulent depends on the geometry of the component, its surface roughness and other experimental factors. During evacuation of a vessel, turbulent flow normally occurs only for a short period of time at the beginning. It has been found to be approximately proportional to the root of the pressure gradient.

$$\operatorname{Re} = \left(\frac{\rho}{\eta}\right) d.v$$

 $\rho$  = gas density (kg/m<sup>3</sup>)  $\eta$  = viscosity (Pa.s) (Pascal-second) v = flow velocity (m/s) d = tube diameter (m)

Gas	Formula	Molecular weight	Gas Density [2] kg/m <sup>3</sup>	Viscosity η Pa.s
Air [1]	$N_2 + O_2$	29	1.1839	1.79 x 10 <sup>-5</sup>
Ammonia	NH <sub>3</sub>	17.031	0.7449	1.01 x 10 <sup>-5</sup>
Argon	Ar	39.948	1.723	2.24 x 10 <sup>-5</sup>
Carbon Dioxide	$CO_2$	44.01	1.9105	1.51 x 10 <sup>-5</sup>
Carbon Monoxide	CO	28.01	1.2082	1.74 x 10 <sup>-5</sup>
Chlorine	Cl <sub>2</sub>	70.906	3.1124	1.34 x 10 <sup>-5</sup>
Helium	He	4.02	0.171	1.99 x 10 <sup>-5</sup>
Hydrogen	$H_2$	2.016	0.0868	8.80 x 10 <sup>-5</sup>
Hydrochloric Acid	HCl	36.5	1.5844	1.46 x 10 <sup>-5</sup>
Hydrogen Sulphide	$H_2S$	34.076	1.4876	1.26 x 10 <sup>-5</sup>
iso-Butane	$C_4H_{10}$	58.12	2.3758	7.49 x 10 <sup>-5</sup>
Methane	$CH_4$	16.043	0.6556	1.11 x 10 <sup>-5</sup>
Nitrogen	$N_2$	28.02	1.2088	1.76 x 10 <sup>-5</sup>
Oxygen	$O_2$	32	1.381	2.02 x 10 <sup>-5</sup>
Propane	C <sub>3</sub> H <sub>8</sub>	44.09	1.8024	8.26 x 10 <sup>-5</sup>
Sulphur Hexafluoride	$SF_6$	146.5	6.27	$1.53 \times 10^{-5}$

Table 8. Gas density and viscosities

Unless otherwise indicated gases referenced from McGraw Hill Chemical Properties Handbook 1 atm 25°C. [1] Air STD Atmosphere at Sea Level: 1 atm 15°C [2] Gas Density Values Interpolated From 15°C Data using Charles Law

http://pipeng.com/index.php/gsts/itdmodflup002a/itddaflup00201

#### 3.6 Viscous Flow

#### Low vacuum, p = 100000 - 100 Pa, where $\lambda \ll d$

What characterises viscous flows, is frequent contact between gas molecules, yet less frequent contact with the walls of the vessel. The mean free path of the gas molecules is therefore very small and the gas flow is limited by the viscosity of the gas being pumped (Viscous Flow). In this case, the mean free path of the gas molecules is significantly shorter than the dimensions d of the vacuum equipment. For both Laminar and Turbulent Flow, Kn < 0.01. In addition, the term viscous flow is used if the product of pressure p and diameter d of the components through which gas is flowing is p.d  $\geq$  60 Pa.cm for air. Whether the flow is in the Laminar or Turbulent flows phases can be calculated using the Reynolds Number formula above.

#### 3.7 Knudsen flow

#### Medium vacuum, p = 100 - 0.1 Pa with $\lambda \le d$

If the Knudsen number is between 0.01 and 0.5, this is termed Knudsen flow. Because many process pressures are in the medium vacuum range, this type of flow occurs with corresponding frequency. Since this is a transitional flow, this range is transited relatively quickly when pumping down vacuum chambers and hence not usually considered in accelerator systems. This means that the influence of this conductivity on pump-down times is correspondingly low. A simple approximation for the Knudsen range can be obtained by adding the laminar and molecular conductivities. The

table in "Conductivities" below shows the conductivities of round, one meter long tubes of differing diameters in all three flow ranges.

#### 3.8 Molecular flow High vacuum, $(p = 0.1 - 10^{-5} \text{ kPa})$ , where $\lambda > d$ and in UHV $(p < 10^{-5} \text{ kPa})$ , with $\lambda >> d$

At Knudsen numbers of Kn > 0.5 molecule-molecule collisions virtually no longer occur. What prevails is molecular flow. In this case, the product of pressure p and component diameter d is p x d  $\leq$  1.3 Pa.cm.



Fig 5. Molecular paths during different flow phases Re reference: http://www.engineersedge.com/fluid\_flow/pressure\_drop/pressure\_drop.htm

#### **3.9 Pump throughput qpV**

The concept of pump throughput is of major significance in practice and should not be confused with the pumping speed! The pump throughput is the quantity of gas moved by the pump over a unit of time, expressed in Pa.1.s<sup>-1</sup>. Conversely, the pumping speed is the capacity of the pump to remove a volume of gas within a specific unit of time, measured in  $m^3/h$  or 1/s.

The throughput value is important in determining the size of the backing pump in relationship to the size of a high vacuum pump with which it is connected in series in order to ensure that the backing pump will be able to take away the gas moved by the high vacuum pump.

The pumping capacity (throughput) for a pump is equal either to the mass (m) flow through the pump intake port in a specified time:

$$q_m = \frac{m}{t}$$

Or to the pV (quantity of gas) flow through the pump's intake port:

$$q_{pV} = \frac{pV}{t}$$

It is normally specified in Pa.l.s<sup>-1</sup>. Here p is the pressure on the intake side of the pump. If p and V are constant at the intake side of the pump, the throughput of this pump can be expressed with the simple equation:

 $q_{pV} = p.S$ 

Where:

S = pumping speed of the pump at intake pressure of p.

#### 3.10 Speed of vacuum pump S

The speed of a vacuum pump is defined as

$$S = \frac{q_{pV}}{p}$$

p = Pressure at the pump inlet

However, pumps are usually connected to vacuum chambers via hoses resulting in a lower effective pumping speed  $S_{eff}$  at the chamber. If  $p_1$  is the pressure at the pump and  $p_2$  is the pressure in the vacuum chamber then:

$$q_{pV} = S.p_1 = S_{eff}.p_2$$

for continuous flow. Combining this with  $q_{pV} = C(p_1 - p_2)$  (see Conductance below) results in an expression for S<sub>eff</sub>

$$S_{eff} = \frac{S.C}{S+C}$$

#### **3.11** Conductance C

In a vacuum system, the volume that is pumped by a vacuum pump exhibits a level of flow resistance (Z) due to chamber and pipe sizes and their geometries, and the various sized orifices and other in-stream devices. The terminology used to describe this effect is the reciprocal of flow resistance which is *conductance* (C) or the systems ability to conduct gas.

Conductance is expressed in either l/s or sometimes  $m^3/h$  and is usually for steady, continuous flow through one or more components in a vacuum system. It is affected by the geometry of the piping element and relative to the flow phase and capacity of the pump used. In the high and ultrahigh vacuum ranges (molecular flows), C is a constant which is independent of pressure. In the rough and medium-high vacuums (turbulent and laminar) it is, by contrast, dependent on pressure. As a consequence, the calculation of C for the piping (beam line) elements must be carried out separately for the individual pressure ranges.

Flow resistance (sec/litre) 
$$Z = \frac{P_1 - P_2}{q_{_{PV}}}$$

Conductance (litre/sec) therefore

 $C = \frac{1}{Z} = \frac{q_{_{PV}}}{P_1 - P_2} \qquad (continued over)$ 

$$q_{pV} = C(p_1 - p_2)$$

Where:  $q_{pV}$  = Throughput flow rate p = Pressure (Pa)

At the molecular level, the conductivity of a system is reduced due to the friction of both the walls of the system and other molecules. As discussed above in "Types of Flow", these two affects occur at different flow phases. The total effect of conductance is the sum of the various conductance elements. In the case where a valve, a trap and an elbow in series each having a different conductance, the sum is:

$$\frac{1}{C_{total}} = \frac{1}{C_{valve}} + \frac{1}{C_{trap}} + \frac{1}{C_{elbow}}$$

If the components with different conductances are connected in the flow path in parallel, for example, 2 different diameter tubes connecting a chamber to a pump then the equation will be:

$$C_{_{Total}} = C_{_{tube1}} + C_{_{tube2}}$$

For the design of vacuum systems used on the accelerators conductance is mostly calculated for systems in molecular flow.



*Fig 6. Diagram for estimating pipe conductance Pupp/Hartmann, Vakuumtechnik, Grundlagen und Anwendugen, Hanser Verlag* 

### 4. Limitations

The most fundamental problems with vacuum systems are leaks and outgassing. Leaks can be categorised as either real leaks where gas enters the vacuum system from outside of the vessel or leaks form trapped voids which is in effect an internal leak. Trapped voids can contain gas that will continue to outgas for long periods reducing the ultimate vacuum that can be reached in a vessel. Real leaks can be found with the aid of a helium leak detector whereas internal leaks from trapped voids may never be found.

Outgassing can be minimised through careful selection of materials used within the vacuum space. The appendices have helpful tables to estimate outgassing rates. Outgassing may be in the form of desorption, diffusion or vaporisation. Permeation is not outgassing as the source of gas originates from outside of the vacuum space.

Examples commonly used substances in the accelerator systems that outgas,:

- Teflon, PVC, Ertalyte
- Viton, neoprene
- Copper, aluminium, stainless steel, brass, tantalum, rubidium, caesium, lithium, zinc
- Vacuum pump oil, vacuum greases, vacuum epoxies

These are just a few but it demonstrates that all materials will outgas at some point when the temperature and vacuum pressure reach their individual vapour pressure.

Neither of the problems is resolved by increasing the pumping speed (capacity) of the vacuum pump. There will be a point where the rate of gas entering the system will equalise with the pumping speed of the pump. The rate may vary depending on the vapour pressure of the substance outgassing and the temperature of that substance.



Fig 7. Limitation of Pumping From: http://www.mse.ncsu.edu/WideBandgaps/classes/MSE%20751/Handouts/HO02\_Leakage.pdf



Fig 8. Unwanted gas source wheel

#### 4.1 Contamination

Vacuum chambers must be clean in order to reach the desired pressure as quickly as possible when they are pumped down. Typical contaminants include oil and grease on screws and seals, process reaction products or condensed vapours, particularly water that is adsorbed on the walls of the vessel. Consequently, it is necessary to ensure that the components are clean when installing vacuum equipment. All components attached in the vacuum chamber must be clean and grease-free. All seals must also be installed dry. If high or ultra high vacuum is to be generated, clean gloves must be worn during the assembly process.

Examples of common contaminants in accelerator vacuum systems

- Rotary pump oil
- Water
- Plasticisers from various plastics
- General airborne dust
- Machining oil
- Residual gases from stripper gases, venting gases, ion source gases
- Sample breakdown

#### 4.2 Condensation and vaporisation

All substances can occur in a liquid, solid or gaseous state. Their aggregate status is a function of pressure and temperature. Liquids are transformed into their gaseous state through vaporisation, solids through sublimation. The separation of liquids or solids out of the gaseous phase is termed condensation. Since normal room air contains approximately 10g of water vapour per m<sup>3</sup>, condensed water vapour is always present on all surfaces.

Adsorption on surfaces is especially pronounced due to the strong polarity of the water molecules. Natural fibres, in particular, such as paper, contain large quantities of water that escape during drying processes under vacuum. Cooled condensers are used to separate the water vapour in this connection. Even some metals (Cd, Zn, Mg) can vaporise in noticeable quantity at temperatures of several 100 °C. Consequently, use of these metals is avoided in plant construction.

#### 4.3 Desorption

In addition to water, other substances (oil) can be adsorbed on surfaces. Substances can also diffuse out of the metal walls, which can be evidenced in the residual gas. In the case of particularly rigorous requirements, stainless steel vessels can be baked out under vacuum, thus driving the majority of the volatile components out of the metal walls.

Gas molecules, (primarily water) are bound to the interior surfaces of the vacuum chamber through adsorption and absorption, and gradually desorb again under vacuum. The desorption rate of the metal and glass surfaces in the vacuum system produces a gas yield that is a function of time, however. A good approximation can be obtained by assuming that after a given point in time  $t > t_0$  to the reduction will occur on a linear basis over time.  $t_0$  is typically assumed to be one hour.



Fig 9. Desorption curves



Fig 10. Adsorption Curves From: http://www.mse.ncsu.edu/WideBandgaps/classes/MSE%20751/Handouts/HO02\_Leakage.pdf

#### 4.4 Diffusion with desorption

At operation below  $10^{-4}$  kPa, desorption of plastic surfaces, particularly the seals, assumes greater significance. Plastics mainly give off the gases that are dissolved in these plastics, which first must diffuse on the surface.

Following extended pump downtimes, desorption from plastics can therefore dominate the metal surfaces. Although the surface areas of the seals are relatively small; the decrease in desorption rate over time occurs more slowly in the case of metal surfaces. As an approximation it can be assumed that the reduction over time will occur at the square root of the time.

The gas produced from plastic surfaces can thus be described as:

Desorption from plastic material  $Q_{diff} = q_{diff} \cdot A \sqrt{t_o/t}$ 

Where Ad denotes the surface area of the plastics in the vacuum chamber and  $q_{diff}$  denotes the surface area-specific desorption rate for the respective plastic. At even lower pressures, similar effects also occur with metals, from which hydrogen and carbon escape in the form of CO and CO<sub>2</sub> and can be seen in the residual gas spectrum.

#### 4.5 **Permeation and leaks**

For a gas passing through small holes in a thin wall in the Knudsen Flow regime, the number of molecules that pass through a hole is proportional to the pressure of the gas and inversely proportional to its molecular weight.

To Convert to Leakage	Multiply Helium Leak Rate by:				
Rate of:	Laminar Flow	<b>Molecular Flow</b>			
Argon	0.88	0.316			
Air	1.08	0.374			
Nitrogen	1.12	0.374			
Water vapour	2.09	0.469			
Hydrogen	2.23	1.410			

Table 9. Conversation table for leak rates

Seals, and even metal walls, can be penetrated by small gas molecules, such as helium, through diffusion. Since this process is not a function of time, it results in a sustained increase in the desired ultimate pressure. The permeation gas flow is proportional to the pressure gradient  $p_0 / d$  (d = wall thickness,  $p_0 =$  atmospheric pressure = ambient pressure) and to the permeation constants for the various materials  $k_{perm}$ .

Permeation

 $k_{perm}$ .  $Q_{perm} = k_{perm}$ .  $A \cdot p_0/d$ 

Permeation first manifests itself at pressures below  $10^{-6}$  kPa.  $Q_1$ , denotes the leakage rate, i.e. a gas flow that enters the vacuum system through leaks at a volume of V. The leakage rate is defined as the pressure rise  $\Delta p$  over time  $\Delta t$ :

Leakage rate  $Q1 = (\Delta p \cdot V)/\Delta t$ 

If a vessel is continuously pumped out at a volume flow rate S, an equilibrium pressure  $p_{gl}$  will be produced. Throughput is equal to the leakage rate  $Q_l = S \cdot p_{gl}$ . A system is considered to be

adequately tight if the equilibrium pressure  $p_{gl}$  is approximately 10 % of the working pressure. If, for example, a working pressure of  $10^{-4}$  kPa is attained and the vacuum pump that is being used has a pumping speed of 100 I/s, the leakage rate should not be more than  $10^{-3}$  kPa I/s. This corresponds to a leak of approximately 20.20  $\mu$ m<sup>2</sup> in size. Leakage rates  $Q_l$  of less than  $10^{-6}$  kPa I/s can usually be easily attained in clean stainless steel vessels. The ultimate pressure achievable after a given period of time *t* primarily depends upon all of the effects described above and upon the pumping speed of the vacuum pump. The prerequisite is naturally that the ultimate pressure will be high relative to the base pressure of the vacuum pump.



Fig 11. Permeation of Different Gases From: http://www.mse.ncsu.edu/WideBandgaps/classes/MSE%20751/Handouts/HO02\_Leakage.pdf

#### 4.6 Leaks

The source of leaks is as wide and varied as can be imagined. There is no such thing as a common leak however there are several types generally seen. The following lists types in rough order of prevalence:

- Dirty seals elastomer type seals with traces of dust, dirt and fibres on the surface
- Jarred seals ill fitted flanges where the seal lays across the sealing surface
- Vacuum pumping tubes where a fitting has been fitted without care and the pump cannot reach its full capability
- Distortion over tightened or misaligned flanges and fittings that "lift" seals off the sealing surface
- Stress cracks usually in places around flanges and fittings that have been incorrectly tightened
- Pressure dependant mostly related to accelerator tubes that develop leaky seals allowing insulation gas to enter when the pressure is greater than atmospheric pressure.

#### 4.7 Bake-out

The following prerequisites must be satisfied in order to achieve lower pressures ( $<10^{-6}$  kPa):

- The base pressure of the vacuum pump should be a factor of 10 lower than the required ultimate pressure
- Stainless steel vacuum recipients and components must be used
- Metallic seals (CF flange connections) are required
- Leaks must be avoided and eliminated prior to activating the heater (use helium leak detectors!)
- Clean work is a must, i.e. all parts must be thoroughly cleaned and must be installed with grease-free gloves
- Pump and equipment must be baked out

Bake-out significantly increases desorption and diffusion rates, and this produces significantly shorter pumping times. Bake-out temperatures of up to 300°C are used. The instructions of the pump manufacturers relating to maximum bake-out temperatures and maximum permissible radiation levels in the pump flange must be observed.

Following installation the equipment is switched on, and after reaching a pressure of  $P < 10^{-3}$  kPa the heater is then switched on. During the heating process, all gauge heads must be operated and degassed at intervals of 10 hours. In the case of stainless steel vessels and the use of metallic seals, bake-out temperatures of 120°C and heating times of approximately 48 hours are sufficient for advancing into the pressure range of  $10^{-8}$  kPa. Bake-out should be continued until 100 times the expected ultimate pressure is attained. The heaters for the pump and vacuum chamber are then switched off. After cool-down, the desired ultimate pressure will probably be achieved. In connection with pressures  $P < 5 \times 10^{-8}$  kPa and large interior surface areas, it will be advantageous to use a titanium sublimation pump that pumps the hydrogen escaping from the metals at a high volume flow rate.

#### 4.8 Residual gas spectrum

When leaks have been solved in a vacuum system and poor vacuum persists than a residual gas analysis (RGA) measurement should be made to determine the composition of the gas load. This will give vital information on where the vacuum problem may be. Interpreting the information will be trial and error as users will have to think about all possible sources of gases (and outgassing) that may be present. Also relative ratios of gases should be considered to make sense of the measurement. Care should be taken especially if making a measurement near a source such as a cryopump. Some gases that have been trapped on the cryopump may be liberating from the cold surface and will make up a part of the gas spectrum. Other places to take care are near the accelerator tubes and ion sources.

If developing an ultra high vacuum system it is important to know all sources of gas otherwise moving lower than  $1 \times 10^{-6}$  Pa will be an unnecessary challenge. For all other systems it is nice to know but not essential unless residual gases affect ion beam measurements. The dominate gases (and their masses) that may be seen include:

Gas	Mass	Gas	Mass
Water	18	Carbon Monoxide	28
fragment HO	17	Carbon Dioxide	44
Nitrogen	28	Argon	40
Oxygen	32	Sulphur Hexafluoride	146
Hydrogen	2	Helium	4

Table 10. Table of masses for common "in vacuum" gases

# **Appendix 1 – Structural Calculations for Scientific Vacuum Vessel Design**

Calculations for Vacuum Vessel wall and end plate thickness

The following formulae are from the Vacuum Society of Australia (VSA) training course notes which have been in circulation since the early 1980's. VSA continues to support these calculations for training vacuum technologists around Australia. The calculations provide conservative estimates of vacuum vessel wall thicknesses as compared with formulas used in ASME Section VIII – Division 1 UG-28 (Thickness of Shells and Tubes under External Pressure) and associated documents, which have been developed for larger industrial type vacuum vessels.

#### Cylindrical Vessel – Stainless Steel @ 20°C

Thickness of plate in a given diameter (h):

$$\frac{D}{h} \le 105, \ \frac{h}{D} \ge \frac{1}{105}$$

Where: D = diameterh = thickness

Maximum length of vessel for a given diameter (L<sub>c</sub>):

$$\frac{L_c}{D} \le 11.5$$

Where: D = diameter $L_c = \text{length}$ 

#### **End Plates – Stainless Steel**

Thickness of plate in a given diameter (h<sub>1</sub>):

$$\frac{D_1}{h_1} \le 89$$
 ,  $\frac{h_1}{D_1} \ge \frac{1}{89}$ 

Where:  $D_1$  = diameter  $h_1$  = thickness

Minimum thickness for given deflection at centre:

 $\frac{h_1}{\delta} \ge 3$ 

Where:  $\delta$  = deflection  $h_1$  = thickness For Hemispherical End (h<sub>2</sub>):

$$\frac{R}{h_2} \le 830 \text{ requires } \frac{h_2}{R} \ge \frac{1}{830}$$

Where: R =radius  $h_2 =$  thickness

**Example 1:** To construct Stainless Steel vacuum chamber at  $20^{\circ}$  C as shown – Length L = 50 cm = 500 mm, D = 40 cm = 400 mm, Maximum allowable deflection at centre  $\delta = 1$  mm. Find  $h, h_1, h_2$  to satisfy vacuum chamber strength requirements.

#### For Cylinder

1. 
$$\frac{D}{h} \le 105$$
,  $\frac{h}{D} \ge \frac{1}{105}$   $h \ge 400/105$ ,  $h \ge 3.8 \text{ mm}$   
2.  $\frac{L_c}{D} \le 11.5$ ,  $L_c \le 11.5 \times 400$   $L_c \le 4600 \text{ mm}$  with  $L = 500 \text{ mm} \checkmark$ 

#### **For End Plates**

1. 
$$\frac{D_1}{h_1} \le 89$$
,  $\frac{h_1}{D_1} \ge \frac{1}{89}$   $h_1 \ge 396.2/89$ ,  $h_1 \ge 4.45 \text{ mm}$   
2.  $\frac{h_1}{\delta} \ge 3$ , with  $\delta = 1 \text{ mm}$  then require  $h_1 \ge 3 \text{ mm}$ , however  $h_1 = 4.45 \text{ mm} \checkmark$ 

#### For Hemispherical End

1. 
$$\frac{R}{h_2} \le 830$$
 requires  $\frac{h_2}{R} \ge \frac{1}{830}$   $h_2 \ge 198.1/830 \ge 0.24$ mm

Dimension of cylindrical, planar and hemispherical parts of vacuum enclosures

	At	Cylin	nders	End Plates		Hemispherical
Material	Temp (°C)	D/h	$L_c/D$	$D_1/h_1$	$h_l/\delta$	<i>R/h</i> <sub>2</sub>
Copper	20	84	10	52	15	600
Copper	500	58	8.5	-	-	-
Nickel	20	100	11	73	8	780
Nickel	500	90	10.5	-	-	-
Aluminium Alloy	20	70	9	37	57	470
Aluminium Alloy	500	62	8.7	-	-	-
Stainless Steel (304)	20	105	11.5	89	3	830
Stainless Steel (304)	500	89	10.5	-	-	-
Glass (hard)	20	70	9	16	117	470
Neoprene	20	2.5	1.7	10	0.2	30
Teflon	20	12	3.8	14	9	-
PVC (Tygon)	-	3.7	2.1	-	-	-
Perspex	-	_	-	30	-	-
Mica	-	_	-	58	15	-

Factor tables for thickness calculations of common materials used in high vacuum systems

 $\delta$  = maximum permissible deflection at centre of plate L<sub>c</sub> = critical length for cylinder = 1.11D(D/h)1/2

**Example 2:** Using the ASME Boiler and Pressure Vessel Code (the Code) Section VIII, Division 1 for the safe design pressures for a vacuum vessel is used to calculate the maximum external pressure acceptable for a given wall thickness. The following example uses the same vessel dimensions as in Example 1, with a wall thickness of 3.8 mm as calculated using the VSA method.

The calculations reference the Vacuum Vessel Cylindrical Shell Thickness using paragraphs UG-28, UG-27 of ASME Boiler and Pressure Vessel Code.

Vessel shell thickness t = 3.8 mm  $D_o = 400$  mm (outer diameter of shell)  $D_o/t = 105.26$  Since this ratio is greater than 10, follow UG-28(c)(1) L = 500 mm (length of stiffened shell)  $L/D_o = 1.25$ E = 28 x 10<sup>6</sup> (modulus of elasticity for 304 stainless steel)

From Table G in Sec II, Subpart 3 to determine Factor A

A = 1 x 10<sup>-3</sup> (Factor A, from Table G of Sec. II, Subpart 3)  $P_{ext} = \frac{2AE}{3(\frac{D_o}{t})}$ 

 $P_ext = 177.33$  psi (max allowable working external pressure for given *t*) = 1222 kPa

A wall thickness of t = 3.8 mm results in a maximum allowable working external pressure of P\_ext = 325 kPa, which is greater than the external pressure that the vacuum vessel will see.

**Example 3:** For comparison, calculate the maximum external pressure as in Example 2 but with a wall thickness of 2.5 mm.

 $D_{o} = 400 \text{ mm}$ t = 2.5  $D_{o}/t = 160$ L = 500 L/D\_{o} = 1.25 E = 28 x 10<sup>6</sup> (modulus of elasticity for 304 stainless steel)

From Table G in Sec II, Subpart 3 to determine Factor A

A =  $5.3 \times 10^{-4}$  (Factor A, from Table G of Sec. II, Subpart 3)

$$P\_ext = \frac{2AE}{3(\frac{D_o}{t})}$$

P\_ext = 61 psi (max allowable working external pressure for given t of 2.5 mm) = 420 kPa

Therefore a wall thickness of 2.5 mm has a P\_ext of 420 kPa.

**Example 4:** This is a worked example for a large vacuum vessel from ASME Boiler and Pressure Vessel Code (the Code) Section VIII, Division 1, paragraph UG-28. It is in imperial units.

Calculate the maximum allowed external working pressure following UG-28. The minimum required thickness takes into account the support of the stiffening rings. Note that the actual vessel shell thickness t = 0.375-inch

 $D_o = 38.0$  inch (965 mm) (outer diameter of shell) t = 0.261 inch (6.63 mm) (minimum required thickness of shell for external pressure of 14.5-psia)  $D_o/t = 145.594$  Since this ratio is greater than 10, follow UG-28(c)(1) L 235.7 inch (6057.5 mm) (length of stiffened shell)  $L/D_o = 6.203$ E =29x10<sup>6</sup> psi (modulus of elasticity for SA-516 Gr 60 carbon steel)

From Table G in Sec II, Subpart 3 to determine Factor A A =  $1.108 \times 10^{-4}$  (Factor A, from Table G of Sec. II, Subpart 3)

$$P\_ext = \frac{2AE}{3(\frac{D_o}{t})}$$

P\_ext = 14.709 psi (maximum allowable working external pressure for given t) = 101.35 kPa

A wall thickness of t = 0.261 inch results in a maximum allowable working external pressure of P\_ext = 14.7 psia, which is greater than the external pressure that the vacuum vessel will see. Since the actual wall thickness is 0.375 inch (9.5 mm), the vessel design is adequate for the working external pressure.



Factor A table for Stainless Steels

# **Appendix 2 – Low Outgassing Specialist Non Metal Materials**

#### • Celazole® PBI (PolyBenzImidazole) http://www.boedeker.com/celazo\_p.htm

Celazole® is the highest temperature-capable plastic available. However, it is very brittle (almost ceramic-like) and quite difficult to machine. That said, is it frequently used for bushings, bearings, rollers, and spacers in extreme environments. Its outgassing values are listed as 2.50% TML, 0.00% CVCM, 0.40% WVR.

#### • Vespel® (Polyimide)

http://www2.dupont.com/Vespel/en\_US/assets/downloads/vespel\_gen/E61500.pdf

DuPont Vespel® SP-1 is one of the most-used high-temperature plastic materials used in applications where high-purity and electrical properties are needed. Vespel is frequently used in ultra-clean semiconductor and chemical applications. It is also one of the most expensive materials sold, but is flight-approved for NASA, USAF and other aerospace agencies. Its NASA outgassing values are listed as 1.09% TML, 0.00% CVCM, 0.40% WVR.

#### • Duratron® XP (Polyimide)

http://www.portplastics.com/download/pdf/plastics/highPerformance/highPerformance26.pdf

Duratron® XP is the first real alternative to Vespel ... it was developed specifically to replace Vespel in extreme applications at a slightly lower price. It contains less than 1% metallic impurities as measured using the ICP-MS test standard. Duratron XP is ideal for use in high-energy gas plasma etch and strip processes. Outgassing values for Duratron XP are 0.75% TML, 0.00% CVCM, 0.49% WVR.

#### • Torlon® 4203 unfilled PAI (PolyAmide-Imide) http://www.boedeker.com/torlon-4203.htm

Unfilled Torlon® 4203 has high dielectric properties and low thermal expansion, and is much less expensive than some advanced polymers. Torlon 4203 is typically used for insulators, spacers, and mechanical parts up to 520°F. Its outgassing values are listed as 1.85% TML, 0.00% CVCM and 0.49% WVR.

#### • Torlon® 5530 glass-filled PAI (PolyAmide-Imide) http://www.boedeker.com/torlon-5530.htm

Torlon 5530 (30% glass-filled) is typically used for applications where dimensional stability over a wide temperature range is needed, as with temperature test sockets, nests, and fixtures. Its outgassing values are listed as 0.58% TML, 0.00% CVCM (% WVR is not shown). NOTE: Torlon's moisture absorption is a bit high, so critical dimensional stability can be an issue.

#### • Semitron® ESd 500HR (filled PTFE)

http://www.portplastics.com/download/pdf/plastics/staticControl/staticControl18.pdf

Semitron® ESd 500HR is antistatic/conductive PTFE. This material is relatively clean, readily machinable, dissipates static electricity reliably ... as a result it is used in test handling equipment, fixtures, and other applications where static generation may cause failures and/or errors in production environments. This material has low outgassing values of 0.04% TML, 0.00% CVCM and 0.01 % WVR. PTFE has good mechanical properties up to approximately 500°F.

#### • Neoflon® PCTFE (PolyChloroTetraFluoroEthylene)

http://www.aftonplastics.com/materials/pdfs/neoflan\_pctfe.pdf

PCTFE exhibits high chemical resistance, low and high temperature capability, resistance to most chemicals (including strong acids and bases), low friction, electrical and thermal insulation, and "slipperiness". PCTFE has the lowest outgassing values of any thermoplastic material we sell ... 0.01% TML, 0.00% CVCM, 0.00% WVR.

• **PEEK (PolyEtherEtherKetone)** http://www.dotmar.com.au/ketron-peek-1000/ketron-peek-1000-polyetheretherketone.html

PEEK is pure, easily machinable, chemically resistant, stable, and also has relatively low outgassing values (0.31% TML, 0.00% CVCM, 0.06% WVR). PEEK has good mechanical properties, but will not take temperatures over  $350^{\circ}$ F, so it may not have the mechanical or thermal performance needed.

• **Techtron® PPS (PolyPhenylene Sulfide)** http://www.dotmar.com.au/techtron-hpv-pps/techtron-hpv-pps.html

Techtron® PPS is easily machined to close tolerance, has excellent mechanical, thermal and chemical stability and has one of the lowest outgassing values of any thermoplastic material we offer (0.04% TML, 0.00% CVCM ... % WVR is not shown). Techtron PPS is generally a bit less expensive than PEEK or Torlon, but again, will not take as high temperatures.

• Ultem® PEI (PolyEtherImide) http://www.boedeker.com/ultem\_p.htm

Ultem<sup>®</sup> has good dielectric properties and low thermal expansion, and is considerably less expensive than some other polymers. PEI is also clean and stable, but is not particularly resistant to chemicals or solvents ... it has outgassing values of 0.40% TML, 0.00% CVCM and 0.06 % WVR. PEI has good mechanical properties up to approximately 410°F.

• Semitron® ESd 410C (filled PEI) http://www.boedeker.com/sem410\_p.htm

Semitron® ESd 410C is antistatic/conductive PEI. This material is relatively clean, readily machinable, dissipates static electricity reliably ... as a result it is used in test handling equipment, fixtures, and other applications where static generation may cause failures and/or errors in production environments. This material has outgassing values of 0.46% TML, 0.00% CVCM and 0.17 % WVR. PEI has good mechanical properties up to approximately 340°F.

#### • Ertalyte® PET-P (Polyethylene Terephthalate) http://www.boedeker.com/ertaly\_p.htm

Ertalyte® offers the dimensional stability of acetal with the wear resistance of nylon. Ertalyte® PET-Polyester is clean, chemically resistant, stable, and also has relatively low outgassing values (0.13% TML, 0.00% CVCM ... % WVR is not shown). PET-P is considerably less expensive than most of the other materials listed above, but may not have the mechanical or thermal performance needed for all applications.

#### • Semitron® ESd 225 (filled acetal) http://www.boedeker.com/sem225\_p.htm

Semitron® ESd 225 is antistatic/conductive acetal. This material is relatively clean, readily machinable, dissipates static electricity reliably ... as a result it is used in test handling equipment, fixtures, and other applications where static generation may cause failures and/or errors in production environments. This material has low outgassing values of 1.00% TML, 0.05% CVCM and 0.60 % WVR. Acetal has good mechanical properties up to approximately 180°F.

# Appendix 3 – Materials for use in Vacuum

The choice of materials is limited for vacuum systems to a range that has little impact on vacuum production and ultimate base vacuums.

#### 1.1 Metals

- <u>Stainless Steel</u> is used for the majority of vacuum system designs due to its:
  - o Durability
  - Surface finishing
  - Ability to withstand moderate temperatures for system bake out
  - Low magnetic susceptibility
  - Low thermal conductivity
  - Resistance to oxidisation
  - Low vapour pressure

Type 304 or 316 stainless steel is ideally suited for vacuum vessel construction because of its machining/welding characteristics, excellent corrosion resistance and overall cost effectiveness.

Not all stainless alloys are acceptable. Free-machining 303 steel contains sulphur, which tends to outgas. Alloys with good weldability using TIG or MIG welding are usually chosen.

- 304 or 316 stainless steel is a common choice of a stainless steel.
- $\circ~$  304L stainless steel, a low-carbon variant of 304 steel, is used for ultra-high vacuum systems.
- 347 stainless steel does not accept high polish.
- $\circ$  321 stainless steel is chosen when low magnetic permeability is needed.
- <u>Mild steel</u> is okay for low to moderate vacuums above 10<sup>-4</sup> Pa. Outgassing can be reduced with suitable plating such as nickel. It has high permeability to hydrogen and tendency to rust. Mild steel must not be used for any components or chambers used near the bending magnets
- <u>Aluminium alloys</u> are easily machined and have a low vapour pressure, unless the alloys contain high proportion of zinc. 6061 grade is good for general use. Components used in high vacuums or better must not be anodized, as the oxide layer can trap water vapour and outgas. Aluminium and its alloys have low strength at high temperatures, distort when being welded, and the copper-containing ones are poorly weldable. Aluminium wire rings can be used as cheap gaskets in demountable seals. Soft alloys must be used to ensure wire gaskets do not damage the seal faces. Aluminium has high thermal conductivity, good corrosion resistance, and low solubility of hydrogen. Loss of strength at high temperatures limits its use in bakeable applications, but aluminium is advantageous for large-size systems due to its lower weight and lower cost than stainless steel. Aluminium is not recommended for vacuum chambers.
- <u>Brass</u> is not acceptable for vacuum chambers however it may be suitable for some small beam line inserts for specific applications. Once used widely but copper can cause problems with neutron production if struck by proton beams of particular energies. Brass is for high thermal conduction applications in cooling baffles or sample mounts that may be heated. Although bare

brass has good corrosion resistance the zinc content may cause outgassing problems. This can be reduced by plating with nickel.

- <u>Nickel</u> is widely used in vacuum technology, e.g. as mechanical parts in vacuum tubes. It is relatively low-cost, can be spot welded, can be easily machined, has high melting point and is resistant to many corrosive fluids and atmospheres. Its potential drawback is its ferromagnetism, which restricts applications that would be influenced by magnetic fields.
- <u>Beryllium</u> is used primarily for x-ray windows.
- <u>Oxygen-free copper</u> is okay for high vacuum but it is difficult to outgas completely. Copper is insensitive to hydrogen and impermeable to hydrogen and helium, has low sensitivity to water vapour, but is attacked by mercury. Although, oxygen-free copper is widely used as it is easily machined and has good corrosion resistance. It is unsuitable for bakeable vacuum envelopes due to its tendency to oxidize and create scales. Conflat flange seals are made from copper. Its strength falls sharply above 200 °C. Its vapour pressure becomes significant at above 500 °C.
- <u>Indium</u> wire is used as a gasket in demountable seals. Not suitable for high temperature above  $\sim 100^{\circ}$ C applications.
- <u>Gold</u> wire is used as a gasket in demountable seals for ultra-high vacuum.
- <u>Tantalum</u> is acceptable in vacuum systems but it is difficult to work with due to its hardness. It is expensive so it is usually purchased in sheet form. Typically mounted as a shield where ion beams contact a surface as it has a low production of secondary electrons. Good for apertures and defining slit faces and anywhere that a metal edge is used to intersect the ion beam.
- <u>Zirconium</u> is corrosion-resistant. Like tantalum, it has low production of secondary electrons, so it is used as a shield of areas where reducing their production is important. It is used for neutron windows. It is expensive and rarekly used. Zirconium and zirconium hydride are used for gettering.

#### **1.2 Plastics**

Plastics or ceramics are not to be used as the primary vacuum chamber structure without engineering advice from ANSTO or a specialist scientific vacuum instrument company. Plastic is very good for electrical insulators, bushes or light weight components are necessary in vacuums.

- <u>Polytetrafluoroethylene</u>, PTFE or Teflon as it is commonly known is suitable for use inside of vacuum systems from low to ultra high vacuums. Being soft PTFE can flow (creep) so for applications such as sample positioning systems or other systems requiring high stability then other plastics may need to be considered. PTFE has be far the highest dielectric strength, for extruded PTFE, 19.7 kV/mm 60 173 kV/mm for an insulating film. This compares with air which is around 1 kV/mm. It is self-lubricating, tolerant to fairly high temperatures, and has low outgassing. It is not suitable for barrier between vacuum and atmosphere, due to its permeability.
- <u>Ertalyte</u> offers the dimensional stability of acetal with the wear resistance of nylon. It is stable with relatively low outgassing values and has very low water absorption. It is very good for small mechanical loads requiring electrical isolation in vacuum systems.

- <u>Polyvinyl Chloride (PVC)</u> is acceptable in vacuum systems but not where heat is above 50°C. It is usually an amorphous thermoplastic material with excellent chemical resistance and dielectric properties, good tensile, flexural and mechanical strength, low moisture absorption, exceptional dimensional stability and good flammability characteristics.
- Other plastics can be used in vacuum systems but must have low vapour pressure. Care must be taken to ensure they are not used in applications that may require temperatures that will cause high outgassing rates.
- <u>Perspex</u> (acrylic plastic) is another plastic which has good dielectric properties but a high vapour pressure especially at raised temperatures making it unsuitable in vacuums however it can be used in small amounts in a well pumped high vacuum system.
- <u>Vespel</u>, a polyimide, is very expensive, but machines well, has good electrical insulator properties and is compatible with ultra-high vacuum. It does however absorb moisture and requires a longer pump down time. It also performs well in extremely low cryogenic temperatures. Good for bushes or where a mechanically stable plastic is required.
- <u>Nylon</u> is self-lubricating but has high outgassing rate and relatively high water absorption.
- <u>Polycarbonates and polystyrene</u> are good electrical insulators with moderate outgassing.
- <u>Mylar</u> is used to make thin windows that allow ion beams to penetrate from vacuum to positive pressures with little energy loss. For example gas detectors mounted onto beam lines for AMS have gas inside of the detector separated from the beam line which is under vacuum. Ion beams pass through the window as they move from the vacuum side to the positive pressure side. Other materials in this category include Kapton which is rated for higher temperatures.

#### **1.3 Elastomers**

Some elastomers have sufficient vacuum properties and are used widely as vacuum seals in the form of 'o' rings.

- <u>Viton</u> is the standard seal used throughout the accelerator area as it is long lasting, low vapour pressure as compared with other elastomers and is bakeable to 200 °C.
- <u>Nitrile</u> rubber is used for vacuum seals. Does tend to break down over time. First signs of breakdown are small splits. If used for vacuum seals then they must be inspected regularly.
- <u>Natural</u> rubber is not typically used in vacuum systems other than inside of vacuum pumps usually on the low vacuum side of the system.
- <u>Silicone</u> rubber is not generally used as a seal in high vacuum environments. Silicone is soft when compared with other elastomers. It has a relatively low vapour pressure.

Further reading: Materials for high vacuum technology: an overview, S. Sgobba, CERN, Geneva, Switzerland. http://cdsweb.cern.ch/record/983744/files/p117.pdf

#### 1.4 Ceramics and glass

• <u>Alumina</u> ceramic based ceramics in vacuum systems perform well provided the ceramic is not porous which can trap gas bubbles. Ceramics are readily available bonded onto metal flanges or mounting plates. Ceramic can also be baked if necessary to speed up the outgassing of surfaces.

Glass is similar to ceramics in this application. It is very low vapour pressure but difficult to work with. Glass viewports can be purchased already mounted in a metal flange.

- <u>Borosilicate glass</u> is often used for smaller assemblies and for viewports.
- <u>Porcelain</u> ceramics, when fully vitrified and therefore non-porous, are excellent insulators usable to 1500 °C. it is generally commercially available bonded to metal in electrical insulators and other feedthroughs.
- <u>Mica</u>, although it is neither ceramic or glass it best fits in this category. Mica has been used in vacuum systems for both electrical and thermal insulation but because Mica is a series of laminations it contains trapped air which is not suitable in high to ultra-high vacuums.
- <u>Macor</u> is a machinable glass made by Corning Incorporated with similar properties to ceramics. It has a low vapour pressure and thermally stable up to 1000°C making it bakeable. It is made from mica and borosilicate glass. It is by far the most flexible of the glass-ceramic options.

 Leakage Testing Handbook, Prepared for Liquid Propulsion Section, Jet Propulsion Laboratory, National Aeronautics and Space Administration, Pasadena, California
 Nondestructive Testing Handbook, Volume One, Leaktesting, American Society for Nondestructive Testing.
 Leakage Testing Handbook, Revised Edition, July 1969, General Electric.
 Fluid Flow in Small Passages, Mars Hablanian, J.W.Marr, Varian

#### 1.5 Greases and oils

There are few acceptable greases and oils for use in vacuum systems. Users must ensure they purchase greases and oils specifically designed for use in vacuums. Typically they will have low vapour pressures, extremely low water absorption and easy to remove for cleaning.

- <u>Apiezon family</u> For further information http://www.apiezon.com/ . These have been used throughout the history of accelerators at ANSTO with excellent results. Wherever greases are used remember to use the minimalist amount.
- <u>Greases</u>
  - Apiezon H High Temperature Vacuum Grease, silicone and halogen free
  - Apiezon L Ultra high vacuum grease, silicone and halogen free
  - Apiezon M High vacuum grease, silicone and halogen free
  - Apiezon N Cryogenic high vacuum grease, silicone and halogen free
  - Apiezon T Medium temperature grease, silicone and halogen free
  - o Apiezon AP100 Ultra High Vacuum Lubricating Grease, silicone free
  - o Apiezon AP101 Anti seize Vacuum Grease, silicone free
- <u>Waxes</u>
  - Apiezon® Waxes and Compounds
  - Apiezon Wax W (Hard Vacuum Sealing)
  - Apiezon Wax W40 (Softer Vacuum Sealing)
  - Apiezon Wax W100 (Softest Vacuum Sealing)

- Apiezon Sealing Compound Q
- <u>Vacuum Oil</u> is used in few applications around the accelerator area. With a change to oil free pumps most oil consuming pumps are redundant. However these types of pumps may still in use in other applications. Oil vane roughing pumps and diffusion pumps were the largest consumers of oil. More recent brands of vane pumps were specifically tuned to oils of particular specifications so generic oils may not be compatible. Diff pumps are more forgiving and high quality generic synthetic oils may be used. The list below is a small sample of what is available and previously used.
  - $\circ\,$  Shell Vitrea 68 oil for roughing pumps. Check specification on pump to ensure compatibility with this mineral oil
  - Fomblin PFPE (Perfluorpolyether) oils designed for rotary pumps. Check specification on pump to ensure compatibility with this mineral oil
  - Apiezon® Diffusion Pump Fluids Apiezon AP 201 The Apiezon diffusion pump fluid is known for its exceptional higher degree of oxidation resistance.

# **Appendix 4 – Surface finishing**

In most cases a clean polished surface will yield the least outgassing as there are minimal surface traps for gas. Also, a polished surface is easier to clean and keep clean. Achieving a polished surface may not be practicable due to accessibility into tubes, chambers and small components but a combination of other methods may provide an adequate surface finish that will allow the desired vacuum level to be reached. The best method for a given surface will depend on the material, geometry and accessibility to its surfaces.

- <u>Polishing</u> A polished surface may be either smooth or uneven but the metal will lustre in either case. Polishing is a method of removing a layer of metal to expose clean metal underneath. This can be done with a mechanical buff or by hand with the aid of a polishing compound. Care must be taken to ensure all traces of the polishing compound are removed during final cleaning. All residues must be removed.
- <u>Grinding, sanding and other coarse to fine abrasive methods</u> These methods tear the surface dragging metal over metal. Some metal breaks away exposing clean metal underneath. The problem with this type of surface preparation is that the metal can fold over and not break away leading to voids of trapped gases. It is important that a fine grit is the last grit used to ensure larger metal drifts on the surface are torn away.
- <u>Grit blasting with grit or beads</u> This method propels grit or beads onto the metal surface causing fragments of metal to be removed with the impact. Like the abrasive techniques metal can be layered on metal (peening) if the grit is too coarse. Best to finish with a fine grit then a mechanical polish. Grit blasting can provide a more uniform surface finish and is very good for removing scale that may be present from rolling or extrusions.
- <u>Pickling</u> by far a very good way to clean welds and other heat induced decolourisations in the metal. It will remove a thin layer of metal, scale, oils, etc. Care must be taken to ensure any chemicals used are neutralised and thoroughly cleaned away. Residues cannot be tolerated. Makes general maintenance of the surface easier.
- <u>Passivating</u> a form of chemical polishing and surface protection. Can be stimulated with an electric current. Passivating is the spontaneous formation of a hard non-reactive surface film that inhibits further corrosion. It isn't a preferred method of surface preparation in vacuum systems as damaged surfaces can form micro cracks trapping water and other gases. For example, damage to anodised aluminium may allow aluminium oxide to form which can readily trap water. Passivation is not typically used for stainless steel as the surfaces are self healing, provided sufficient oxygen is available.

Further reading on surface passivation can be found at: http://www.euro-inox.org/pdf/map/Passivating\_Pickling\_EN.pdfGetter surfaces

• Nickel Plating – has been used on vacuum components to provide stability to the surface of normally oxidising metals such as steel, copper and brass. It is important to ensure that the nickel has uniform bonding to the parent metal. Cases have been observed where the plating has lifted creating voids in the vacuum space. Although nickel is resistant to corrosion it will form a thin layer if exposed to moist atmospheres. A periodic clean is recommended to minimise problems with corrosion.

Element	Melting Point °C	Critical Temp °C	Phase at Critical T
Aluminium	660	1124	Liquid
Americium	1176	896	Solid
Antimony	631	455	Solid
Arsenic	84	270	Liquid
Barium	729	574	Solid
Beryllium	1289	1102	Solid
Cadmium	321	226	Solid
Caesium	29	114	Liquid
Calcium	847	538	Solid
Californium	900	1402	Liquid
Cerium	798	1602	Liquid
Chromium	1863	1220	Solid
Cobalt	1492	1418	Solid
Copper	1084	1146	Liquid
Dysprosium	1412	1025	Solid
Erbium	1529	1139	Solid
Europium	822	547	Solid
Gadolinium	1312	1450	Liquid
Gallium	30	954	Liquid
Germanium	938	1260	Liquid
Gold	1064	1291	Liquid
Hafnium	2231	2255	Liquid
Holmium	1474	1146	Solid
indium	157	836	Liquid
Iridium	2447	1199	Solid
Iron	1538	1253	Solid
Lanthanum	918	1586	Liquid
Lead	328	622	Liquid
Lithium	181	465	Liquid
Lutetium	1663	1517	Solid
Magnesium	651	386	Solid
Manganese	1246	887	Solid
Mercury	-39	22	Liquid
Molybdenum	2623	2319	Solid
Neodymium	1021	1219	Liquid
Neptunium	639	1781	Liquid
Nickel	1455	1409	Solid
Niobium	2468	2501	Liquid
Osmium	3033	2721	Solid
Palladium	1555	1348	Solid
Platinum	1769	1800	Liquid
Plutonium	640	1506	Liquid
Potassium	64	165	Liquid
Praseodymium	931	1381	Liquid
Protactinium	1572	2636	Liquid
Rhenium	3186	2817	Solid
Rhodium	1963	1874	Solid
Rubidium	39	129	Liquid

# **Appendix 5 – Critical Vapour Pressures**

Substance	Vapour Pressure (Pa)	Temperature (°C)	
Acetaldehyde	98.7	20	
Acetone	24640	20	
Butane	22	20	
Carbonyl sulphide	1255	25	
Ethanol	5.83	20	
Ethylene glycol	0.5	20	
Formaldehyde	435.7	20	
Freon 113	37.9	20	
Methanol	12800	20	
Methyl isobutyl ketone	26.48	25	
Nitrogen (N <sub>2</sub> )	63200	20	
Oxygen (O <sub>2</sub> )	54200	20	
Propane	2200	55	
Propanol	2.4	20	
Tungsten	0.1	3203	
Water (H <sub>2</sub> O)	2.3	20	
Xenon difluoride	0.6	25	

# **Appendix 5 – Vapour Pressures continued**

Substance	Vapour Pressure (Pa)	Melting point (°C)
Apiezon Oil J	0.13 at 200°C 1.3 x 10 <sup>-4</sup> at 20°C	-
Apiezon Oil K	0.13 at 300°C 1.3 x 10 <sup>-7</sup> to 10 <sup>-8</sup> at 20°C	-
Apiezon Grease L	0.13 at 100°C 1.3 x 10 <sup>-7</sup> to 10 <sup>-9</sup> at 20°C	47
Apiezon Grease M	0.13 at 200°C 1.3 x 10 <sup>-5</sup> to 10 <sup>-6</sup> at 20°C	44
Apiezon Grease N	0.13 at 200°C 1.3 x 10 <sup>-6</sup> to 10 <sup>-7</sup> at 20°C	43
Apiezon Grease T	About 1.3 x 10 <sup>-6</sup> at 20°C	125
High Vacuum Grease Dow Corning	< 1 x 10 <sup>-4</sup> at 20°C	-
Nylon	~ 1 x 10 <sup>-3</sup> at 20°C	-
Teflon	< 1 x 10 <sup>-4</sup> at 20°C	-
Viton	1 x 10 <sup>-6</sup> at 20°C	-

# Appendix 6 – Outgassing tables for various materials Reference site: http://home.fnal.gov/~mlwong/outgas\_rev.htm

		Note the different methods of measurement and treatment of samples.					
Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	(hours)	Test method	Reference	Year	
Aluminium	None	1x10 <sup>-6</sup>	1h		Schamus (ref Markley, et al)	1999	
Aluminium	Degassed	$1.7 \times 10^{-7}$	1h		Schmaus (ref Markley, et al)	1999	
Aluminium	Degassed	2.7x10 <sup>-8</sup>	10h		Schmaus (ref Markley, et al)	1999	
Aluminium 6061-T6	Baked 13.5h @ 300°C	1.4x10 <sup>-8</sup>	10h		Schmaus (ref Das)	1999	
Aluminium	Cleaned	8x10 <sup>-9</sup>	10h		Schmaus (ref Blears, et al)	1999	
Aluminium	Fresh, degreased w/ trichloroethylene & cleaned w/ ethyl alcohol	6.3x10 <sup>-9</sup>	1h	conductance	Elsey (ref Schram)	1975 (1963)	
Aluminium	Degassed	$4.6 \times 10^{-9}$	100h		Schmaus (ref Markley, et al)	1999	
Aluminium 6061-T6	Baked @ 200°C	4.5x10 <sup>-9</sup>	10h		Schmaus (ref Das)	1999	
Aluminium	Degassed 24h, degreased w/ trichloroethylene & cleaned w/ ethyl alcohol	4.14x10 <sup>-9</sup>	1h	conductance	Elsey (ref Schram)	1975 (1963)	
Aluminium 6061-T6	None	2.5x10 <sup>-9</sup>	10h		Schmaus (ref Das)	1999	
Aluminium 5083-O	Bell jar, as received, room temp.	2.18x10 <sup>-9</sup>	10.3h	Rate-of-rise	Schrank, et al	1964	
Aluminium 5083-O	Bell jar, as received, room temp.	1.27x10 <sup>-9</sup>	8h	Conductance	Schrank, et al	1964	
Aluminium 5083-O	Bell jar, as received, room temp.	6.6x10 <sup>-10</sup>	24h	Conductance	Schrank, et al	1964	
Aluminium 5083-O	Bell jar, as received, room temp. after baking @ 220°C	$4.6 \times 10^{-10}$	50h (baked @27-31h)	Conductance	Schrank, et al	1964	
Aluminium 6061-T6	Baked 15h @ 300°C	$1.6 \times 10^{-10}$	10h		Schmaus (ref Das)	1999	
Aluminium	Degassed 24h, degreased w/ trichloroethylene & cleaned w/ ethyl alcohol	3.06x10 <sup>-10</sup>	1h	conductance	Elsey (ref Schram)	1975 (1963)	
Aluminium 5083-O	Bell jar, no. 4 finish (lapped), room temp.	$2.87 \times 10^{-10}$	6.0h	Rate-of-rise	Schrank, et al	1964	
Aluminium	Fresh, degreased w/ trichloroethylene & cleaned w/ ethyl alcohol	6.0x10 <sup>-10</sup>	10h	conductance	Elsey (ref Schram)	1975 (1963)	
Aluminium, type 1100	Cleaned w/ detergent, rinsed w/ acetone, pumped 24 hours	~10 <sup>-10</sup>	0	conductance	Young	1968	
Aluminium	LEP vacuum chamber, chem clean, baked in situ @ 150°C	$2.3 \times 10^{-11}$	24h		Mathewson, et al	1988	
Aluminium 6061-T4	Degreased in acetone w/ methanol rinse; baked 100°C	6x10 <sup>-12</sup>	24h	Rate-of-rise & conductance	Halama, Herrera	1976	

Outgassing rates of aluminium Note the different methods of measurement and treatment of samples.

Aluminium 6061-T4	Degreased in acetone w/ methanol rinse, chem polished, baked 100°C	3x10 <sup>-12</sup>	24h	Rate-of-rise & conductance	Halama, Herrera	1976
Aluminium 6061-T4	Degreased in acetone w/ methanol rinse, chem polished, glow discharge in Ar, baked 100°C	5x10 <sup>-13</sup>	24h	Rate-of-rise & conductance	Halama, Herrera	1976
Aluminium, type 1100	Above plus baked 15h @ 250°C under vacuum	$4x10^{-13}$	24h @ room temp	conductance	Young	1968
Aluminium	PETRA vacuum chamber, glow discharge@145°C	$1 \times 10^{-13}$	Up to 200h	In situ glow discharge, conductance	Mathewson, et al	1977

# Outgassing rates of stainless steels, along with the surface treatment and pumping time.

Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	Time (hours)	Test method	Reference	Year
Stainless steel	None	6.4x10 <sup>-7</sup>	1h		Schamus (ref Markley, et al)	1999
Stainless steel	degreased	4x10 <sup>-7</sup>	1h		Schamus (ref Markley, et al)	1999
Stainless steel	None	2x10 <sup>-7</sup>	1h		Schamus (ref Blears, et al)	1999
Stainless steel	None	2x10 <sup>-8</sup>	10h		Schamus (ref Blears, et al)	1999
Stainless steel NS22S	Fresh	1.4x10 <sup>-8</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel ICN 472	fresh	1.3x10 <sup>-8</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel	None	1.2x10 <sup>-8</sup>	10h		Schamus (ref Das, et al)	1999
Stainless steel ICN 472	sanded	8.3x10 <sup>-9</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel NS22S	electro polished	4.3x10 <sup>-9</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel NS22S	mechanically polished	1.7x10 <sup>-9</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel	fresh	1.5x10 <sup>-9</sup>	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel ICN 472	Polished & vapor degreased	1.4x10 <sup>-9</sup>	10h		Schamus (ref Dayton, et al)	1999
Stainless steel	None	1.4x10 <sup>-9</sup>	10h		Schamus (ref Das, et al)	1999
Stainless steel NS22S	fresh	1.3x10 <sup>-9</sup>	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel ICN 472	sanded	1.0x10 <sup>-9</sup>	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel	None	7.6x10 <sup>-10</sup>	1h		Schamus (ref Das, et al)	1999
Stainless steel NS22S	mechanically polished	$4.6 \times 10^{-10}$	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel NS22S	electro polished	$4.3 \times 10^{-10}$	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel	Baked 24h @ 200°C	$1.5 \times 10^{-10}$	1h		Schamus (ref Das, et al)	1999

Stainless steel	None	$1.1 \mathrm{x} 10^{-10}$	100h		Schamus (ref Das, et al)	1999
Stainless steel 304	Degrease + water rinse	$4.0 \times 10^{-11}$	40h	Conductance	Strausser	1973
Stainless steel U15C	Baked 25h @ 300°C	$4.5 \times 10^{-12}$			Elsey (ref Calder, et al)	1975
Stainless steel 304	Degrease + water rinse, baked in vacuum 150°C for 12h	4.0x10 <sup>-12</sup>	5h after bakeout	Conductance	Strausser	1967
Stainless steel 304	Electropolished, baked 30h @ 250°C	$3.0 \times 10^{-12}$			Elsey (ref Young)	1975
Stainless steel U15C	Baked 45h @ 360°C	$2.6 \times 10^{-12}$			Elsey (ref Calder, et al)	1975
Stainless steel	Baked 24h @ 200°C	$9.3 \times 10^{-13}$	100h		Schamus (ref Das, et al)	1999
Stainless steel U15C	Baked 3h in vacuum @ 1000°C + 25h in situ @ 360°C	1.6x10 <sup>-14</sup>			Elsey (ref Calder, et al)	1975

Other cleaning methods although outgassing rates are not measured:

Material	Treatment	Results	Reference	Year
Aluminium	Quench a hot aluminium extrusion in an Ar- $O_2$ atmosphere	Dense, thin (~20Å thick) oxide layer	Sasaki, Y.T.	1990
Aluminium 6063	Clean with an alkaline detergent (Almeco 18)	Removes MgO, C, and Al <sub>2</sub> O <sub>3</sub>	Sasaki, Y.T.	1990
Aluminium	Vapour degreasing, pump for 120h before and after bake	Dominant residual gases: H <sub>2</sub> O, H <sub>2</sub> , CO, CO <sub>2</sub> , & CH <sub>4</sub> ; after baking @ 150°C and pumping, H <sub>2</sub> O & CH <sub>4</sub> gone, outgassing rates of other gases reduced 1 order of magnitude except H <sub>2</sub>	Mathewson, A.G., et al	1989
Aluminium	Vapour degreasing, then light alkaline etch (CERN basic procedure), pump for 120h before and after bake	Dominant residual gases: H <sub>2</sub> O, H <sub>2</sub> , CO, & CO <sub>2</sub> ; after baking @ 150°C and pumping, only H <sub>2</sub> exists	Mathewson A.G., et al	1989
Aluminium	Strong alkaline etching in NaOH, pump for 120h before and after bake	Dominant residual gases: H <sub>2</sub> O, H <sub>2</sub> , CO, CO <sub>2</sub> , & CH <sub>4</sub> ; after baking @ 150°C and pumping, only H <sub>2</sub> exists	Mathewson A.G., et al	1989
Aluminium	Vapour degreasing, Ar glow discharge @ 150°C	On initiation of discharge, large pressure increase (normal in Ar glow discharge) was too large for accurate measurement	Mathewson A.G., et al	1989
Aluminium	Vapour degreasing, then light alkaline etch (CERN basic procedure), Ar glow discharge @ 150°C	H <sub>2</sub> , CO dominant gases desorbing from surface during discharge cleaning	Mathewson A.G., et al	1989
Aluminium	Strong alkaline etching in NaOH, Ar glow discharge @ 150°C	H <sub>2</sub> , CO dominant gases desorbing from surface during discharge cleaning at order of magnitude less than light alkaline etch	Mathewson A.G., et al	1989
SS	H <sub>2</sub> glow discharge - 22°C	Dominant residual gases: $CH_4$ , $CO$ , & $C_2H_4$ ; water production insignificant; frequent arcing	Dylla, H.F.	1988

		on all exposed surface noted during initial discharge period		
SS	$H_2$ glow discharge – 150°C	Dominant residual gases: $CH_4$ , $CO$ , & $C_2H_4$ , and $H_2O$ ; frequent arcing on all exposed surface noted during initial discharge period	Dylla, H.F.	1988
SS	Ar glow discharge – ambient temp	Dominant residual gases that were significantly removed: $CO_2$ , $H_2$	Dylla, H.F.	1988
SS	Ar/O <sub>2</sub> glow discharge	Improved removal of CO and CO2; minor amts of implanted Ar that can be removed by baking @ 350°C	Dylla, H.F.	1988
	Ar/ $O_2$ glow discharge 2h, exposed to air, then Ar/ $O_2$ glow discharge 2h	Some degree of passivation: residual gases were removed with 1 <sup>st</sup> cleaning & did not show up during the 2 <sup>nd</sup> cleaning	Dylla, H.F.	1988
	O <sub>2</sub> glow discharge	Rapid removal of carbon, not of hydrocarbons (which can be baked out initially), increased surf. Oxidation, sputtering of the base metal, and possible enhanced outgassing of O <sub>2</sub> - containing molec.	Dylla, H.F.	1988

#### Different outgassing measurements of epoxies

Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	Time (hours)	Test method	Reference	Year
NMA w/ solid alumina filler	Cured 300°F	$1.6 \times 10^{-7}$			Hanson, Patel	1970
Polyimide polymer	Thermosetting; measured @ 40°C	~1.1x10 <sup>-7</sup>	4h+9min	conductance	Kendall, Zabielski	1965
Polyimide polymer	Thermosetting; measured @ 155°C	~5x10 <sup>-8</sup>	4.5h increase temp + 6h	conductance	Kendall, Zabielski	1965
Silicone resin	Needs dicumyl peroxide catalyst; thermosetting, measured @ 35°C	~5x10 <sup>-8</sup>	4h	conductance	Kendall, Zabielski	1965
Araldite epoxies	Cured ~150°C for 15h	~10 <sup>-8</sup>	51h pumping	conductance	Barton, Govier	1965

# Kapton is a newer material and little data exists about its outgassing rate. Below are 3 references

Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	Time (hours)	Test method	Reference	Year
Kapton cable		1.0x10 <sup>-5</sup>	0h	conductance	Postma	1999
Kapton foil		$1.0 \times 10^{-7}$	40h		Ferro-Luzzi	1999
Polyimide	Bake @ 300°C	4.0x10 <sup>-8</sup>	12h		Weston	1970
Kapton cable		$2.4 \times 10^{-8}$	168h	conductance	Postma	1999
Kapton cable		2.7x10 <sup>-9</sup>	336h	conductance	Postma	1999
Kapton cable		$6.5 \times 10^{-10}$	504h	conductance	Postma	1999

#### Outgassing rates of several types of plastics

Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	Time (hours)	Test method	Reference	Year
Nylon		1.2x10 <sup>-5</sup>	1h		Elsey (ref	1975
					Power, et al)	
Nylon		6.0x10 <sup>-6</sup>	1h		Elsey (ref	1975

					Power, et al)	
PVC	24h @ 95% relative	8.5x10 <sup>-7</sup>	1h	conductance	Elsey (ref	1975
	humidity				Santler)	
PTFE	Fresh	$1.7 \mathrm{x} 10^{-7}$	1h	conductance	Elsey (ref	1975
					Santler)	
Teflon		6.5x10 <sup>-8</sup>	1h	conductance	Elsey (ref	1975
					Santler)	
PTFE	Fresh	3.3x10 <sup>-8</sup>	10h	conductance	Elsey (ref	1975
					Santler)	
Teflon		$2.5 \times 10^{-8}$	10h	conductance	Elsey (ref	1975
					Santler)	
PVC	24h @ 95% relative	$2.0 \times 10^{-8}$	10h	conductance	Elsey (ref	1975
	humidity				Santler)	
G-10		~10 <sup>-8</sup>			Beams	2001
					Division -	
					FNAL	

Reference to the following has been lost but it is believed to have originated in an early Vacuum Catalogue from the 1990's

 $K_1$  is the outgassing rate (air equivalent) after 1 hour pumping  $\alpha_1$  is the slope of the (log K - log t) curve at 1 hour

Material	K <sub>1</sub> Torr litre/sec cm <sup>2</sup> (x 10 <sup>-10</sup> )	α1	K <sub>10</sub> Torr litre/sec cm <sup>2</sup> (x 10 <sup>-10</sup> )	a <sub>10</sub>
Aluminium Alloy (fresh)	63	1	6.0	1
Aluminium Alloy (degassed 24 hours)	41.4	3.2	3.06	0.9
Aluminium Alloy (3 hours in air)	65.5	1.9	4.75	0.9
Aluminium Alloy (anodised -2µ pores)	2760	0.9	322	0.9
Aluminium Alloy (bright rolled)	-	-	75	1
Duralumin	1700	0.75	350	0.75
Brass (wave guide)	4000	2.0	100	1.2
Copper (fresh)	400	1	41.5	1
Copper (mechanical polish)	35	1	3.56	1
OFHC Copper (fresh)	118	1.3	12.6	1.3
OFHC Copper (mechanical polish)	19	1.1	1.63	1.1
Gold (fresh wire)	1580	2.1	5.1	1
Mild steel	5400	1	500	1
Mild steel (slightly rusty)	6000	3.1	130	1
Cr plated steel (fresh)	70.5	1	6.8	1
Cr plated steel (polished)	91	1	8.0	1
Ni plated steel (fresh)	42.4	0.9	4.84	0.9
Ni plated steel	27.6	1.1	2.33	1.1
Chemically Ni plated steel (fresh)	83	1	7.05	1
Chemically Ni plated steel (polished)	52.2	1	4.6	1
• • • • •				

Araldite (moulded)	116	0.8	35.2	0.8
Araldite D	800	0.8	220	0.78
Araldite D	190	0.3	125	0.5
Araldite F	150	0.5	73	0.5
Celluloid	860	0.5	430	0.5
(PTFE) (fresh)	16.6	0.8	3.31	0.9
Kel-F Oak ridge	4	0.57	1.7	0.53
Methyl methacrylate	420	0.9	140	0.57
Mylar V-200 (24 hr at 95% RH)	230	0.75	40	-
Nylon	1200	0.5	600	0.5
Pertinax	620	0.18	290	0.5
Perspex	72	0.44	27	0.44
Perspex	310	0.4	180	0.4
Polyamid	460	0.5	230	0.5
Polyester - glass laminate	250	0.84	80	0.81
Polyethylene	23	0.5	11.5	0.5
Polystyrene	2000	1.6	200	1.6
Polystyrol	56	0.6	12	0.61
Polyvinylcarbazol	160	0.5	80	0.5
PTFE	30	0.45	15	0.56
P.V.C. (24 hr at 95% RH)	85	1.00	2	-
Teflon	6.5	0.6	2.5	0.2
Terephenil (fresh)	62.2	0.5	16.8	0.5
Neoprene	3000	0.4	-	-
Viton	114	0.8	-	-
	1	1		

# **Appendix 7 – Local Plant and Safety Regulations**

Regular maintenance inspections are carried out on ANTARES and STAR accelerator vacuum equipment to ensure operational status, vacuum quality, vacuum pump performance, and the mechanical and electrical integrity of the systems.

Generally, the integrity of the vacuum systems can be measured as a direct function of the known static vacuum pressures in a given vacuum system. That is, a lowering of vacuum from the known average vacuum pressure in a given system will indicate either/and, a leak in a seal, a failing vacuum pumping system or a structural failure.

The following is an overview of the application of the ANSTO OHSE Plant and Safety Standard, AS 2309, to vacuum systems in the accelerator facilities. *Reference: http://docushare.ansto.gov.au/Get/File-32340/Plant\_Safety\_Standard.pdf* 

Risk	Hazard	Risk rating	Controls	Risk rating (with controls in place)
Catastrophic failure vacuum vessels	Implosion – scattering of material fragments and loose fittings becoming missiles	Very low	<ul> <li>Use of vacuum standards to design equipment for safe usage.</li> <li>Protective barriers around equipment and fittings that may be susceptible to knocking</li> </ul>	Very low
Cotostrophia failura	• Implosion – scattering of material fragments and loose fittings becoming missiles	Very low	<ul> <li>Use of vacuum standards to design equipment for safe usage.</li> <li>Purchased equipment from reputable companies/designers only</li> </ul>	Very low
catastrophic failure vacuum beam lines and associated equipment	• Electric shock from fault in high vacuum gauge	Very low	• Vacuum gauges using high voltages in key areas will automatically switch off at low vacuums/atmospheric pressure	Very low
	• Failure of welded joint	Very low	• System will leak to atmospheric pressure at a rate commensurate with the conductance of the failed joint. No control is deemed necessary.	Very low
Electric shock from a failed vacuum pump or powered fitting	Electric shock from frayed lead or failure of earth inside of equipment	Low	<ul> <li>All equipment is protected from short circuits by circuit breaker protection.</li> <li>Most circuits now have RCD protection.</li> <li>A program is in place to put all circuits onto RCD protection</li> </ul>	Very Low
Failure of support structures	Crush, fall, bump, trip hazards during and after failure	Low	<ul> <li>Use of vacuum standards to design equipment for safe usage</li> <li>Access to equipment at height via approved ladders or dedicated platforms</li> <li>Non acceptance of standing on beam line stands and mounts</li> </ul>	Very Low
breakages to	<ul> <li>Fragments of broken</li> </ul>	LOW	<ul> <li>Barriers around exposed equipment</li> </ul>	LOW

1) Risk assessment of plant – Accelerator Facilities Only

fittings and equipment protruding from vessels and beam lines	<ul> <li>parts become missiles.</li> <li>Cuts to personnel bumping into fittings</li> <li>Secondary injuries including electric shock from exposed wiring</li> </ul>		<ul> <li>that is deemed to be hazardous</li> <li>Access control into accelerator areas restricted to accelerator personnel and visitors who are escorted</li> <li>See electric shock hazard controls above</li> </ul>	
Over pressure of vacuum systems during venting	Positive pressure in beam lines when venting with gases from high pressure storage	Very Low	<ul> <li>Pressure relief valves in use at systems where regular vacuum cycling is necessary</li> <li>Use of portable pressure relief fittings where local venting is required.</li> </ul>	Very Low
Implosion of glass view ports	Shards of glass scattering towards operators	Low	<ul> <li>Use purpose designed commercial vacuum windows</li> <li>Use small diameter (&lt;50mm x 6 mm thick) quartz windows on vacuum systems where ion beams can strike. Quartz is not known to crack through but to develop small surface anomalies</li> <li>Use double rubber seals to support windows to minimise mechanical stresses</li> </ul>	Low
Broken film windows	Sudden venting of vacuum to atmospheric pressure	Very Low	<ul> <li>Damage to equipment – high vacuum gauges must have auto shutdown at low vacuums</li> <li>Vacuum system isolated with gate valve to minimise loss of vacuum in adjoining systems</li> </ul>	Very Low
Breach of accelerator tubes with SF6	Vacuum systems pressurised with SF6 gas	Low	<ul> <li>Large gate valves either end of the accelerator vacuum tubes will shut if pressure rises to maintain SF6 within the vacuum vessels</li> <li>Some gas will enter the vacuum systems outside of the isolated section. The pressure will be relieved through blow off venting ports at each end of the vessel.</li> <li>Oxygen depletion alarms will sound if dangerous levels of SF6 are released. Personnel will be ordered to leave building through PA announcement</li> </ul>	Low

- 2) Risk control measures for eliminating or reducing the assessed risks *See table above*
- 3) Registration or notification of design of plant *Not deemed necessary. See AS 4343-2005*
- 4) Licensing of plant Not deemed necessary. See AS 4343-2005
- 5) Maintenance of plant

*Regular maintenance applied via Accelerator Maintenance system. See ENV-I-076-004 Vacuum Systems Maintenance manual* 

- 6) Plant operations and risk controls *See table above*
- 7) Requirements in relation to specific types of plant *Only vacuum system plant*
- 8) Training and supervision Vacuum technology training is provided in-house to world's best practices. Additional training has been provided by the Vacuum Society of Australia through their training courses.
- 9) Monitoring and evaluation Regular maintenance applied via Accelerator Maintenance system. See ENV-I-076-004 Vacuum Systems Maintenance manual

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# Vacuum Technology

# and

# Vacuum Design Handbook

# for

# **Accelerator Technicians**

Prepared by David Garton November 2011 Revision 0 Vacuum Technology and Vacuum Design Handbook for Accelerator Technicians

This handbook is a compilation of information gathered from over 50 years of direct hands-on experience to applicable information widely available from the vacuum technology industry. It seeks to address common and specific vacuum technology problems whilst clarifying the design standards and philosophies adopted for use in the ANSTO accelerator facilities. The author wishes to thank the reviewers and the many technicians from ANSTO that have contributed directly and indirectly to this booklet.

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# Chapter 1

# Vacuum design standards and good practises for the Accelerator Area

- \* This chapter must be read in conjunction with the detailed vacuum information in this document.
- \* Based on best practises developed at ANSTO and other local Australian accelerator laboratories in conjunction with Australian Standards and industry technical guides, referenced throughout this document.
- \* Where a standard is unavailable for a specific design, best practices are used from industry's best practises.
- \* *Refers to all vacuum chambers, beam lines and other enclosures used on the accelerators or stand alone.*

# 1. Basic design

- All vacuum vessel designs must be consistent with the design standards in Australian Standard AS 1210-2010 and maintain a Hazard Level of E as per the design conditions tabled in "Hazard Levels of Pressure Equipment" in Section 2, Hazard Levels of Pressure Equipment in Australian Standard AS 4343-2005.
  - The vacuum equipment designed by/for the accelerator area maintains a Hazard level of E. All pressure equipment with a pV  $\leq$  30, pressure (p in MPa) x volume (litres)  $\leq$  30 is classed as Hazard level of E. For vacuum equipment in Hazard Level E the derating value of 0.1 is applied, ie 0.1pV  $\leq$ 30. Note the amount of stored energy at this level is very small. For example a Ø 1000 mm chamber, 1000 mm high will have a pV for vacuum equal to 7.85.
  - AS 4343-2005, Section 2.1.2 Typical hazard levels, part (d), states for Hazard Level E, "This equipment is usually exempt from special regulatory control but is <u>covered</u> by general plant safety regulations". See Appendix 7.
- All designs of chambers excluding general beam lines and fittings should be checked for structural integrity using finite elements analysis. Sound designs with acceptable structural integrity will be released for manufacture. A copy of the design acceptance tests must be filed with the project file. The standard finite element analysis can be performed utilising Solidworks. (Modelling for irregular shaped chambers, calculations Appendix 1 where appropriate for regular shapes)
- Stainless steel (304 or 316) will be used to construct chambers, beam lines and general fittings unless it is a *special* requirement to use other metals. Do not choose substances that have high outgassing rates or can contaminate vacuum systems.
- Measurement chambers and other large volume chambers should be cylindrical unless the chamber is an enclosure between pole faces (eg magnet box) or is an odd shape to accommodate steering plates or devices. When non-cylindrical chambers are required deflection of chamber walls should be modelled and taken into consideration.
- Other chambers including magnet boxes, ESA and deflector coffins to have suitable rib strengthened rectangular or curved sides where it is not practicable to increase the wall thicknesses.
- Minimum wall thickness calculation methods can be found in Appendix 1. As a general rule care must be taken when using formulas for vacuum calculations as historically many units other than SI units have been used to form some equations. For example length in cm, and pressure in torr is common in old notes.

- Top and bottom flanges to have integrated structural support rim which becomes the flange and seal for chamber base and top (user) flanges.
- Ports must be a suitable length to ensure unobstructed insertion for screws into flanges. Suitable weld preparation must allow for adequate weld penetration. See Welds below.
- All internal edges must have a smooth radius. No sharp edges to be left after welding or surface finishing.
- All machined finishes other than vacuum seals to be equal to or better than 1.6 microns
- No sections within the vacuum space will have metal to metal contact where gas can be trapped unless it is at the edge of a seal joint. When this is necessary, pump out grooves must be incorporated in the design.
- Vacuum windows shall be made from toughened glass or quartz. No plastic to be used for windows unless the window is a flange on a chamber that is designed to withstand more than 100 kPa. The large plastic flange/windows should be made from >18 mm thick up to Ø200 and >25 mm thick for up to Ø400 in clear Perspex or polycarbonate.
- For ANTARES beam lines the preferred flanges are Dependex, nominal diameter 100 mm. See section 8 for detail on Dependex flanges.
- For STAR there is no preferred flange type. It must be selected depending on the desired base vacuum and interface to other adjoining flanges.
- As a guide, the MDC catalogue from Vacuum Products Corporation 2003 onwards, Building Blocks for Vacuum Science and Technology, provides vacuum tubing dimensions for most common sizes. The dimensions must be assumed to be minimum dimensions. Other tubing sizes should be calculated using the formulas in Appendix 1.
- Vacuum pumping ports leading to the vacuum pumps must be designed to optimise conductance.
- Pressure relief valves to be incorporated where a vacuum system can be vented to >10 kPa above atmospheric pressure.
- ASME state that the design of an external pressure vessel must consider Material type, diameter of chamber, unstiffened length, temperature and wall thickness

# 2. Welds

- TIG is preferred for vacuum sealing welds as minimal scale and flux is produced. Electron beam welding is suitable for thin walled materials. TIG produces high quality welds with or without filler rods to produce flat, smooth well penetrated welds. TIG comes into its own where the weld preparation allows for melting together of the parent materials without filler rods. MIG has a higher deposition of filler material so care must be taken to ensure welds are kept smooth and flat.
- No standard exists for vacuum welding of compact scientific vacuum equipment where pressures approaching zero are necessary. Equipment designed in the accelerator area and welded in the ANSTO workshops since 1990 has had penetrations of ≥1mm but ≤2mm on a butt weld and ≥2 mm on a fillet weld. Experience has demonstrated the adequacy of these figures. No failure reported.
- For thin walled materials ie < 1 mm, such as bellows, manufacturers' will use their industry standard. ANSTO does not weld thin wall materials.
- Weld preparation must be provided where possible to ensure even heat penetration.

- Electric arc with rods is not recommended due to flux trapping. This can cause pits which have long lasting outgassing periods.
- All welds that form a seal against atmospheric or other positive pressures must be made to the internal side (vacuum side) of the joint.
- Tack (stitch) welding is recommended on the external side only with less than 50% of the diameter covered in small intervals. This minimises the chance of trapped voids and facilitates He leak testing post welding.
- All welds must be free from pitting and scale. Must be cleaned to bare metal. If chemicals are used to clean welds care must be taken to remove all traces of chemicals.
- Some chambers such as magnet boxes will need to have some joints welded externally due to access problems on thin chambers. In this case, good penetration must be achieved to ensure minimal gas trapping points.

# 3. Surface finishes

- The inside of the chamber and ports must be 1.6 micron or better for mechanical finishes.
- Can be chemically cleaned (polished) but all traces of chemicals must be neutralised and removed prior to use in vacuum.
- All machining oil to be removed with an alcohol scrub then acetone rinse, minimum. The use of kerosene, turpentine or other oily solvents is not encouraged however where they are used the final clean must be with acetone followed by alcohol. Water based cutting fluids can be used as they are more readily removed therefore having less effect on outgassing.
- Nitrile gloves to be used when assembling cleaned components. Other un-powdered gloves may be used but only those that do not react with the solvents being used.
- Care must be given to cleaning and handling surfaces that will be exposed to vacuum. Gloves should be used to minimise the transfer of dirt and oils from the skin to vacuum surfaces.
- A suitably clean area should be used for the preparation of vacuum systems and all tolls used should also be cleaned (degreased).
- Stainless steel can be cleaned in nitric acid to remove scale that remains from rolling or wire cutting. It is may also be used to prepare stainless steel components for use in ultra-high vacuum systems.

# 4. Flanges

- Flanges must be standard flanges selected from the group referenced this document for compatibility reasons. Exceptions may be made but all 'o' ring seals must be to Australian Standard AS 2842-1986. External flange rings not in contact with the vacuum space may be aluminium alloy. Stainless flanges are not economical or necessary unless the seal requires forces to crush metal seals such as copper ConFlat, aluminium or indium wire.
- Minimum thicknesses for top and bottom flanges to be calculated using the standard in Appendix 1 Structural Calculations for Scientific Vacuum Vessel Design.
- All other flanges to have threads consistent with their design standard.
- ANTARES Primary flanges will be Dependex unless interface is necessary to accommodate other standard flanging.
- For Dependex, screws to be imperial sizes consistent with UNC series.

• STAR utilises many of the major vacuum flange/seal designs. The most appropriate design must be chosen that meets the specific application.

# 5. Seals

- 'O' ring sizes and sealing groove dimensions must comply with the Australian Standard AS 2842-1986. Adjustments to groove sizes can be made where specific 'crush' is necessary for special applications.
- Viton rubber 'o' rings to be used unless a special requirement for an alternate elastomer is identified.
- Standard flange and seal sizes to be used unless an application prohibits their use.
- It is recommended that vacuum sealing surfaces are cleaned just before a seal is made to minimise the possibility of dust and hair settling on sealing surfaces.
- Metal seals may be used in specifically designed sealing surfaces including, aluminium and indium wires. They are very good as ultra-high vacuum seals.

# 6. Screws

- All screws for flanges other than Conflat will be made from stainless steel in either 304 or 316 grades.
- Screws used in Conflat flanges will be made from A2-70 or A4-70.
- All screws to be socket head unless access necessitates hexagon or other special head. Slotted or Philips head must not be used.
- Stainless steel flat washers to be used under all screws where practicable. Do not use lock or star washers on standard flanges.
- In the vacuum space, no screws to be used in holes unless the holes are through holes and can be pumped from both sides. Screws can be used in blind holes if they have a minimum Ø1 mm hole drilled through the centre to allow trapped gas/air to escape.
- Unless a standard flange uses non metric thread, all screw threads must be within the metric series. Unless there is a specific need, avoid using intermediate metric sizes, that is, 0.5 mm steps such as M1.5 mm, M2.5 mm etc.
- Anti-seizing compounds can be used on threads but extreme care must be taken to ensure they do not enter the vacuum space. Graphite powder can be used as a lubricant inside the vacuum space. It can be applied using ethanol to wet the surface to be lubricated. Allow solvent to evaporate prior to pumping.

# 7. Feedthroughs

- Sliding seals should have dual 'o' ring seals with a gland formed seal at least at one end. The shaft finish must be 0.8 micron or better.
- All sliding shafts must have retainers to stop them being pulled through into the vacuum space.
- All coax feedthroughs to be glass or ceramic hermetically sealed. No plastic.

# 8. Lubricants, vacuum greases and epoxies

- Only vacuum specific grease and oils to be used in vacuum systems.
- Silicone based or particular Apiezon family lubricants design for vacuum use, are recommended on sliding seals. Thin smears only.
- No vacuum greases should be used unless there is a small leak through a damaged sealing surface and all other options are not feasible. If grease must be used it must be designed for high vacuum use and only a very thin smear to be used.
- Do not use grease to fill suspected leaks in welds or fittings.
- Where a vacuum epoxy must be used the surface must be well prepared to ensure appropriate bonding. Care must be taken to ensure the epoxy seal doesn't create a trapped void of air which may become an outgassing problem. 'Torr-Seal' is commonly used due to its relatively low vapour pressure, and thermally stable to 120°C. (this is a Varian product)
- Vacuum oils are not recommended for general lubrication as they can creep on surfaces spreading contamination and increasing the outgassing surface.
- See Appendix 3 section 1.5.

#### 9. Mounting

- The load of the chamber assembly must rest on a standard accelerator beam line table ANSTO, HVEC, HVEE or NEC or a purpose built stand that minimises vibration.
- Vibration decoupling will be achieved by supporting the chamber separately from the vacuum pump and using an edge welded bellows between the chamber and pump.
- For ANTARES beam line vacuum equipment the beam axis alignment is nominally 1750 mm from floor height.
- For STAR beam line vacuum equipment the beam axis alignment is nominally 1193 mm from floor height.

#### 10. Access

- The working face of the chamber must face an area that is easily and comfortably accessible. The floor space immediately in front of the working face should be raised using steps to facilitate access.
- Heavy or awkward shaped vacuum equipment must be designed to be lifted with the aid of over cranes for example lifting lugs.

#### 11. Testing

- Vacuum equipment must be tested prior to routine use to minimise time spent chasing leaks in completed systems.
- Vacuum vessels must be helium leak tested to ensure all seals are better than  $1 \ge 10^{-10}$  Pa.m<sup>3</sup>/s ( $1 \ge 10^{-9}$  mbar.l/s). As a standard, during a leak test there should be no deviation from this baseline level for a leak-tight system. A record of the test is to be noted on the engineering drawing showing, date, maximum leak rate, leak location/s, leak detector used and operator name.

- For all other vacuum equipment in-service testing may be necessary to allow complete operation of various feedthroughs and positioners.
- Caution must be taken before positive pressure testing vacuum equipment as the equipment may be irreversibly damaged. Warning Do not use water testing on any vacuum equipment.
- A Residual Gas Analyser (RGA) can be used to give more detailed information about the gas composition inside a vacuum volume. An RGA can be used as a substitute for a helium leak detector with the benefit of providing more information about outgassing, or virtual leaks inside the vacuum volume. The pressure of the vacuum system must be  $< 1 \times 10^{-2}$  Pa to prevent damage to the filament inside the instrument. Instruction manuals must be checked before using the RGA head to ensure safe and effective use.

# 12. Operation and Continued Monitoring

- All chambers should have a high vacuum gauge sensing within the chamber space. This includes all adjoining compartments that may be isolated by a valve.
- The chamber must have valves attached that allow complete vacuum isolation from the beam lines and other equipment.
- To protect high vacuum gauges against the effects of operating in low vacuum, high vacuum gauges should be automatically isolated if the vacuum pressure rises above  $1 \times 10^{-2}$  Pa.
- High vacuum gauges must be cleaned at minimum every 3 years or whenever a gauge develops instability as compared with adjacent gauge readings.
- Systems indicating higher than normally observed pressure must be isolated and repaired. A helium leak test is recommended if the leak location is not clear.
- To avoid undesired contamination, vacuum chambers should be vented with dry nitrogen or argon instead of air. This minimises water vapour and oil from depositing on the vessel walls.

# Chapter 2

# Design Criteria, Information and Philosophies for Vacuum Systems used in the Accelerator Area

#### Overview

In an accelerator system ion beams are transported from ion sources through to end stations (or detectors) through vacuum space maintained inside of beam tubes (lines). The relatively low number of gas molecules in the vacuum ensures a higher mean free path for the accelerated ions and hence less loss of ions through collision which is crucial especially for AMS measurements when counting a very small number of ions. A high vacuum also minimises the formation of plasmas in an electric field at high voltages which can avalanche into spark breakdown damaging sensitive equipment.

The vacuum systems used in the accelerator area are designed to be fail-safe to protect people and equipment. A very high proportion of problems with vacuum systems relate to, seals leaking on first use, being incorrectly mounted or failing due to aging 'o' rings. Other problems, however less frequent, include mechanical damage to sealing surfaces or fatigued parts such as bellows. The problems are all characterised by a poor vacuum or a previously steady vacuum that begins degrading slowly as observed during the operation of the vacuum system over its life time. The remaining problems are due to poor welds or vacuum fittings breaking. Poor welds are usually found during the initial helium leak test. They are remedied by either removing and re-welding the section or using a commercial vacuum epoxy. The choice is dependent on whether the weld is on a structural component or just a seal. Broken vacuum fittings are replaced or repaired.

Within the accelerator area. physicists work directly with technicians who apply broad knowledge and experience in vacuum technology to develop their concepts for use in the accelerator environment. This mix has been successful for 50 years. However not all designs are completed in the accelerator area. There are cases where commercial projects call for a more production based design or designs of chambers with complex noncylindrical shapes. These may go to the ANSTO drawing office for completion or a specialist vacuum design company.



Fig 1. ANSTO designed and developed beam lines on the STAR accelerator in building 22

It is not possible and often not necessary to standardise the design requirements for all components used in the accelerator area so the following information is intended to help designers of vacuum equipment to comply with accelerator specific and Australian standards. As of 2011, there are no known commercial workshops in Australia who specialise in the production of vacuum vessels. Local representatives from vacuum suppliers can provide access to overseas specialist workshops.

# 1. Terminology

**ANTARES** – Australian National Tandem for Applied RESearch. The HVEC FN Tandem accelerator in B53.

Backing – To pump on the exhaust (back) of a high vacuum pump

Beam line – The evacuated tubes that carry the ion beams from the ion sources to the end stations

**Cryopump** – Cryogenic vacuum pump. Uses a helium cooling interface to the vacuum space to freeze gases.

**Dependex** – A type of flange which is the standard flange used on ANTARES and ANSTO built beam lines.

**Fore line** – The vacuum line, tube, pipe, whatever between the high vacuum pump and the backing pump

High vacuum – See details in Chapter 3, para 1.3

ISO - International Standards Organisation

KF – Klein (small) Flange

LF – Large Flange

Low Vacuum – See details in Chapter 3, para 1.3

Outgassing - the liberation of gaseous vapours from the surfaces within a vacuum space

Pa – Unit of pressure, Pascal. 1 Pa = 1 N/m<sup>2</sup> = 0.01 mBar = 7.5 mTorr = 1.45 x 10<sup>-4</sup> PSI

**Pump down = Pump out** – evacuating gas from a vacuum space

**Roughing or Roughing-out** – Usually used to describe the initial transfer of gas from atmospheric pressure to low vacuum.

STAR – Small Tandem for Accelerator Research. The HVEE Tandetron accelerator in B22.

Turbo – an abbreviation for turbomolecular vacuum pump

**Ultra-high vacuum –** See details in Chapter 3, para 1.3

Venting – the equalisation of negative pressure to atmospheric pressure in vacuum equipment

Vessel – generic for a vacuum chamber where an instrument is mounted or interfaced into the vacuum space.

# 2. Standard Units

# 2.1 General

Although metric standard is adopted across all ANSTO accelerator facilities it does not mean that all equipment is metric as the US built FN Tandem is mostly imperial, conversely, the Dutch built HVEE Tandetron is all metric. There is a combination of metric and imperial devices used throughout the facilities but designers must ensure that where practicable all new designs are metric.

# 2.2 Threads

New equipment for use in the accelerator area, where practicable will be designed with metric standard threads.

• Contrary to the standard, on ANTARES there is a combination of threads in use. The accelerator high voltage generator and parts that have originated from the United States are

mostly imperial. Typically, UNC and some UNF. Most beam lines and endstations are metric but all Dependex and most Conflat flanges are imperial.

• ON STAR all threads are metric. ANSTO designed and built beam lines are metric except for the Dependex flanges which are UNC.

Care must be taken to identify threads and use the correct screws. Where practicable, metric threads are to be used other than on Dependex flanging.

# 2.3 Pressure

All pressure indicating devices shall be in the SI unit, Pascal. Historically, Torr was used but this was replaced by Pa in the late 1980's. Most international accelerator labs use mbar. The conversion is as simple as multiplying mbar by 100 to get Pa. Pascal refers to a physical force of newtons per metre squared.

# 3. General Design Philosophies

There are many varieties of vacuum chambers and equipment in use in the accelerator area. Most have been designed and made at ANSTO. Examples include vacuum equipment measurement chambers where samples are measured, beam line transport components, ion source components or sensitive detectors, to name a few. Each has its own unique characteristics and vacuum design considerations.

In order to maintain a suitable mean free path for ion beams whether being transmitted from a source or scattered from a sample into a detection system, high to ultra-high vacuums are necessary. In a high vacuum the density of gas is low enough to minimise electron flow from surfaces at high voltages to surfaces at lesser potentials. It is therefore prudent to ensure designs of vacuum equipment that directly couple to the accelerator are designed for a minimum of high vacuum operation.

In every case the designers must ask the following questions:

- What shape? How big? Where will it fit? How will it be mounted? What level of vacuum?
- Will it be ridged enough and have low enough distortion to mount ion optical components?
- Is the design flexible enough to enable small scope changes in the future? Is it designed with consideration for reuse in another application when the project finishes?
- Does the design use vacuum technology best practises?
- Does it meet Australian and local standards where applicable?
- Is it a safe design?
- Is it achievable with resources available?
- Is it cost effective. Can we modify existing equipment to achieve a similar result?
- Does it have low environmental impact?

# 3.1 The Shape

The first consideration is to define the actual use of the vacuum equipment relative to the experiment. Where possible cylindrical shapes should be considered first as they are stronger under vacuum and consequently being round, ports can be welded into the chamber aligned radially to the centre which ideally aligns instruments including detectors and cameras with the intersection point of the ion beam and the sample being measured.

Non-cylindrical chambers must be carefully designed to included adequate strengthen ribs to minimise distortion and hence misalignment of instruments.



Fig 2. A few examples of vacuum vessels used in the accelerator area

# 3.2 The size

The size is dictated by the space needed inside of the vessel or equipment in order to meet the operational demands. In the case of measurement chambers too big may mean problems with the distance between samples and detection systems and too small may mean manual handling of samples may be inhibited. Large chambers may have to be certified as per AS 4343-2005, Hazard Levels of Pressure Equipment in Australian Standard.

# 3.3 Location

It will be an agreement between the accelerator user groups as to where equipment and beam lines will be located. This agreement will endeavour to minimise conflicts in beam line space, usage, accessibility and resourcing.

# 3.4 Mounting

In general equipment must be secured onto the floor unless it is designed as mobile. An accelerator compliant beam line table should be used with standard beam line supports. Vibration must be identified during the design stage and decoupling stages provided.

Insertion bellows must be provided in long sections of beam lines to enable compression of the beam lines to facilitate dismantling and to ensure minimal strain on vacuum joints.

Designs must consider trip, bump and fall hazards. Structures that support platforms or heavy equipment that may otherwise fall must not be reliant on the structure supporting the vacuum chambers and beam lines. For example, ladders, walkways, etc.

- For ANTARES beam line vacuum equipment the beam axis alignment is nominally 1750 mm from floor height.
- For STAR beam line vacuum equipment the beam axis alignment is nominally 1193 mm from floor height.

# 3.5 Flexible design

It is prudent to ensure where practicable designs ensure some redundancy for feedthroughs, access ports and internal space. A major problem with early chambers was the lack of space allowed for inserting samples by hand or manually manipulating internal equipment and wiring. It is easier to design flexibility into the chamber or equipment at the design stage rather than having to dismantle it and have it reworked in the workshops later.

# 3.6 Can it be made?

When designing any equipment seek advice from a qualified machinist. Often designs include machining that may be very expensive or not possible. Early in the design phase ensure that the resources are available to service the vacuum equipment. It is recommended to consider the following early in the design stage:

- Power
- Cooling water
- Air cooling
- Signal lines
- Control lines
- Add-on electronic and mechanical instrumentation

# 3.7 Safe design

There are no cases that the author has been able to find of scientific vacuum chambers or equipment, like those used in accelerator facilities, failing and causing injury. Vacuum pressure is relatively low, -100 kPa, or about half the equivalent magnitude in a car tyre.

For safe designs the structure must meet the minimum dimensions as calculated in Appendix 1 - Structural Calculations for Scientific Vacuum Vessel Design.

Care must be taken with the design of the following:

- Glass view ports (most glass breakages are in the form of a crack which leak to atmospheric pressure quickly without the spread of shards)
- No internal sharp edges; in the case where a person must insert their hands blindly to access components.
- Venting limits pressure relief for positive pressure?
- Evacuation rate regulation; especially in the case where very thin material windows separate different pressure systems within a single vacuum space for example beryllium windows on x-ray detectors.
- Equipment mounts/stands; where an operator must climb up high to operate devices on the system
- If oil type vacuum pumps are used then mist filters must be fitted to the exhausts.

# **3.8** Cost effective design

All *bells and whistles* may not be necessary even after considering some design redundancy for future changes. Where possible, designs should consider integrating components that are available off the shelf. Designing what has already been designed by someone else is wasting time and effort.

It may be cheaper to modify an existing chamber for use rather than start from the beginning. Many chambers used around the accelerator area have successfully had one or more lives.

# **3.9** Design by numbers

This is a matter of ensuring structural integrity and being able to prove it. Likewise, designing the vacuum pumping system requires calculating the most appropriate size pump rather than going for the "biggest"!

There are formulas in Chapter 3 that allow must crucial calculations to be made from wall thicknesses to conductance. Good enough is not acceptable. Replication of previously designed equipment is acceptable.

# **3.10** Australian Standards

Compliance is mandatory however there are few standards that are relevant to scientific vacuum vessels and equipment. The industry has developed its own standards for flange and seal designs which can be seen in the catalogues of most leading manufacturers of vacuum equipment. It is the responsibility of the designer to ensure compliance with all relevant Australian Standards. Chapter 1 discusses what standards are included.

# 3.11 Environmental impact

Most vacuum systems used in the accelerator area today are designed to be oil free. That is, they do not use oils for lubrication of vacuum seals. There is however some oil vane and diffusion pumps still in use but the oils are responsibly disposed of through waste management at ANSTO. Some old diffusion pumps had been used with mercury as the pumping agent. Personnel must be cautious of using old diffusion pumps and their associated backing pumps as there may be residual mercury on surfaces and in the backing pump oil.

Solvents used for cleaning are used in small amounts and usually for wiping rather than immersing. Where baths are needed the solvents are reused when possible. The solvent bath used for degreasing, self recycles the solvent by design however there is a small loss of solvent over time into the atmosphere.

Where possible solvents used are selected with the environmental impact in mind.

# 3.12 Manufacture

If ANSTO cannot provide the level of manufacture quality in-house then scientific vacuum equipment is best manufactured by companies that specialise in high tolerance machining and welding. A general metal fabricator will not suffice.

Attention must be paid to considering standard sized tooling during manufacture. Some companies will charge for the purchase of tooling that they consider to be non-standard.

It is imperative that the drawings for manufacture state all tolerances and qualities for the finished product.

On all design drawings, where the angle of the welded ports is crucial, ensure angular tolerances are clear and achievable. If necessary, specify the use of mandrels to hold ports in place during welding.

Some success has been made outsourcing the manufacture through vacuum companies to overseas manufacturers who specialise in high quality small runs.

# 3.13 Testing

Testing the vacuum equipment or chamber is essential to establish compliance with the design and the all important ultimate base vacuum. Helium leak testing is the standard method used for chambers and most equipment however on some equipment in-service testing may be the only option. This is where add on vacuum equipment doesn't change the original base vacuum of the system it is connected to.

#### 3.14 Inspection

All parts should be cleaned prior to inspection. This doesn't need to be the final clean but enough to ensure all welds and surfaces can be clearly inspected. Welds must be checked for pits and alignment along the weld lines and seals must be checked for surface finishes. All crucial dimensional must be checked. It is recommended that any non-complying findings are photographed and documented in the project file. All non-complying parts should be reworked rather than *patched up*.

#### 3.15 Certification

As most vacuum vessels and equipment in the accelerator area fall within Hazard Level E of AS 4343-2005, certification is a local process. For large measurement vacuum vessels, results from the inspection and leak testing will be recorded on the engineering drawing and registered in the local accelerator register and/or ANSTO drawing register as appropriate.

#### 3.16 Documentation

For all large measurement chambers requiring any level of design calculations, design documents must be kept with project files.

#### 3.17 Maintaining

Vacuum chambers and beam lines do not require high levels of maintenance other than a general periodic clean and occasional seal replacement. Vacuum fittings and devices will have their own level of maintenance so access to these components must not be difficult.

The design must be easy to maintain especially moving parts, sliding or rotating seal and other parts in the vacuum space that may wear. Also vacuum pumping systems that may be mounted under or near the vacuum chambers. Generally any part that will require any level of routine maintenance must be accessible without the need for a major disassembly.

Vacuum pumping systems other than oil free systems, connected onto beam lines and chambers must include suitable oil traps between the backing and high vacuum pumps and where mist filters on all exhausts.

Some high vacuum gauges will require periodic cleaning. On ANTARES high vacuum cold cathode gauges are scheduled for cleaning every 12-18 months or max 3 years for difficult to service or less crucial instrument locations. Otherwise gauges will be cleaned on demand within the periods.

# 4. Performance and Operations

#### 4.1 Ultimate Vacuum

The ultimate vacuum is the maximum vacuum or lowest pressure the vacuum equipment typically reached after 3-5 days of uninterrupted pumping. This is a benchmark value which is used to assess the long term quality of the vacuum system. It is sometimes referred to as the *base vacuum or static vacuum*.

Generally, vacuums in the beam lines and chambers are well within the high vacuum range. Vacuums lower than this range will indicate a higher than normal gas load, a failing seal or a pump malfunction. On the accelerators the two main types of high vacuum pumps are turbomolecular and cryogenic pumps. For a system designed for high vacuum or better with no leaks and low outgassing then vacuums better than  $4 \times 10^{-6}$  Pa should be readily achieved.

The ultimate vacuum is improved by the correct choice of materials, seal types, high vacuum pumps used cleanliness and correct operation. All fittings must be designed or selected deliberately for high to ultrahigh vacuum use. Surface preparation and cleanliness will contribute to improved vacuums. A finger print can outgas for long periods of time. A bigger (higher speed, higher ultimate vacuum) vacuum pump does not mean a better vacuum!

# 4.2 Working Vacuum

The working vacuum may be lower than the ultimate vacuum due to sample cycling (changing), introduced gas loads from adjoining systems or outgassing samples. A system must be designed to ensure the high vacuum pumping system has the optimum pumping speed and ultimate vacuum. Once a vacuum system reaches the "working vacuum" little operator intervention is necessary to maintain it but there are some important points regarding ongoing monitoring to ensure a failure is not immanent.

For sections of beam lines that are holding a static vacuum a cursory glance at the local vacuum gauge will indicate the stability of the vacuum. Generally, the (vacuum) pressure will change slightly throughout a day for example due to diurnal changes in ambient temperature and perceived changes due to ionisation gauges "wandering" as they become dirty.

In sections of beam lines that are in the vicinity of operating beam lines or near end stations the vacuum will be continually changing. Factors that influence these changes include increased gas loads from samples outgassing or surfaces that have just be exposed to high humidity, outgassing due to temperature changes within the vacuum space (including bake outs), outgassing from ion beams striking plastics, etc. Gas molecules don't have a preference to whether they travel towards or away from a pump during molecular flow so the source of gas can come from just about anywhere in the open system. These factors must be considered when monitoring vacuums throughout an entire system.

If a vacuum system's pressure rises slowly over many days or weeks if it is not related to the cryopump loading then it may be a seal failing. In this case a helium leak test is recommended.

A fundamental flaw in many vacuum system designs is the neglect for monitoring the backing pressure between the backing pumps and turbo pumps. Pirani gauges which are best suited for this role are relatively cheap and are the best diagnostic tool for determining overall vacuum system performance. The backing pressure should be consistent with changes in the high vacuum pressure. By monitoring the backing pressure over time, the base vacuum will be realised relative to the high vacuum so a pending failure of the backing pump can be determined, hopefully, well before it happens.

Care must be taken to maintain systems that have the potential to degrade over time for example an oil vane roughing pump will back stream oil mist that may accumulate in the high vacuum side of the system over time. This will contribute to lower vacuums due to higher gas loads from the partial pressures of contaminants.

# 4.3 Sustainable Operation

Now that the vacuum system is designed, manufactured and commissioned for routine service, operating procedures have to be defined and adhered to, to maintain reliable trouble-free service. There are 3 processes that summarise the continuous cycle of operating a vacuum system:

- Pumping down from atmospheric pressure
- Using the working vacuum
- Venting to atmospheric pressure

The accelerator beam lines are divided into stand alone vacuum systems but in essence function as one. Each system can impact on a neighbouring system if things go wrong. In the case of end stations which cycle up and down in pressure more than any other devices an operator must be 100% sure of the pump down sequence in order to avoid damaging, pumps, samples, high voltage devices, detectors, etc. It is possible to shut down the accelerators as well. Most sections of the accelerator vacuum systems are interlocked to isolate a pressure surge to small area. Beam line vacuum systems are design so that both the vacuum pump and the beam line are isolated via gate valves. This is a typical arrangement for endstations as well.

The following must be read remembering that all 3 processes work in a cyclic fashion in a continuous loop.

# 4.4 **Operational Protocols**

Key vacuum systems operate continuously, 24 hours per day, seven days per week. There are several vacuum systems that are cycled on and off as usage demands. All vacuum systems have one or more high vacuum gauges, some of which are interlocked to make the systems fail-safe. Any slow leaks or major catastrophic failures will close valves and isolate the system. In the few cases where no interlocks are used, a catastrophic failure will result in the high vacuum pump shutting down.

- For a <u>turbo pump</u>, as the pressure rises the gas load on the pump will cause the pump to draw more current to maintain it's speed. It will eventually fail when the current trip level is reached in the controller.
- For a <u>cryopump</u>, as the pressure rises the inflowing gas will condense or freeze onto the in the pump until it begins to warm up. Eventually there will be a thermal trip activated o the compressor which will shut is down. In both cases it is fail-safe.

As part of the accelerator's maintenance system, daily monitoring of all vacuum pressures is carried out. Where a pressure is seen to rise by a few percent without a particular reason, further monitoring will occur and corrective action started. Instantaneous failures are dealt with immediately. All observations and whether any maintenance carried out is noted on the maintenance log.

Where a vacuum system is isolated for service or where it has failed the appropriate tags are placed on the power leads.

Unattended out of hour's operation notification is not normally used as vacuum systems run continuously.

#### 4.5 Pump down

#### General

Pump down time is when most mistakes are made and significant time lost. Before a pump down begins the operator must ensure that all seals that were disturbed had been cleaned and replaced if damaged. The system must then be sealed ready for pump down and the roughing-out valve on the rough-out port, closed.

If a dry pump is to be used, such as a scroll pump, for rough-out then the tube is connected to the pumping port. If an oil vane rotary pump is used then ensure the cold trap has been filled beforehand.

#### Rough-out

Start the roughing pump and allow it to reach base vacuum. This is your first indicator of whether you have a leak in the system being pumped and when you have reached the pumping limit of the

pump. Slowly open the pump out valve on the system. This is where mistakes are made and damaged caused. Relatively speaking the volume in most sections of beam lines and endstations is small and the bulk of the gas can be removed very quickly. The problem is that a reduction in pressure too quickly can cause mechanical shock to fragile parts that must adjust slowly in order to minimise stress such as thin windows on detectors and detectors or devices that contain carbon foils.

Operators must always open roughing out valves slowly then gradually increasing as the gas load reduces. The change in the sound of the loaded pump is a very good indicator. A good design for more sensitive pump outs is to use a metering valve in line with the rough-out port to act as a regulating orifice.

#### Cryogenic Vacuum Pumps

For systems using cryopumps as the primary high vacuum pump it is essential to bring the base pressure down to the limit of the roughing pump before changing over pumping to the high vacuum pump. This reduces the gas load to be trapped (captured) on the cryo pump and extends the service life of the cryopump. Cryopumps are widely used on ANTARES as the primary high vacuum pumps but their limitation is that the warm up as they trap more gas causing a rise in base pressure. That is, when a cryopump is reaching saturation the temperature on the surface increases. More energy is required to hold the molecules on the surface and since it is almost fixed by design the temperature increases. A cryopump's effective pumping speed depends on the freezing and boiling points of the gases being pump relative to the cryopump's temperature. As the temperature increases more molecules leave the surface (boil away) raising the pressure. Some molecules are re-trapped as they hit the surface of the arrays and loose kinetic energy but they have a short residency time and soon add to the rising pressure in the system. Eventually the base pressure in the system becomes unusable so the pump must be regenerated.

#### Turbomolecular Vacuum Pumps

The other type of high vacuum pump widely used in the accelerator area is a turbo pump. Unlike a cryo pump that traps or captures gas molecules a turbo pump transfers the gas by conducting it almost one-way out of the vacuum space. The lowest roughing pressure before changing to a high vacuum pump is not as crucial when a turbo pump is being used. In saying this, the bulk of the gas must be removed before changing from roughing out to high vacuum pumping otherwise the vanes on the turbo pump may be overly stressed due to the pressure surge. A typical changeover pressure range is 5 - 50 Pa.

The optimum changeover time to minimise pump down times can be estimated with the assistance of the formulas found within the document. Letting the roughing pressure reduce to the limit of the roughing pump before changing over to the turbo pump is an inefficient method of system pumping. A turbo pump is more efficient at removing gas molecules as the flow moves towards molecular flow. Changing pumping to a turbo pump just before molecular flow is reached will increase the pumping speed and base vacuum will be reached sooner.

# Pumping Speed

Each type of gas will pump away at a different speed depending on the mean velocity of the molecule which is a function of its mass. A hydrogen molecule of 0.02 kg/mol and mean velocity of 1762 m/s will reach a pump much quicker than say a Xenon atom of 0.13 kg/mol with a mean velocity of 217 m/s.

# **Limitations**

In rare circumstances roughing pumps may be left pumping high vacuum systems by mistake. It is unlikely to damage either pump but the ultimate vacuum will not be reached as the roughing pumps are ineffective at conducting away molecules in molecular flow. Water vapour and residual solvents are difficult to remove from a vacuum space with *normal* vacuum pumping. Most low vacuum backing pumps provide gas ballast which can speed up the removal of water vapour and solvents that may otherwise condense inside of the pump during the compression stage. By introducing a small amount of air near the outlet of the compression stage the small increase in pressure helps carry water vapour and solvents to the exhaust before they can condense.

Some vacuum systems have been designed to allow chambers and beam lines to be heated. Heating causes contaminants to be removed from surfaces at lower than normal vacuums. The higher the temperature the faster the removal will be. Care must be taken to ensure heating doesn't inadvertently cause plastics and elastomers used within the vacuum space to outgas. Most bakeable systems use ceramics and metals only.

It is important that where vacuum systems are permanently connected to high voltage devices for roughing out, the insulated (plastic) pump out lines between the roughing pump and high voltage device must be brought up to atmospheric pressure before high voltages are reapplied. Otherwise the low molecular density of gas in the tube may ionise causing burning of the tube or even spark damage that can puncture the tube causing a leak.

# 4.6 Venting

# General

Venting can cause damage to vacuum components just like pumping out a system from atmospheric pressure too fast.

#### Venting Speed

As discussed earlier the volume of vacuum systems on the accelerators is measured in litres to a several tens of litres. A catastrophic loss of vacuum will be more of a brief audible event rather than a dangerous mechanical failure. Little energy is required to change these systems by 100 kPa. There are a few devices in the vacuum space that may be damaged due to pumping out too fast but none that are considered a danger to personnel.

Other than venting too fast the second point to consider is the introduction of water vapour if venting with air. Key vacuum systems on the accelerators are vented with either dry nitrogen or argon both of which are delivered through a closed reticulation system from gas bottles. The choice of gas is dependent on molecular contamination within the system for example nitrogen is not preferred to vent the 846 ion source as nitrogen is an element that is known to bond with other elements causing molecular ion beams.

#### Regulation of Flow

There are many low flow regulators and rotameters (tubular flow meters) available that provide adequate control over venting. A simple in-house gas regulating system which works well is a tee piece connected between the venting port and the venting gas supply line. The centre port of the tee piece faces upwards and a ping pong ball sits over the hole with a cage over it so that it cant be blown away. Under the ball is an 'o' ring seal. Before the venting valve is opened the venting gas is allowed to flow. The pressure of the gas lifts the ping pong ball of its seal. The gas flow is adjusted depending on the height of the ball above the seal. The ball can only travel a centimetre or so. Once the flow is set the venting valve is opened and the now regulated gas flows into the vacuum space and the balls drops onto the seal stopping air from entering the vacuum space. As the pressure reaches equilibrium the ball begins to rise off the seal until eventually it rises to the original set height indicating the vacuum space is now at atmospheric pressure.

#### Precautions

- Before venting a vacuum system it is wise to isolate all ports and beam lines that must remain under vacuum. To save stressing a beryllium window on an x-ray detector due to the changes in pressure the system should be designed so that the x-ray detector can be isolated with a gate valve. The same logic can be applied to all equipment that may be effected. It is also important to ensure all power supplies and high voltages that may be hazardous to personnel or equipment are isolated prior to venting. This can be done either by administrative protocols or using a vacuum switch such as programmable relays in a vacuum gauge controller.
- Some vacuum gauge controllers use the signal from a Pirani gauge measuring at the same location as a high vacuum gauge to switch off the high vacuum gauge when the pressure becomes too high. This saves the gauge from ionising air when the system is at atmospheric pressure which will significantly reduce its life time.
- Another problem with venting too quickly is the disturbance of dust. Accelerator tubes must be vented slowly to minimise equalisation stresses and also the disturbance of dust. In this case a metering valve is used to limit the vent flow rate.
- Venting can cause loud high frequency noise so care must be taken to dampen the noise to a comfortable level by controlling the air admittance rate, or use hearing protection.
- Most beam line vacuum pumping systems are interlocked to isolate either the beam line or pump or both. In the event where an accidental venting occurs the gate valves will close minimising the effect on surrounding systems.

# 5. Safety

# 5.1 General

Catastrophic failure causing injuries are not common and no reference is known from overseas accelerator facilities. This is almost certainly due to the low pressures involved in vacuums and the size and nature of the scientific apparatus. There are of course many failures reported for industrial sized tanks and equipment.

The vacuum systems are essentially fail-safe from absolute vacuum to atmospheric pressure. The worst case has been a number of beam lines and measurement chambers developing leaks and thin windows that have ruptured. In each case there has been a gradual rise in pressure to atmospheric pressure.

However, a few rules do apply.

- All vacuum systems must have one or more vacuum gauges attached with the exception to some static chambers used for storing moisture sensitive items or radiation detectors.
- High vacuum gauges must be switched off before vacuum systems are vented to atmospheric pressure.
- Only nitrogen, argon or other inert gas to be used for venting. No flammable gases to be used such as hydrogen.
- Turbomolecular pumps must not be vented from full speed. See manufacturer's recommendations.
- All designs must be consistent or better than the best practises outlined in this document. Inferior equipment must be isolated and reported to the Leader of Accelerator Operations.

- Cryopumps must not be operated without a serviceable pop off valve.
- Cold traps must be warmed and blown dry at least every 6 months to minimise  $O_2$  concentrating (and for de-icing as necessary).

# 5.2 Interlocks

Nearly all vacuum systems on the accelerators are interlocked via a set of integrated relays in a vacuum gauge controller, to protect equipment. Generally, if a rise in pressure is detected above a particular set level then the interlock relays will activate to close corresponding gate and line valves. This is designed to minimise the number of vacuums that must be regenerated in a fault. The interlock system also protects accelerator tubes from exposure to moisture and in the event of a catastrophic failure of the accelerator tubes; the vacuum system will close the main accelerator gate valves to trap SF6 gas.

If an interlocked failed causing a gate valve to stay open in the event of a pressure rise then the next interlock should activate the next gate valve. If the next interlock fails it is possible that the two interlocks share the same vacuum gauge controller which is at fault. In that case the next interlock should activate. If no interlocks activated there would be no increased risk of injury to personnel but all vacuum systems will require regeneration or a restart after the interlocks were repaired and tested.

# 5.3 Fumes from exhausts

There are a number of oil vane pumps in use on the accelerators. In the past, the exhaust on the pumps has released oil mist into the atmosphere inside the buildings. Now, on nearly all oil vane pumps in continual service there are mist filters attached or the exhaust is plumbed into an extraction system that vents the exhaust fumes outside of the building.

# 5.4 Earthing

Some vacuum gauge controllers are prone to high voltage transient surge damage. These sometimes cause unnecessary isolation of systems requiring resetting. It is very important that all vacuum gauge controllers are well earthed.

# 6. Serviceability

# 6.1 Access

Most beam lines with the exception to some measurement chambers are static volumes of vacuum requiring little operator intervention other than during preventative maintenance. Vacuum pumping systems do require a higher level of intervention during maintenance and operation. With these points in mind the designer must ensure that all controls and monitoring devices are localised at the working face of the beam line or chamber.

Due to beam lines being so high above floor level, working on them usually requires some form of ladder or steps. When designing beam lines, be sure to have pump out ports facing the passageway beside the beam line. The same applies to vacuum chambers and other large volume vessels.

Measurement chamber will need to have the access port accessible from a location near the control side of the chamber. A platform is recommended to allow easy reach and eay viewing into the ports.

# 6.2 Maintenance

A well designed vacuum system will require routine maintenance including:

- Changing faulty seals cleaning seals that are regularly opened
- Cleaning vacuum gauges especially cold cathode type high vacuum gauges
- General cleaning inside of measurement chambers and equipment that is; regularly opened and touched, whenever it looks dirty, when the base vacuum degrades or when the surfaces are exposed to the atmosphere for long periods at a time (weeks).
- Servicing of vacuum pumps the group of the most routine maintenance tasks includes; cryopump regeneration, turbo pump bearings, scroll pump tip seals and oil vane pump oil changes to name a few
- Actuator service sliding shafts that penetrate into the vacuum space

# 6.3 Cleaning

Cleaning is by far the most essential part of general maintenance of a vacuum system. Poor cleanliness will lead to poor vacuums and possibly sample contamination. It is a general philosophy that cleaning is done with solvents, wiped over and/or scrubbed and thoroughly dried.

# **Solvents**

Typically ethanol, methanol and acetone are used. These solvents remove most greases and oils that are encountered. What ever solvent is chosen the most crucial point to remember is that it must not leave any residue.

For surface finishing see section on 'Surface Finishes'.

Chemical polishing is not commonly used in the accelerator area due to the size of the baths that are needed for large parts, the problem of cleaning or neutralising residual chemicals and not having a suitable facility for handling the types of chemicals used. Although once used electro-polishing is no longer encourages for the same reasons. If suitable facilities were available both chemical and electro-polishing are real options for very good cleans.

A general clean will consist of a number of different levels of cleaning. A single clean with the same solvent will not remove some oils and greases therefore a progressive approached is used using a variety of solvents and wiping materials. The following is a guide only of some such techniques.

Using a dry gas or filtered compressed air, most loose dust can be blown away. This is sometimes the best starting point for cleaning. A final blow down is also useful to ensure complete removal of lint.

The first clean will take away coarse particulates or chemicals. Ethanol dampened tissues if good for this level of cleaning. The ethanol helps remove some oils and greases and mixes with water to reduce the evaporation time. It is also good for mopping up dirt and dust. Scour pads (or 1200 wet or dry paper) moistened with ethanol is good for removing stubborn dirt. This must be continued until better than 95% of dirt, dust, oil and grease is removed. For stubborn dirt it may be necessary to use a petroleum based solvent followed by an ethanol rinse and wipe. Wiping is key to cleaning. The light mechanical brushing dislodges most dirt.

The second clean starts with ethanol but finishes with acetone. Only lint free wipes are used such as Kimwipes. At the end of this level of cleaning the equipment must look ready for use in the vacuum. That doesn't mean it is. It will more than likely have residues remaining on the surface.

Final clean. It is recommended that methanol is used for the final clean or another low residue solvent. The final clean must use lint free wipes dampened with methanol and lightly rubbed over the entire surface. An alternative final clean is to use the solvent bath and suspend the equipment in the hot vapour for a few minutes. Care must be taken with the bath as the hot vapour will degrease skin very quickly.

If a shiny (healthy looking) clean is desired then a metal polish such as Brasso can be used. Whenever Brasso is used the residue must be cleaned off with ethanol. Care must be taken not to get metal polish pastes into gaps that cannot be thoroughly cleaned out. Brasso is an enemy of vacuum, it traps water.

# 6.4 leak detection

In the accelerator area, a leak is characterised by a flow of gas molecules into a space that is below atmospheric pressure. The pressure of the gas entering the space will restrict the vacuum system from reaching its design pressure which is typically in the high vacuum range. As a benchmark, if a vacuum is  $1 \times 10^{-5}$  Pa or more then disregarding the effects from outgassing, there may be a leak. In system designed for ultrahigh vacuum the benchmark will be in the order of  $1 \times 10^{-7}$  Pa.

Leak detection is usually only necessary for new chambers or equipment. It can be done offline or after the part has been mounted into a beam line. The most common method of leak detection is a helium leak test. Helium atoms being so small will find their way through openings that are too small for most other molecules. Generally, the smaller the leak, the less helium that can make its way along a leak path. The leak detector is usually a mass spectrometer tuned to have highest sensitivity for helium. The detector is connected directly to the device being tested. Any helium that makes its way through the leak will eventually be measured in the mass spectrometer. Residual Gas Analysers (RGA), although not specifically leak detectors, can be used for just that. Working on the same principle, mass spectrometry, the RGA can detect helium as well as other gases.

In some cases leaks can be too large for a helium leak test, that is, the amount of gas in the device being tested is too high diluting the helium to an ineffective percentage. Also, if the pressure is too high the leak detector cannot be opened to the device as the pressure gradient will be too high. Leak detectors work best from the higher end of the low vacuum range.

Often there are multiple leaks in systems. It may mean repairing large leaks in order to find small leaks. Where a leak is too large for the helium leak detector a few other methods can be used to locate leaks.

- Solvents have a very low viscosity and vaporise very quickly in air. Wetting surfaces with small amounts of acetone or ethanol can show small changes on Pirani and Penning gauges.
- Isolation of seals can, by process of elimination, restrict the testing area allowing large leaks to be isolated. For example, if a chamber with many ports and devices attached has a leak. It may be prudent to close all valves leading away from the chamber except for the leak detector valve then sequentially blank off the ports one at a time (using a sound flange and new seal) and test for a leak.

# 7. Soldering wires vs mechanical joints

It is preferable that wires in a vacuum space are joined together with a mechanical clamping type joint. The clamp must allow for gas to escape. Where a direct joint is necessary then soldering is permitted. Standard lead/zinc with resin core is okay provided that ALL flux is removed with ethanol. This may require some soaking in solvent with gentle agitation. Solder without resin core

can be used on stainless steel with a zinc chloride based eutectic flux, used sparingly. Ensure all flux is removed especially between the wire strands.

Be aware that in Faraday cups the continual flexing of wires can work harden the wire causing it to break. These are typically soldered joints. If soldering, be sure not to melt the insulation. It is highly recommended to replace normal plastic insulation with loose fitting PTFE (Teflon) spaghetti.

# 8. Flanges and seals

# 8.1 Common vacuum seals used in the accelerator area

A variety of standard vacuum seals are used on all vessels depending on the degree of vacuum to be achieved and the type of fitting that will be used. The types are widely used in the vacuum industry. ANTARES uses the Dependex style seals and flanges. This was developed for use with HVEC accelerators over 50 years ago. Other types in use include Conflat, ISO, and KF are used as well.

Туре	Vacuum level	Flange type	Seal type
KF	Low to High	Clamp (typically)	Viton 'o' ring, Al, PTFE
Edwards	Low	Threaded retaining nut	Viton 'o' ring
Dependex	High	Ring retained by spring clip	Viton 'o' ring supported by a ring
LF or ISO	High	Ring or clamps	Viton 'o' ring supported by a ring or in a groove
Conflat	Ultra high	Ring fixed or rotating	Copper ring, Square section 'o' ring
Specials	Various	Various	Aluminium or indium wire

Table 1. Common types of vacuum flanges and seals. Each type is available in multiple sizes

# 8.2 KF – Klein Flange

A common flange and seal used for general low vacuum applications due its ease of assembly and vacuum performance. It is common to have KF flanges and seals within the high vacuum side of a system. Vacuum performance can be enhanced by the use of aluminium or PTFE seals which directly replace the Viton 'o' ring.

Many general and specialised vacuum equipment is fitted with KF fittings as the range of adaptors from KF to other major types is readily available.





Fig 3. Assembly drawing for a typical KF flanges seal and a photo of the flange, seal and clamp

# 8.3 Edwards

This is no longer a preferred type that is used in the accelerator area however some equipment still uses this type.

One end of the fitting is shaped to retain an 'o' ring while the other end has an angled sealing surface that mates and crushes the 'o' ring to make a seal. They are connected by a threaded nut which when tightened squeezes the two halves together. It is normally for low vacuum applications.

# 8.4 Dependex

This was designed for use on HVEC Van der Graaff accelerators and beam lines. It was introduced to ANSTO in the early 1960's when the 3MV Van de Graaff operated in B22. Here, 1", 2" and 4" Dependex was used. When ANSTO purchased the FN Tandem nearly all fittings used were 4" Dependex (100 mm nominal). It is suitable for high vacuum use and often mated with Conflat flanges in systems that operate near ultra high vacuum.

The Dependex seal consists of a supported 'o' ring sealing between two opposing  $5^{\circ}$  metal faces. The 'o' ring is supported around its inner diameter by a ring to ensure the 'o' ring aligns with the centre of the 2 faces. The inner ring also sets the maximum crush on the 'o' ring that is the ring fits into a step on both halves of the Dependex seal keeping them separated at a fixed distance. Another ring which fits on the outer diameter of the 'o' ring does little more than keep the 'o' ring clean. The rings are often called *egg rings* due to their resemblance.

The Dependex flanges are held in place on a tube, or other fitting that is designed to accept Dependex, by a large circlip that fits into an outer groove a few mm from the end of the sealing face.

The flanges are normally drilled such that the holes alternate between being tapped or clearance. 1" and 2" Dependex use <sup>1</sup>/<sub>4</sub>" UNC, cap (socket or Allen) head screws and 4" (100 mm) Dependex use 5/16" UNC, cap (socket or Allen) head screws. Only use stainless steel screws (of one length) and washers. Discard plated steel screws as they are identified during maintenance. Washers are recommended as most flanges are aluminium alloy.

To complete the Dependex assembly, the flanges must be placed over the tube ends and then retained in place with a circlip. Secondly, the 'o' ring is stretched onto an inner ring and then an outer ring is placed around the assembly. The two Dependex ends are brought close together then the seal assembly is placed between them. The inner ring is aligned with the step on one side then the other tube is aligned and than pushed onto the ring. At this point it is important to ensure the 'o' ring assembly stays in place until the bolts are tightened. Screws should be finger-tightened and the flange faces must be parallel. Tighten screws in an opposite pattern. Some old flanges will have worn threads so screws may only be turned with the aid of an Allen key.

#### Problems

There are a number of things that can go wrong with assembling Dependex fittings.

- There have been cases where inner egg rings have been made too wide so that when fitted between the tubes the 'o' ring is prevented from crushing adequately to form a vacuum seal.
- Another problem with the inner rings is where some Dependex seals have been designed with too shallow steps so that a standard inner ring becomes too wide. In this case special inner rings have been made which are narrower. Personnel servicing non-standard joints must take care to reuse the right sized inner ring. A label should be placed on all non-standard joints identifying them.

- Some steps for inner rings have also been found to be out of tolerance preventing standard size inner rings from locating in the step. In this case some operators have cut a small section from the inner rings so that they can be squeezed together. Cutting inner rings like this is only recommended to solve this type of problem.
- Some flanges have been design for special applications with slightly different PCD's. These have unfortunately bee mixed up with standard stocks. As personnel who identify them (sometimes after a frustrating encounter) should put them aside and label them accordingly.
- Old flanges that have been over-tightened may have stretched threads. They are identifiable by the difficulty in starting screws. Either re-tap the threads or discard the flange. There have been case were tight threads have mislead personnel into thinking they have tightened the Dependex joint only to find leaks.
- Circlips can stretch especially those in over or under sized slots. They are easily identified by there distorted shape. Only use circlips that are flat when not under tension. A discerning technician should discard rusty circlips that cannot be successfully cleaned.
- Care must be taken to make sure the two faces of the Dependex joint are parallel before the joint is closed and bolted together. Failing to do this may cause a leak in the joint. It is acceptable if a bellows is used between sections that have misalignments.
- Twisted 'o' rings can cause leaks although small. There have been cases where a twist has caused the 'o' ring to bulge towards the outer ring making a less than adequate seal.

# 8.5 Special Flanges

These consist of flanges that have been designed to fit application where "off-the-shelf" flanges and seals cannot be used. They are typically based on common designs but modified to suit the application.

# Example

The Alphatross ion source uses two stainless steel flanges to hold the heater reservoir in place. Here the vacuum sealing surface is a flat surface about 8mm wide on each flange. The fixed flange is threaded in an imperial thread and the reservoir flange is open hole. The gasket used is aluminium wire which has been fused together to make a ring. The joint is similar in size to the wire. The wire is supported in place using about 3 to 4 strips of aluminium foil looped around the wire and sticky taped to the edges of the flange. The flange is tightened in a circular pattern to ensure maximum crush on the wire.

# Problems

- The wire is essentially unsupported so vacuum must not be applied until the wire is completely crushed.
- Bulbous joints in the aluminium wire dent the sealing faces on the flanges as they are tightened so they must not be used.

# 8.6 ISO or LF (Large Flange)

This is a European range of fittings designed for high vacuums and to suit most tube sizes used in the vacuum industry. There are two types of design which meet most light and heavy duty applications.

ISO-K flanges are a robust joint which are suitable for supporting larger and heavier items such as vacuum pumps and large gate valves. They are characterised by the outer clamping system which holds the two halves of the joint together.

ISO-F flanges are more of a traditional bolted flange type joint like Dependex. ISO-F and ISO-K can be joined together using a half clamp which fits onto a standard ISO-F flange. The half clamps then attach to the ISO-K flange.

Available in sizes from 63 mm nominal bore to 500 mm NB.



Fig 4. ISO family of Flanges

# 8.7 Conflat CF

Conflat is designed as an all metal vacuum seal for ultra high vacuum applications. A copper gasket is sandwiched between two thick stainless steel flanges that have a knife edge machined into the sealing face. As the flanges are tightened together the knife edges "bite" into the copper to produce an all metal seal with very low outgassing.

The term "ConFlat" is a registered trademark of Varian, Inc., so "CF" is commonly used by other flange manufacturers. Conflat sealed systems can reach vacuums as high as  $1 \times 10^{-11}$  Pa.

The copper gasket locates into a partially recessed in a groove in each flange to provide alignment prior to tightening. It also stops the gasket from moving during bake out. Conflat can be baked up to  $450^{\circ}$ C.

Most Conflat flanges have imperial sized bolts. The original style of bolt had a multi-face head that a ring spanner would fit over. This is to allow more purchase on the head from many angles. It is not uncommon now to find hexagon and socket head screws in use. Be aware that the steel must be a high tensile grade to ensure optimum force can be used to crush the gasket. It is essential that the flange faces are parallel during and on final tightening. A torque wrench may be used to facilitate uniform tightening. It is highly recommended that a molybdenum grease or graphite loaded grease is used on the threads.

Conflat flanges are available in sizes from 10 mm nominal bore to 250 mm NB. Sometimes nominal bore is written DN in front of the size, eg DN63.

Square section Viton 'o' rings may be used with Conflat flanges however this compromises the ultra high vacuum design and ultra high vacuums may not be reached.

# Problems

- Copper gasket not aligned in groove and edge is partially clamped on the bolt hole face of the flange. This lead to partial seal and most likely, a leak. Most Conflat flanges have two small grooves a little more than a millimetre wide machined into the bolt face. These are for locating special clips that hold the gasket in place while the flanges are brought into place. Just prior to tightening they clips are removed. They are sometimes mistakenly called pressure relieving grooves.
- Copper gaskets must only be used once. Unless the copper hasn't been fully compressed after the first use there will not be enough material for the knife edge to reform a seal.
- Due to the forces required for a knife edge to indent a copper gasket the flanges are made of stainless steel and quite thick. Large flanges are quite heavy and care must be taken not to drop them.
- Where Conflat flange bolts have been used more than once the threads in the flange and screws may have stretched. In this case, it is recommended that the same screws are reused in the stretched threads (with a little molybdenum grease) as they will have the same profile. Sometimes using new screws in a stretched thread will cause binding. Re-tap the flange if necessary.
- Stretched threads are possible where nuts and bolts are used through open holes so ensure the nuts go onto the bolt the same way they were removed. Alternatively replace the nuts and bolts outright.
- For ultra high vacuum ensure the gaskets are handled with gloves. Copper reacts well with the sweat in a fingerprint leading to a long outgassing process.

# 9. Bellows

Bellows consist of two styles, edge welded for ease of extension or compression and corrugated style which is has minimal movement. Each style is made from a thin stainless steel that has been electron beam welded. The ends are usually finished in a standard flange to suit the application.

# 9.1 Corrugated style

These are typically used for two purposes. Firstly, to allow sections of beam lines or equipment to be compressed to allow easy removal of sections without dragging the sealing surfaces against each other. Secondly, as bellows can distort and still maintain their structural integrity they are ideal to

join sections of beam line or equipment that have a deliberate mis-alignment. They can be purchased with almost any style of flange including specials. See example pictures.



Large bellows. ~300mm diameter. Stainless steel, wall thickness ~ 0.8 mm. This bellows is used to correct alignment offsets in the magnet box

Medium bellows. ~ 100 mm diameter. Stainless steel. wall thickness <0.5 mm. Used to allow sections of the beam line to be compressed to facilitate beam line component removal.



Small bellows. Electron beam edge welded stainless steel. Material thickness <0.25 mm. Used to isolate vibrations in the beam line.

Fig 5. Examples of bellows

#### 9.2 **Edge Welded Bellows**

These bellows are made up from very thin stainless steel discs stacked together and welded along their edges. The advantage of the design is the concertina style bellows that is very flexible allowing designs that have long extensions and short compressions. They have two main applications. Firstly, applications that requires a device to extend or contract into the vacuum space where an elastomeric seal on a sliding shaft is not adequate. Such as a sample manipulator or Faraday cup. Secondly, where a device such as a detector or sample positioner is sensitive to vibration and must be decoupled from the vibration source.

# 10. Valves

Along the axis of the beam lines gate valves are used to enable the isolation of sections for maintenance and also to minimise loss of vacuum in the whole accelerator if one section develops a leak. Most gate valves are electro-pneumatically operated and many are interlocked to local high vacuum gauges. The few that are manually operated are for maintenance isolations.

On ANTARES the electro-pneumatically operated valves are interlocked to close when the pressure, as measured near the gate valve, rises above 5 x  $10^{-5}$  Pa. This figure can vary depending on the location of the valve. See the Accelerator WIKI for values around the ANTARES. Once an interlock has tripped the switch that operates the gate valve is bypassed. To reset the valve:

- The vacuum must be within the high vacuum range as read on the corresponding vacuum gauge controller with a pressure better than the trip point.
- Once this level is reached the bypass button is held down momentarily.
- At this point the pressure will rise then fall as the gas load is pumped away.
- When the system pressure is less than the trip level the bypass switch can be let go.
- An indicator light near the switch will show its status.

#### Notes:

- Operators and technicians must be aware that gate valves must be orientated so that the gate always seals towards the vacuum pump. That is, they seal best in one direction only. Failure to orientate the gate the correct way may cause a vacuum leak when atmospheric pressure forces the gate off its seal.
- In some cases gate valves may remain sealed after they have been switched to open. This can be due to the withholding (sometimes called back pressure) pressure not being adequate enough to lift the gate off the seal.
- Gate valves, while mostly metal can become hot if a gate is closed in front of an ion beam. If left for long periods with high current beams the elastomer seals may warm up and begin to outgas.
- For roughing ports typically 25KF bellows sealed valves are used. On STAR these same valves are used to isolate vacuum gauges for cleaning.
- Where fine flow control is required for either roughing out or venting, metering valves are used. These are basically multi-turn needle valves for fine adjustment.
- Valves do require maintenance to keep the seals clean and in working order. After long periods of time the 'o' ring seals will deform and often will not recover their original shape.

There have been attempts to build fast acting gate valves for the purpose of isolating the accelerator tank's load of  $SF_6$  in the event of a breach in a tube. While it seemed like a good idea the force of the valve closing at high speed could damage the gate seal causing the valve to leak. Designers must use care to ensure that a standard high differential pressure rated gate valve isn't adequate before launching into these expense type valves.

# **11. External Interfaces**

# **11.1 Electrical Feedthroughs**

a) Low Voltage, Low Current

There are few applications where voltages greater than 24Vac are used within the vacuum space. For these voltages at a few amps coaxial feedthroughs such as BNC, SHV, MHV, etc, are often used in place of more expensive dedicated commercial types. In one application 200Vdc is used for faraday Cup suppressors but current is only a few micro-amps at most.

Notes:

• Do not use multi-pin signal feedthroughs to carry voltages unless the pins used for voltage can be a few unused pins away from used signal pins.

- Feedthroughs can be purchased is almost any flange style but are usually restricted to small size flanges.
- Insulation must be used on all wires carrying voltage. It is highly recommended to use single or stranded wires (preferably nickel coated) with loose fitting PTFE (Teflon) spaghetti.

# b) Signal

Signal feedthroughs other than coaxial such as BNC, SMC, Microdot, etc are from one to multiple pins. It is recommended that connections to multi-pin feedthroughs are by the use of slide on pins. Many are either sliver of gold plated for good electrical contact. Avoid soldering unless care is used to remove all traces of flux and no pin-holes in the solder have been created

On coaxial feedthroughs the coaxial connector will be either both sides of the feedthrough and hermetically sealed or with the coaxial connector on the atmospheric side and a plain pin on the vacuum side, also hermetically sealed. For either type, where coaxial cable must continue into the vacuum space it is recommended to remove the outer plastic insulation and replace it with loose fitting PTFE spaghetti. This is also recommended for all single or stranded wires carrying signals.

# c) High Voltage, High Current

There is a wide range of high voltage and high current feedthroughs available in most flange types. High voltage feedthroughs usually have a larger insulator in ceramic and sometimes glass. These must be kept clean on the atmospheric side. Long term breakdown on high voltage feedthroughs can damage the insulator causing vacuum leaks.

For high current applications such as heaters, dedicated high current feedthroughs must be used. These are characterised by larger cross section wires to carry the higher currents. Like HV feedthroughs they must be kept clean. It is recommended to clean the insulators on signal feedthroughs periodically to remove any accumulation of dust.

# d) Power

There are few feedthroughs designed for powering devices at mains voltages. It is highly recommended that experiments are designed not to have mains voltages in vacuum vessels or equipment due to safety concerns.

# **11.2** Mechanical Feedthroughs

# a) Rotary

Rotary feedthroughs allow the transition of rotary movement into the vacuum space to, for example, operate shutters or position samples or detectors. They are widely available in many of the common flange styles. There are three common types both of which are very good in high vacuums.

The first type is a continuous shaft between the atmospheric side and vacuum side using rubber vacuum seals in the form of either supported 'o' rings, square or odd shaped rings (like common shaft seals), and flat rubber sheet seals. These are best for general rotary actuation rather than continually rotating. Okay in high vacuum but care must be taken to keep the seals clean and periodically replaced or whenever wear is noticed.

The second type is a ferro-fluidic type which also uses a continuous shaft between the atmospheric side and vacuum but the seal is made by encapsulating a dense fluid around the shaft. These types usually have bearings to keep the shaft centred and free moving. They are best for high revolutions in high vacuum applications.

The third type is magnetically coupled rotary feedthroughs. These are expensive. They provide a seal by magnetically coupling the shaft on the atmospheric side to the shaft on the vacuum side across a continuous sealing plate. If either shaft is removed the vacuum is not compromised. These are excellent for ultrahigh vacuums and lower. They are for moderate speed revolving applications and general actuations.

# **b**) Sliding

Usually these feedthroughs are a round shaft that can slide in and out of the vacuum as well as rotate. Sliding feedthroughs are used for positioning devices or where a linear actuation is necessary. The length can vary from a few millimetres to hundreds. In either case there is a shaft which penetrates from the atmospheric side to the vacuum side. The type of seals used varies like in the rotary seal. Sometimes dual seals are used to minimise leakage especially in high actuation rate applications. In some applications the void between the two seals is pumped increasing the reliance of the seal. These types are good in high vacuums. Notes:

- Care must be taken to ensure the shaft is lubricated with a suitable vacuum oil or grease. It is imperative that the oil or grease on the shaft is changed regularly to minimise dust build.
- There are bellows sealed sliding seals available. These types do not rotate and can only move in and out a few tens of centimetres at most. Very good in high to ultrahigh vacuums.
- Some sliding seals are able to move radially which will affect alignment. Where alignment is crucial a linear bearing type is recommended.

# c) Wobbler

A wobbler feedthrough allows users to use a shaft as a lever or pick up. They are designed around a flexible rubber or bellows seal they allows a greater range of circular movement.

# **11.3 Manipulators**

These are a class of feedthroughs design for high to extremely high precision positioning of samples, detectors or other instruments. They can be purchased having all or either X, Y, Z and rotary movement. The X and Y axes can have various travel lengths however they are typically less than 100 mm each. The Z axis (vertical) can have greater lengths but is limited as with the X and Y axes by the physical limitations of the vacuum bellows.

# 11.4 Actuators

'Actuators', combine sliding and rotary feedthroughs with pneumatic, electro-pneumatic or mechanical actuators to do the work.

In the accelerator area, actuators can be found on Faraday Cups to insert the cup into the beam line on a bellows sealed sliding feedthrough and inside of the ANTARES pressure vessel to move the stripper tube in and out of the beam axis.

# 11.5 Load locks

Load locks are not common in the accelerator area but are ideal for moving samples into a high vacuum space without significant loss of vacuum pressure. A sample or device is mounted onto the end of the load lock shaft. It is inserted into a tube connected to a chamber but isolated from the chamber vacuum by an in-line gate valve. The tube is sealed and then evacuated to a vacuum pressure similar to the chamber. Once the pressures equalise the gate valve is opened and the shaft pushed into the vacuum space. From this point the sample or device can be removed from the shaft by other remote means.

# **12. Internal Devices**

There are many devices that are commonly used inside of vacuum systems. Not all are represented in this section however similar reasons are use to assess new and unusually devices for use in vacuum.

# 12.1 Lighting

Lighting is necessary in most sample measurement chambers where the operator must be able to see the sample in order to position it in front of the ion beam. In most cases a standard bare 12Vac halogen bulb is used with good results. Care must be taken to ensure the bulb can be dimmed as when running at maximum power the heat load can only be dissipated through the connecting wires. That is, there is little gas inside a vacuum to conduct head away from the bulb. Bright white LED's are also used with mixed success. They have less radial light output as compared with a bare halogen bulb. If the LED is setup correctly the light can be focussed towards the necessary target.

Preferably, wires must be stranded or solid wire with a loose Teflon sleeve but normal plastic coated hook up wire works in high vacuums. In some cases where the wires as not likely to come into contact with other components then they are used uninsulated. All wiring must be restrained so that it cannot move into the path of the ion beam. As a rule-of-thumb wires should be at least 20 mm from the beam.

A standard double BNC feedthrough can be used, that is, only the centre core connections are used. There are many off-the-shelf electrical feedthroughs available that will be more suited for this specific use.

# 12.2 Heating

Providing power to a heating stage on a sample (target) stick requires a high current feedthrough. If a feedthrough isn't provided from the supplier of the heating stage then many types can be found from larger vacuum suppliers. Ensure the feedthrough selected is designed to carry the maximum current that can be supplied and not the maximum current likely to be used. Like in the lighting section above, care must be taken to ensure wires are insulated and away from the beam path.

Heating stages rely on heat dissipation through the support for the stage which is usually made from stainless steel, a relatively poor conductor. When opening devices for handling be sure they are cool to touch first.

# 12.3 Cooling

Cooling stages come in 2 distinctly different types. The first is the Peltier cooled stage which is all electric. Powering the stage can be managed as for lighting and heating. The second type is a liquid nitrogen cooled system. It uses an insulated tube to carry LN2 through a feedthrough into the vacuum space. A typical design is where LN2 is pumped through a coil attached to a heavy copper braid. The braid is then connected to the cooling stage where heat is conducted away from the mounting stage. Thermal isolation of the cooling stage from the holder can be Teflon or sapphire standoffs or similar materials.

These cooling devices must only be switched on when the vacuum is at the working level otherwise condensation and possibly ice can form on their surface becoming a large outgassing problem. Likewise ensure the stage is close to room temperature before opening up the vacuum system to atmospheric pressure. Always vent with a dry gas.
# **13. Pumping**

#### 13.1 General

It is not the intention of this document to describe the fundamental operation of particular vacuum pumps. Adequate information is available from manufacturer's catalogues. The following is information on what type of systems are used in the accelerator area and the reasons why.

Vacuum pumps don't suck. They act essentially as one way valves in a system where gas is flowing towards them. This is discussed in Chapter 3 of this document. There effectiveness is there ability to stop recoiling gas molecules returning into the vacuum space such as a beam line or vacuum chamber.

When considering what type of pump to use for a vacuum system the following points must be considered.

- What is the application?
- Will it have a relatively high gas load, that is;
- Will it be pumping a static gas load or changing gas load?
- Will there be a high outgassing rate?
- Are there known hidden/trapped voids to be pumped?
- It is a clean or dirty device? Is it contaminated with grease and oil?
- What pumping speed will be needed to maintain a high vacuum?
- What is the volume of the system? What is the conductance of the system? Will a small pump be just as effective as a large pump?
- What gases will be pumped?
- What type of pumps and how many are being used in the vicinity?
- Will the pumping speed need to be regulated such as for pumping near foils or delicate samples, detectors, etc?
- Cycling time whether it will be required to regularly pump from low vacuums
- Will the pumps operate reliably in their chosen application?
- Are they compatible with other pumps used on the accelerators? Are they cost effective?

Another consideration is vibration. Pump vibration can be significant leading to micrphonic interference in detectors and vibration of samples being measured. Vibration can be reduced by selecting pumps with less inherent vibration like turbomolecular pumps or the more fickle ion pumps. If a pump is a source of vibration it can be decoupled from the detector or measurement chamber by the use of a flexible bellows. An fine edge welded bellows is recommended.

#### **13.2 High Vacuum Pumps**

See section 4.5, Pump Down characteristics for cryogenic and turbomolecular vacuum pumps.

#### <u>Cryopumps</u>

For ANTARES the main beam line pumps in use are cryopumps. The types of pumps trap (or immobilise) gas molecules. They are used because they have a long lifetime between major

services, can be regenerated without removing them from service, have a high pumping speed for most gases and can maintain high vacuums for long periods in a static vacuum application.

#### Turbomolecular pumps

Where a vacuum system is cycled such as at a measurement chamber, then turbo pumps are used. Turbo pumps are transfer type pumps meaning, gas is transferred from the vacuum space into the atmosphere. A turbo pump can routinely recover a vacuum from low vacuum after a measurement chamber has been roughed out without needing regeneration as for a cryopump.

Turbo pumps are also more effective at pumping lighter molecules such as helium and hydrogen as compared with cryopumps. Using turbo pumps near systems that use these gases will reduce the effect of loading on cryopumps.

Most new turbo pumps can be serviced in-house. Some models purchased require special balancing of the bearings after they had been changed. In this case they are sent back to the manufacturer at great cost. It is important that models chosen can be maintained in-house.

#### Other high vacuum pumps

There have been few applications where titanium sublimation or ion pumps have been used in the accelerator area. Most have been stand alone UHV systems or cryostats for specialised applications. The practicality of cleaning and regenerating these gas trapping type vacuum pumps on systems requiring relatively frequent cycling is prohibitive.

Getter materials once used widely in electronics in tubes are becoming more used within larger static volumes such as beam lines and associated equipment. They can be coated onto surfaces to assist in maintaining uniform high vacuums in spaces that have poor pumping speeds.

#### **13.3** Low Vacuum, Roughing or backing pumps

#### Oil free pumps

These pumps are categorised by either being an oil type pump or dry (oil free) pump. Oil free vacuum systems are of choice for accelerator systems now that the technology has been proven to be reliable and cost effective. The majority of roughing and backing pumps now in use are scroll pumps as well. Piston pumps have been trialled with little success due to the short life of the seals. Some applications have used diaphragm pumps and multistage roots pumps but scroll pumps remain the most cost effective and easiest to maintain option. The Edwards type scroll was chosen over other brands as this type of scroll pump is designed with the bearings mounted outside of the vacuum which in other brands often caused the bearings to fail from lubricant loss.

#### Oil vane pumps

Low Vacuum, Roughing or backing pumps are all the same type of pump. Oil vane type pumps were widely used in the accelerator area until 2006 when a systematic change began on the STAR accelerator to replace oil vane pumps with oil free scroll pumps. Some oil vane pumps remain in use where some back streaming oil vapour is not considered a problem. If an oil vane pump is to be used for roughing out purposes, it is high recommended that a liquid nitrogen cold trap is used to trap vapours. It is the ultimate aim to have oil free pumping on all accelerators in the future.

# 14. Monitoring

#### 14.1 Vacuum Gauges

The standard type of high vacuum gauge in use is the cold cathode penning type gauge.

- On ANTARES the Pfeiffer brand is in use in two versions; the standard cold cathode type and full range type which has an integrated Pirani gauge.
- On STAR the most widely used brand is Leybold.

Generally, vacuums are measured as an indication only for process controlling and establishing basic quality to ensure pressures are within levels that are accepted for "normal" operation. This varies depending on location and use of the vacuum system for example a measurement chamber is cycled often and affects surrounding vacuum systems. It will more than likely be a much high pressure than surrounding static systems.

In most cases, absolute vacuums are not required to be measured. However on some AMS beam lines where thin windows are used it is important to have accurate measurement of pressures when cycling pressures in the gas detectors. If accuracy is required it must be remembered that different gases ionise at different pressures and so pressures readings from the same volume will be different depending on the dominant background gas. Most gauges have characteristics related to Nitrogen (air).

#### **14.2 Gauge Controllers**

On ANTARES the most widely used gauge controller is the Pfeiffer TPG300. This is an aging model and is being replaced in some key areas with the Pfeiffer Maxi-gauge which allows 6 heads to be connected at once.

The TPG300 has inputs for up to 4 heads whether Penning or Pirani. The limitation with the TPG300 is the electrical robustness when operated near the accelerator high voltage generator. Many failures have been attributed to high voltage transient surges. The TPG300 is now technologically aged and will ultimately be replaced outright. Various other models of TPG's are in use on ANTARES especially where single gauges are required. It is the TPG300 controllers on ANTARES that provides the interlocking capabilities.

The MAXI gauge is still being trialled as a replacement to the TPG300. A unit under test for many years at the HE end of the accelerator has already failed during an accelerator sparking event. Its real place may be away from the accelerator where the 6 heads allow consolidation of a whole beam line's vacuum monitoring system.

On STAR the common high vacuum gauge is the Leybold cold cathode type. It has been integrated into the STAR control system via cat 5 cable. They operate using a similar principle of measuring current flow across the plasma of the ionising gas. These gauges have been far more unreliable than the Pfeiffer equivalent. Unfortunately they are not interchangeable.

If venting with gas other than air or nitrogen be aware that the response on the Pirani gauges may show lower pressure than actual pressure.

#### 14.3 Bourdon Gauges

Bourdon gauges are normally used for indicating that pressure is below atmospheric. They are not used on accelerator systems as they have poor accuracy in the range of interest i.e., below  $1 \times 10^{-3}$  Pa.

# Chapter 3



# **Overview of Basic Vacuum Technology**

The ANSTO accelerators consist of 140 metres of beam lines under vacuum. Supporting these beam lines are 60 individual high vacuum pumping stations. There are over 70 isolation zones along the beam lines to allow isolation of a zone without interrupting the adjacent zones. This facilitates isolations to repair leaking sections, routine maintenance and cycling of pressure in zones to change samples, etc.

The following information are the general tools used by technicians and engineers to design reliable and high performance vacuum systems for the accelerator facilities.

# 1. Vacuum Overview

#### 1.1 What is vacuum?

A vacuum is defined as a diluted gas, or the corresponding state at which its pressure or density is lower than that of the ambient surrounding atmosphere.

Due to the Earth's gravity, atmospheric pressure varies with altitude. As a standard atmospheric pressure at sea level  $p_o = 101.325$  kPa. A Pascal is unit of vacuum relating to a force of newtons per metre squared.

An absolute vacuum of 0 kPa is not practically achievable so in real terms we can only approach zero for example on the ANSTO accelerators we have "operating" vacuums better than  $1 \times 10^{-4}$  Pa. In outer space the vacuum is less than  $10^{-12}$  Pa but not 0 Pa as there are some molecules of gas present which exert a very small partial pressure.

The pressure exerted from a vacuum lies between 0 to 100 kPa (see table below). In an ideal pumped vacuum system the pressure will continue to decease approaching 0 kPa. In accelerator systems the working ranges are medium to ultra high vacuums. In some special cases ion sources may operate in the low vacuum range but generally vacuum systems for the majority of accelerator systems are in the high vacuum range with some vacuum end stations close to ultra high vacuum.

#### 1.2 Units used

The SI unit  $Pa = N/m^2$  or kg·m<sup>-1</sup>·s<sup>-2</sup>, will be used where any vacuum quantity is referenced. In the accelerator area the vacuum units used are in Pascals (Pa) and although the vacuum pressures are below atmospheric pressure "negative" pressures are not represented as -kPa but of a magnitude which approaches absolute zero using scientific notation, e.g. 1 x 10<sup>-6</sup> Pa.

#### **1.3** Vacuum categories

Vacuums used in accelerator applications vary in magnitude from low vacuum to ultra high vacuum. These categories describe the pressure ranges which in the vacuum world become references for vacuum quality in a given system. Low vacuum is managed differently from high and ultra high vacuums due to the physical limits of the system designs. Generally, a vacuum system must be designed with the desired ultimate vacuum in mind to ensure the most appropriate materials, seals, pumps and vacuum management are used.

The table below shows each of the ranges based on the European system. This is the closest to that used for the ANSTO accelerators. The shaded section represents the working ranges used throughout the accelerator for example, ion sources may have low to high vacuums, beam lines may have very high vacuums and some endstation vacuum chambers may use high to very high vacuums. This document describes the design factors and particular management controls for achieving vacuums in the various ranges.

Range	Pressure (Pa)
Atmospheric pressure	1.013 x 10 <sup>5</sup>
Low vacuum	$1 \times 10^5$ to $3 \times 10^3$
Medium vacuum	$3 \times 10^3$ to $1 \times 10^{-1}$
High vacuum	$1 \times 10^{-1}$ to 1 x $10^{-4}$
Very high vacuum	$1 \ge 10^{-4}$ to $1 \ge 10^{-7}$
Ultra high vacuum (UHV)	$1 \ge 10^{-7}$ to $1 \ge 10^{-10}$
Extremely high vacuum	< 1 x 10 <sup>-10</sup>
Outer Space	$< 1 \times 10^{-12}$
Perfect vacuum	0 Pa

Table 2. Reference pressure ranges (European scale).

A. Berman, Vacuum Engineering Calculations, Formulas, and Solved Exercises, Academic Press, Inc. (1992).

In the accelerator area:

Low Vacuum = atmospheric pressure to medium vacuum

High Vacuum = high vacuum to very high vacuum

Ultra high vacuum = all those equal to and higher than ultra high vacuum

## 2. Pressure Fundamentals

#### 2.1 General

To understand the reason why vacuum systems must be carefully engineered to achieve high vacuums consideration must be given to how gases act in a closed chamber.

Any gas enclosed within a volume (a chamber or pipe work) is uniformly distributed or moving towards uniform distribution as in the case of accelerator tubes connected to stripper gas enclosures. The individual gas particles are constantly moving back and forth at high-speed within the volume; upon striking the vessel wall, they exert a force F on surface A due to pulse transmission. The pressure p that is exerted on the wall is defined as:

$$p = F/A$$

If the gas is made up of different types of gases, each of these gases will exert a pressure that corresponds to its concentration; this is called <u>partial pressure</u>. The sum of all partial pressures equals the total pressure. Air is a good example of this: In addition to its main constituents of nitrogen, oxygen and water vapour, air also contains many trace gases, the sum of which equal  $\sim$ 101.3 kPa, total pressure (atmospheric pressure). See table below.

In an accelerator system it would not be uncommon to have various carbon based compounds, sulphur hexafluoride and specific stripper gases present in trace amounts.

Gas	% by volume	Partial Pressure (Pa)			
Ar	0.92	934			
CO <sub>2</sub>	3.26 x 10 <sup>-2</sup>	33			
H <sub>2</sub>	4.9 x 10 <sup>-5</sup>	4.94 x 10-1			
He	5.16 x 10 <sup>-4</sup>	5.23 x 10-1			
Kr	1.1 x 10 <sup>-4</sup>	1.15 x 10-1			
$N_2$	77.17	78180			
Ne	1.8 x 10 <sup>-3</sup>	1.82			
O <sub>2</sub>	20.7	20970			
Xe	8.59 x 10 <sup>-6</sup>	8.7 x 10-3			
H <sub>2</sub> O	1.18	1200			
Other	remainder	remainder			
Total	100 %	101325 Pa			
Notes: H <sub>2</sub> O may vary depending on environment					
"Other" may include: $CH_4$ , $O_3$ , $N_2O$					

Table 3. Partial Pressures in AirThe Vacuum Technology Book – Pfeiffer Vacuum September 2008

#### 2.2 Ideal (General) gas equation

1 mole of any gas at STP occupies 22.414 litres. At a temperature 273.15 K (0 °C) and a pressure of 101,325 Pa (standard pressure) 1 mole of any gas contains 6.02 x  $10^{23}$  particles. This is referred to

as Avogadro's number. The mass of the gas thus enclosed is its molecular weight in grams. The ideal gas equation describes the state of a gas as a function of pressure, temperature and volume.

$$pV = nRT$$

Example:

For a chamber, Ø300 mm x 500 mm long, a volume of 0.035 m<sup>3</sup>, calculate the mass of gas in the chamber at a vacuum of 1 x  $10^{-6}$  Pa.

pV = nRT

n = pV/RT  $n = 1 \times 10^{-6} \times 0.035 / 8.31451 \times 293 = 1.43 \times 10^{-11} \text{ moles}$ Mass of remaining volume = moles x mass = 1.43 x 10<sup>-11</sup> x 29 (air) = 0.415 ng No. of molecules = moles x Avogadro's Number = 1.43 x 10<sup>-11</sup> x 6.02 x 10<sup>23</sup> = 8.6 x 10<sup>12</sup> Per cm<sup>3</sup> = 8.6 x 10<sup>12</sup> / 35000 cm<sup>3</sup> = 246 x 10<sup>6</sup> molecules per cm<sup>3</sup>

Where:

 $p = \text{pressure (Pa)}, V = \text{volume (m}^3), n = moles, R = \text{general gas constant} = 8.314510 \text{ kJ/(kmol K)}, T = \text{thermodynamic temperature (K)}, Avogadro's number = 6.02 x 10^{23}$ 

#### **3.** Flow Fundamentals

#### **3.1** Mean free path $(\lambda)$

The mean free path is the mean path length that a molecule traverses between two successive impacts with other molecules. High vacuums provide a longer mean free path than low vacuums. In accelerator systems high vacuums are crucial as ions that collide with gas molecules in beam lines loose energy and are not transported to the point of measurement. In the accelerator tubes high mean free paths are crucial to ensure low energy loss in the ion beam and optimised ion beam transmission.

Gas (at 0°C and 1 atm)	$\lambda$ (nm)
Air	68
Ar	62.6
Cl <sub>2</sub>	27.4
CO	58.6
CO <sub>2</sub>	39
$H_2$	110.6
Не	173.6
Kr	36
$N_2$	58.8
Ne	124
<b>O</b> <sub>2</sub>	63.3
Xe	26

Table 4. Mean free Paths for various gases

Hirschfelder, Curtiss and Bird (1954) Molecular Theory of Gases and Liquids, Wiley, New York

Pressure (Pa)	Molecules / cm <sup>3</sup>	Molecules / m <sup>3</sup>	Mean free path
101325	$2.7 \times 10^{19}$	$2.7 \times 10^{25}$	68 nm
	<b>—</b> 11 <b>—</b> 31	1	

Table 5. Other characteristics of Air

#### 3.2 Mean Velocity (u) m/s

The residency time of various gases in a system (or the time taken to remove various gases) relates to their individual mean velocity. The following table shows mean velocities for some gases. It can be seen that very light gases travel significant faster than heavy gases. It should be noted that Hydrogen and Helium do not pump efficiently in turbo pumps or cryopumps but heavy gases generally do.

Gas	Molar Mass kg/mol	Mean Velocity m/s	Mach Number
Air	0.028966	463	1.4
Ar	0.039948	394	1.2
Cl <sub>2</sub>	0.0709	296	
СО	0.028011	471	
CO <sub>2</sub>	0.04401	376	1.1
H <sub>2</sub>	0.002016	1762	5.3
He	0.00402	1246	3.7
Kr	0.0838	272	
$N_2$	0.02801	471	1.4
Ne	0.020179	555	
O <sub>2</sub>	0.03199	441	
Xe	0.1313	217	
H <sub>2</sub> O	0.01802	587	1.8

Table 6. Mean Velocity for gases at 20°C

The Vacuum Technology Book – Pfeiffer Vacuum September 2008

$$u = \sqrt{\frac{8.R.T}{\pi.M}}$$

Where: *M* = Molar mass (kg/mol)

For example: Mean velocity of Argon @  $20^{\circ}$ C u =SQRT (8 x 8.31451 x 293 / 0.039948 x 3.1416) u =394 m/s

#### 3.3 Types of flow

The different flow phases need to be understood in order to select the appropriate pumping system for a particular application. It may be that in most cases a chamber or other piece of equipment is

pumped at the highest flow rate achievable with a given pump but it could be the case for example where a chamber has delicate films inside and gas movement past the foils has the potential to tear them apart. In this case a low steady flow rate is needed.

The flow phases in a vacuum system characterise the gas molecule movement related to pumping speed in a vacuum system. Factors that determine flow include pressure differentials, mean free paths, gas type, geometry, dimensions of the system and temperature. There are many different nomenclatures used to represent flow phases but 3 types in particular are used in the accelerator area, Turbulent, Laminar (both in Viscous phase), and Molecular. Less emphasis is placed on the intermediate or transitional flow as once this state is reached it is assumed that the system is pumping well. At atmospheric pressure up to about 100 Pa, the mean free path of the gas molecules is very small. Therefore, the gas flow is limited by the viscosity of the gas being pumped so the type of flow is called Viscous.

In an *ideal* system, if a roughing pump has a pumping speed of say 22 m<sup>3</sup>/hr and there is negligible flow resistance between the pump and the chamber then a chamber of  $\emptyset$ 300 mm ID x 500 long (a volume of 0.35 m<sup>3</sup>) at atmospheric pressure, will take approximately 1 minute to remove the bulk of the gas. During this time the flow remains in the [Viscous] Laminar and/or Turbulent phase.

Where a system has a leak, depending on the magnitude of the leak it is possible that the pumping will plateau at either the Turbulent, Laminar or Molecular flow phase. With outgassing systems the less referred to transitional state may be reached and with systems designed to reach ultra high vacuums outgassing may be occurring well into the molecular flow state.

Pumping	Flow	Phase	Description	Type of vacuum	Flow rate Pa-l/sec
Pumping begins		Turbulent Flow	Initial flow experienced at start of pumping or in a low vacuum environment where the flow rate is high. Chaotic movement of molecules. Eddies and other non-parallel flows occur.	Low Vacuum	>1.01
	Viscous	Laminar Flow	Initial flow experienced at start of pumping or in a low vacuum environment where the flow rate is low. Gas molecules bouncing off other gas molecules (gas-gas) in the vacuum system. Short mean free path. Molecules streaming from system.	Medium Vacuum	10.1 to 1.01 x 10 <sup>-4</sup>
	Transit (Kn	ional Flow udsen)	Intermediate flow between laminar and molecular. Flow is dominated by both gas-gas and gas-wall collisions.	Medium to High Vacuum	1.01 x 10 <sup>-2</sup> to 1.01 x 10 <sup>-5</sup>
Base vacuum reached	Molec	ular Flow	Remaining gas molecules bounce off the walls (gas-wall) of the vacuum system and not each other. Long mean free paths. Flow rate very small.	High to Ultra-High Vacuum	<1.01 x 10 <sup>-5</sup>

Table 7. Vacuum flow phases

By evaluating the Knudsen (Kn) and Reynolds (Re) number, one can predict the various flow phases. These phases can be seen in the picture below which also shows how the gas molecules behave in the different flow phases moving through the opening d.

#### 3.4 Knudsen Number (Kn)

The Knudsen number is a dimensionless number defined as the ratio of the molecular mean free path length to a representative physical length scale. The Knudsen number in a viscous flow phase < 0.01 and molecule-molecule collisions dominate gas behaviour which behaves as a fluid. In molecular flow Kn > 1 and molecule-surface collisions dominate.

$$Kn = \frac{\lambda}{d}$$

 $\lambda$  = Mean free path

d = diameter of the opening that gas will pass through

#### 3.5 Reynolds Number (Re)

Reynolds number indicates whether the flow of a gas is absolutely steady (laminar flow) or on average steady but with small, unsteady changes (turbulent flow). The Reynolds number, Re, has no dimensions and is defined as the size of the flow.

Osborne Reynolds demonstrated in 1883 that the change from laminar to turbulent flow in a pipe occurs when the value of the Reynolds number exceeds 2,100. The exact value of Re for which the flow changes from laminar to turbulent depends on the geometry of the component, its surface roughness and other experimental factors. During evacuation of a vessel, turbulent flow normally occurs only for a short period of time at the beginning. It has been found to be approximately proportional to the root of the pressure gradient.

$$\operatorname{Re} = \left(\frac{\rho}{\eta}\right) d.v$$

 $\rho$  = gas density (kg/m<sup>3</sup>)  $\eta$  = viscosity (Pa.s) (Pascal-second) v = flow velocity (m/s) d = tube diameter (m)

Gas	Formula	Molecular weight	Gas Density [2] kg/m <sup>3</sup>	Viscosity η Pa.s
Air [1]	$N_2 + O_2$	29	1.1839	1.79 x 10 <sup>-5</sup>
Ammonia	NH <sub>3</sub>	17.031	0.7449	1.01 x 10 <sup>-5</sup>
Argon	Ar	39.948	1.723	2.24 x 10 <sup>-5</sup>
Carbon Dioxide	$CO_2$	44.01	1.9105	1.51 x 10 <sup>-5</sup>
Carbon Monoxide	CO	28.01	1.2082	1.74 x 10 <sup>-5</sup>
Chlorine	Cl <sub>2</sub>	70.906	3.1124	1.34 x 10 <sup>-5</sup>
Helium	He	4.02	0.171	1.99 x 10 <sup>-5</sup>
Hydrogen	$H_2$	2.016	0.0868	8.80 x 10 <sup>-5</sup>
Hydrochloric Acid	HCl	36.5	1.5844	1.46 x 10 <sup>-5</sup>
Hydrogen Sulphide	$H_2S$	34.076	1.4876	1.26 x 10 <sup>-5</sup>
iso-Butane	$C_4H_{10}$	58.12	2.3758	7.49 x 10 <sup>-5</sup>
Methane	$CH_4$	16.043	0.6556	1.11 x 10 <sup>-5</sup>
Nitrogen	$N_2$	28.02	1.2088	1.76 x 10 <sup>-5</sup>
Oxygen	$O_2$	32	1.381	2.02 x 10 <sup>-5</sup>
Propane	C <sub>3</sub> H <sub>8</sub>	44.09	1.8024	8.26 x 10 <sup>-5</sup>
Sulphur Hexafluoride	$SF_6$	146.5	6.27	$1.53 \times 10^{-5}$

Table 8. Gas density and viscosities

Unless otherwise indicated gases referenced from McGraw Hill Chemical Properties Handbook 1 atm 25°C. [1] Air STD Atmosphere at Sea Level: 1 atm 15°C [2] Gas Density Values Interpolated From 15°C Data using Charles Law

http://pipeng.com/index.php/gsts/itdmodflup002a/itddaflup00201

#### 3.6 Viscous Flow

#### Low vacuum, p = 100000 - 100 Pa, where $\lambda \ll d$

What characterises viscous flows, is frequent contact between gas molecules, yet less frequent contact with the walls of the vessel. The mean free path of the gas molecules is therefore very small and the gas flow is limited by the viscosity of the gas being pumped (Viscous Flow). In this case, the mean free path of the gas molecules is significantly shorter than the dimensions d of the vacuum equipment. For both Laminar and Turbulent Flow, Kn < 0.01. In addition, the term viscous flow is used if the product of pressure p and diameter d of the components through which gas is flowing is p.d  $\geq$  60 Pa.cm for air. Whether the flow is in the Laminar or Turbulent flows phases can be calculated using the Reynolds Number formula above.

#### 3.7 Knudsen flow

#### Medium vacuum, p = 100 - 0.1 Pa with $\lambda \le d$

If the Knudsen number is between 0.01 and 0.5, this is termed Knudsen flow. Because many process pressures are in the medium vacuum range, this type of flow occurs with corresponding frequency. Since this is a transitional flow, this range is transited relatively quickly when pumping down vacuum chambers and hence not usually considered in accelerator systems. This means that the influence of this conductivity on pump-down times is correspondingly low. A simple approximation for the Knudsen range can be obtained by adding the laminar and molecular conductivities. The

table in "Conductivities" below shows the conductivities of round, one meter long tubes of differing diameters in all three flow ranges.

#### 3.8 Molecular flow High vacuum, $(p = 0.1 - 10^{-5} \text{ kPa})$ , where $\lambda > d$ and in UHV $(p < 10^{-5} \text{ kPa})$ , with $\lambda >> d$

At Knudsen numbers of Kn > 0.5 molecule-molecule collisions virtually no longer occur. What prevails is molecular flow. In this case, the product of pressure p and component diameter d is p x d  $\leq$  1.3 Pa.cm.



Fig 5. Molecular paths during different flow phases Re reference: http://www.engineersedge.com/fluid\_flow/pressure\_drop/pressure\_drop.htm

#### **3.9 Pump throughput qpV**

The concept of pump throughput is of major significance in practice and should not be confused with the pumping speed! The pump throughput is the quantity of gas moved by the pump over a unit of time, expressed in Pa.1.s<sup>-1</sup>. Conversely, the pumping speed is the capacity of the pump to remove a volume of gas within a specific unit of time, measured in  $m^3/h$  or 1/s.

The throughput value is important in determining the size of the backing pump in relationship to the size of a high vacuum pump with which it is connected in series in order to ensure that the backing pump will be able to take away the gas moved by the high vacuum pump.

The pumping capacity (throughput) for a pump is equal either to the mass (m) flow through the pump intake port in a specified time:

$$q_m = \frac{m}{t}$$

Or to the pV (quantity of gas) flow through the pump's intake port:

$$q_{pV} = \frac{pV}{t}$$

It is normally specified in Pa.l.s<sup>-1</sup>. Here p is the pressure on the intake side of the pump. If p and V are constant at the intake side of the pump, the throughput of this pump can be expressed with the simple equation:

 $q_{pV} = p.S$ 

Where:

S = pumping speed of the pump at intake pressure of p.

#### 3.10 Speed of vacuum pump S

The speed of a vacuum pump is defined as

$$S = \frac{q_{pV}}{p}$$

p = Pressure at the pump inlet

However, pumps are usually connected to vacuum chambers via hoses resulting in a lower effective pumping speed  $S_{eff}$  at the chamber. If  $p_1$  is the pressure at the pump and  $p_2$  is the pressure in the vacuum chamber then:

$$q_{pV} = S.p_1 = S_{eff}.p_2$$

for continuous flow. Combining this with  $q_{pV} = C(p_1 - p_2)$  (see Conductance below) results in an expression for S<sub>eff</sub>

$$S_{eff} = \frac{S.C}{S+C}$$

#### **3.11** Conductance C

In a vacuum system, the volume that is pumped by a vacuum pump exhibits a level of flow resistance (Z) due to chamber and pipe sizes and their geometries, and the various sized orifices and other in-stream devices. The terminology used to describe this effect is the reciprocal of flow resistance which is *conductance* (C) or the systems ability to conduct gas.

Conductance is expressed in either l/s or sometimes  $m^3/h$  and is usually for steady, continuous flow through one or more components in a vacuum system. It is affected by the geometry of the piping element and relative to the flow phase and capacity of the pump used. In the high and ultrahigh vacuum ranges (molecular flows), C is a constant which is independent of pressure. In the rough and medium-high vacuums (turbulent and laminar) it is, by contrast, dependent on pressure. As a consequence, the calculation of C for the piping (beam line) elements must be carried out separately for the individual pressure ranges.

Flow resistance (sec/litre) 
$$Z = \frac{P_1 - P_2}{q_{_{PV}}}$$

Conductance (litre/sec) therefore

 $C = \frac{1}{Z} = \frac{q_{_{PV}}}{P_1 - P_2} \qquad (continued over)$ 

$$q_{pV} = C(p_1 - p_2)$$

Where:  $q_{pV}$  = Throughput flow rate p = Pressure (Pa)

At the molecular level, the conductivity of a system is reduced due to the friction of both the walls of the system and other molecules. As discussed above in "Types of Flow", these two affects occur at different flow phases. The total effect of conductance is the sum of the various conductance elements. In the case where a valve, a trap and an elbow in series each having a different conductance, the sum is:

$$\frac{1}{C_{total}} = \frac{1}{C_{valve}} + \frac{1}{C_{trap}} + \frac{1}{C_{elbow}}$$

If the components with different conductances are connected in the flow path in parallel, for example, 2 different diameter tubes connecting a chamber to a pump then the equation will be:

$$C_{_{Total}} = C_{_{tube1}} + C_{_{tube2}}$$

For the design of vacuum systems used on the accelerators conductance is mostly calculated for systems in molecular flow.



*Fig 6. Diagram for estimating pipe conductance Pupp/Hartmann, Vakuumtechnik, Grundlagen und Anwendugen, Hanser Verlag* 

## 4. Limitations

The most fundamental problems with vacuum systems are leaks and outgassing. Leaks can be categorised as either real leaks where gas enters the vacuum system from outside of the vessel or leaks form trapped voids which is in effect an internal leak. Trapped voids can contain gas that will continue to outgas for long periods reducing the ultimate vacuum that can be reached in a vessel. Real leaks can be found with the aid of a helium leak detector whereas internal leaks from trapped voids may never be found.

Outgassing can be minimised through careful selection of materials used within the vacuum space. The appendices have helpful tables to estimate outgassing rates. Outgassing may be in the form of desorption, diffusion or vaporisation. Permeation is not outgassing as the source of gas originates from outside of the vacuum space.

Examples commonly used substances in the accelerator systems that outgas,:

- Teflon, PVC, Ertalyte
- Viton, neoprene
- Copper, aluminium, stainless steel, brass, tantalum, rubidium, caesium, lithium, zinc
- Vacuum pump oil, vacuum greases, vacuum epoxies

These are just a few but it demonstrates that all materials will outgas at some point when the temperature and vacuum pressure reach their individual vapour pressure.

Neither of the problems is resolved by increasing the pumping speed (capacity) of the vacuum pump. There will be a point where the rate of gas entering the system will equalise with the pumping speed of the pump. The rate may vary depending on the vapour pressure of the substance outgassing and the temperature of that substance.



Fig 7. Limitation of Pumping From: http://www.mse.ncsu.edu/WideBandgaps/classes/MSE%20751/Handouts/HO02\_Leakage.pdf



Fig 8. Unwanted gas source wheel

#### 4.1 Contamination

Vacuum chambers must be clean in order to reach the desired pressure as quickly as possible when they are pumped down. Typical contaminants include oil and grease on screws and seals, process reaction products or condensed vapours, particularly water that is adsorbed on the walls of the vessel. Consequently, it is necessary to ensure that the components are clean when installing vacuum equipment. All components attached in the vacuum chamber must be clean and grease-free. All seals must also be installed dry. If high or ultra high vacuum is to be generated, clean gloves must be worn during the assembly process.

Examples of common contaminants in accelerator vacuum systems

- Rotary pump oil
- Water
- Plasticisers from various plastics
- General airborne dust
- Machining oil
- Residual gases from stripper gases, venting gases, ion source gases
- Sample breakdown

#### 4.2 Condensation and vaporisation

All substances can occur in a liquid, solid or gaseous state. Their aggregate status is a function of pressure and temperature. Liquids are transformed into their gaseous state through vaporisation, solids through sublimation. The separation of liquids or solids out of the gaseous phase is termed condensation. Since normal room air contains approximately 10g of water vapour per m<sup>3</sup>, condensed water vapour is always present on all surfaces.

Adsorption on surfaces is especially pronounced due to the strong polarity of the water molecules. Natural fibres, in particular, such as paper, contain large quantities of water that escape during drying processes under vacuum. Cooled condensers are used to separate the water vapour in this connection. Even some metals (Cd, Zn, Mg) can vaporise in noticeable quantity at temperatures of several 100 °C. Consequently, use of these metals is avoided in plant construction.

#### 4.3 Desorption

In addition to water, other substances (oil) can be adsorbed on surfaces. Substances can also diffuse out of the metal walls, which can be evidenced in the residual gas. In the case of particularly rigorous requirements, stainless steel vessels can be baked out under vacuum, thus driving the majority of the volatile components out of the metal walls.

Gas molecules, (primarily water) are bound to the interior surfaces of the vacuum chamber through adsorption and absorption, and gradually desorb again under vacuum. The desorption rate of the metal and glass surfaces in the vacuum system produces a gas yield that is a function of time, however. A good approximation can be obtained by assuming that after a given point in time  $t > t_0$  to the reduction will occur on a linear basis over time.  $t_0$  is typically assumed to be one hour.



Fig 9. Desorption curves



Fig 10. Adsorption Curves From: http://www.mse.ncsu.edu/WideBandgaps/classes/MSE%20751/Handouts/HO02\_Leakage.pdf

#### 4.4 Diffusion with desorption

At operation below  $10^{-4}$  kPa, desorption of plastic surfaces, particularly the seals, assumes greater significance. Plastics mainly give off the gases that are dissolved in these plastics, which first must diffuse on the surface.

Following extended pump downtimes, desorption from plastics can therefore dominate the metal surfaces. Although the surface areas of the seals are relatively small; the decrease in desorption rate over time occurs more slowly in the case of metal surfaces. As an approximation it can be assumed that the reduction over time will occur at the square root of the time.

The gas produced from plastic surfaces can thus be described as:

Desorption from plastic material  $Q_{diff} = q_{diff} \cdot A \sqrt{t_o/t}$ 

Where Ad denotes the surface area of the plastics in the vacuum chamber and  $q_{diff}$  denotes the surface area-specific desorption rate for the respective plastic. At even lower pressures, similar effects also occur with metals, from which hydrogen and carbon escape in the form of CO and CO<sub>2</sub> and can be seen in the residual gas spectrum.

#### 4.5 **Permeation and leaks**

For a gas passing through small holes in a thin wall in the Knudsen Flow regime, the number of molecules that pass through a hole is proportional to the pressure of the gas and inversely proportional to its molecular weight.

To Convert to Leakage	Multiply Helium Leak Rate by:		
Rate of:	Laminar Flow	<b>Molecular Flow</b>	
Argon	0.88	0.316	
Air	1.08	0.374	
Nitrogen	1.12	0.374	
Water vapour	2.09	0.469	
Hydrogen	2.23	1.410	

Table 9. Conversation table for leak rates

Seals, and even metal walls, can be penetrated by small gas molecules, such as helium, through diffusion. Since this process is not a function of time, it results in a sustained increase in the desired ultimate pressure. The permeation gas flow is proportional to the pressure gradient  $p_0 / d$  (d = wall thickness,  $p_0 =$  atmospheric pressure = ambient pressure) and to the permeation constants for the various materials  $k_{perm}$ .

Permeation

 $k_{perm}$ .  $Q_{perm} = k_{perm}$ .  $A \cdot p_0/d$ 

Permeation first manifests itself at pressures below  $10^{-6}$  kPa.  $Q_1$ , denotes the leakage rate, i.e. a gas flow that enters the vacuum system through leaks at a volume of V. The leakage rate is defined as the pressure rise  $\Delta p$  over time  $\Delta t$ :

Leakage rate  $Q1 = (\Delta p \cdot V)/\Delta t$ 

If a vessel is continuously pumped out at a volume flow rate S, an equilibrium pressure  $p_{gl}$  will be produced. Throughput is equal to the leakage rate  $Q_l = S \cdot p_{gl}$ . A system is considered to be

adequately tight if the equilibrium pressure  $p_{gl}$  is approximately 10 % of the working pressure. If, for example, a working pressure of  $10^{-4}$  kPa is attained and the vacuum pump that is being used has a pumping speed of 100 I/s, the leakage rate should not be more than  $10^{-3}$  kPa I/s. This corresponds to a leak of approximately 20.20  $\mu$ m<sup>2</sup> in size. Leakage rates  $Q_l$  of less than  $10^{-6}$  kPa I/s can usually be easily attained in clean stainless steel vessels. The ultimate pressure achievable after a given period of time *t* primarily depends upon all of the effects described above and upon the pumping speed of the vacuum pump. The prerequisite is naturally that the ultimate pressure will be high relative to the base pressure of the vacuum pump.



Fig 11. Permeation of Different Gases From: http://www.mse.ncsu.edu/WideBandgaps/classes/MSE%20751/Handouts/HO02\_Leakage.pdf

#### 4.6 Leaks

The source of leaks is as wide and varied as can be imagined. There is no such thing as a common leak however there are several types generally seen. The following lists types in rough order of prevalence:

- Dirty seals elastomer type seals with traces of dust, dirt and fibres on the surface
- Jarred seals ill fitted flanges where the seal lays across the sealing surface
- Vacuum pumping tubes where a fitting has been fitted without care and the pump cannot reach its full capability
- Distortion over tightened or misaligned flanges and fittings that "lift" seals off the sealing surface
- Stress cracks usually in places around flanges and fittings that have been incorrectly tightened
- Pressure dependant mostly related to accelerator tubes that develop leaky seals allowing insulation gas to enter when the pressure is greater than atmospheric pressure.

#### 4.7 Bake-out

The following prerequisites must be satisfied in order to achieve lower pressures ( $<10^{-6}$  kPa):

- The base pressure of the vacuum pump should be a factor of 10 lower than the required ultimate pressure
- Stainless steel vacuum recipients and components must be used
- Metallic seals (CF flange connections) are required
- Leaks must be avoided and eliminated prior to activating the heater (use helium leak detectors!)
- Clean work is a must, i.e. all parts must be thoroughly cleaned and must be installed with grease-free gloves
- Pump and equipment must be baked out

Bake-out significantly increases desorption and diffusion rates, and this produces significantly shorter pumping times. Bake-out temperatures of up to 300°C are used. The instructions of the pump manufacturers relating to maximum bake-out temperatures and maximum permissible radiation levels in the pump flange must be observed.

Following installation the equipment is switched on, and after reaching a pressure of  $P < 10^{-3}$  kPa the heater is then switched on. During the heating process, all gauge heads must be operated and degassed at intervals of 10 hours. In the case of stainless steel vessels and the use of metallic seals, bake-out temperatures of 120°C and heating times of approximately 48 hours are sufficient for advancing into the pressure range of  $10^{-8}$  kPa. Bake-out should be continued until 100 times the expected ultimate pressure is attained. The heaters for the pump and vacuum chamber are then switched off. After cool-down, the desired ultimate pressure will probably be achieved. In connection with pressures  $P < 5 \times 10^{-8}$  kPa and large interior surface areas, it will be advantageous to use a titanium sublimation pump that pumps the hydrogen escaping from the metals at a high volume flow rate.

#### 4.8 Residual gas spectrum

When leaks have been solved in a vacuum system and poor vacuum persists than a residual gas analysis (RGA) measurement should be made to determine the composition of the gas load. This will give vital information on where the vacuum problem may be. Interpreting the information will be trial and error as users will have to think about all possible sources of gases (and outgassing) that may be present. Also relative ratios of gases should be considered to make sense of the measurement. Care should be taken especially if making a measurement near a source such as a cryopump. Some gases that have been trapped on the cryopump may be liberating from the cold surface and will make up a part of the gas spectrum. Other places to take care are near the accelerator tubes and ion sources.

If developing an ultra high vacuum system it is important to know all sources of gas otherwise moving lower than  $1 \times 10^{-6}$  Pa will be an unnecessary challenge. For all other systems it is nice to know but not essential unless residual gases affect ion beam measurements. The dominate gases (and their masses) that may be seen include:

Gas	Mass	Gas	Mass
Water	18	Carbon Monoxide	28
fragment HO	17	Carbon Dioxide	44
Nitrogen	28	Argon	40
Oxygen	32	Sulphur Hexafluoride	146
Hydrogen	2	Helium	4

Table 10. Table of masses for common "in vacuum" gases

# **Appendix 1 – Structural Calculations for Scientific Vacuum Vessel Design**

Calculations for Vacuum Vessel wall and end plate thickness

The following formulae are from the Vacuum Society of Australia (VSA) training course notes which have been in circulation since the early 1980's. VSA continues to support these calculations for training vacuum technologists around Australia. The calculations provide conservative estimates of vacuum vessel wall thicknesses as compared with formulas used in ASME Section VIII – Division 1 UG-28 (Thickness of Shells and Tubes under External Pressure) and associated documents, which have been developed for larger industrial type vacuum vessels.

#### Cylindrical Vessel – Stainless Steel @ 20°C

Thickness of plate in a given diameter (h):

$$\frac{D}{h} \le 105, \ \frac{h}{D} \ge \frac{1}{105}$$

Where: D = diameterh = thickness

Maximum length of vessel for a given diameter (L<sub>c</sub>):

$$\frac{L_c}{D} \le 11.5$$

Where: D = diameter $L_c = \text{length}$ 

#### **End Plates – Stainless Steel**

Thickness of plate in a given diameter (h<sub>1</sub>):

$$\frac{D_1}{h_1} \le 89$$
 ,  $\frac{h_1}{D_1} \ge \frac{1}{89}$ 

Where:  $D_1$  = diameter  $h_1$  = thickness

Minimum thickness for given deflection at centre:

 $\frac{h_1}{\delta} \ge 3$ 

Where:  $\delta$  = deflection  $h_1$  = thickness For Hemispherical End (h<sub>2</sub>):

$$\frac{R}{h_2} \le 830 \text{ requires } \frac{h_2}{R} \ge \frac{1}{830}$$

Where: R =radius  $h_2 =$  thickness

**Example 1:** To construct Stainless Steel vacuum chamber at  $20^{\circ}$  C as shown – Length L = 50 cm = 500 mm, D = 40 cm = 400 mm, Maximum allowable deflection at centre  $\delta = 1$  mm. Find  $h, h_1, h_2$  to satisfy vacuum chamber strength requirements.

#### For Cylinder

1. 
$$\frac{D}{h} \le 105$$
,  $\frac{h}{D} \ge \frac{1}{105}$   $h \ge 400/105$ ,  $h \ge 3.8 \text{ mm}$   
2.  $\frac{L_c}{D} \le 11.5$ ,  $L_c \le 11.5 \times 400$   $L_c \le 4600 \text{ mm}$  with  $L = 500 \text{ mm} \checkmark$ 

#### **For End Plates**

1. 
$$\frac{D_1}{h_1} \le 89$$
,  $\frac{h_1}{D_1} \ge \frac{1}{89}$   $h_1 \ge 396.2/89$ ,  $h_1 \ge 4.45 \text{ mm}$   
2.  $\frac{h_1}{\delta} \ge 3$ , with  $\delta = 1 \text{ mm}$  then require  $h_1 \ge 3 \text{ mm}$ , however  $h_1 = 4.45 \text{ mm} \checkmark$ 

#### For Hemispherical End

1. 
$$\frac{R}{h_2} \le 830$$
 requires  $\frac{h_2}{R} \ge \frac{1}{830}$   $h_2 \ge 198.1/830 \ge 0.24$ mm

Dimension of cylindrical, planar and hemispherical parts of vacuum enclosures

	At	Cylin	nders	End I	Plates	Hemispherical
Material	Temp (°C)	D/h	$L_c/D$	$D_1/h_1$	$h_l/\delta$	<i>R/h</i> <sub>2</sub>
Copper	20	84	10	52	15	600
Copper	500	58	8.5	-	-	-
Nickel	20	100	11	73	8	780
Nickel	500	90	10.5	-	-	-
Aluminium Alloy	20	70	9	37	57	470
Aluminium Alloy	500	62	8.7	-	-	-
Stainless Steel (304)	20	105	11.5	89	3	830
Stainless Steel (304)	500	89	10.5	-	-	-
Glass (hard)	20	70	9	16	117	470
Neoprene	20	2.5	1.7	10	0.2	30
Teflon	20	12	3.8	14	9	-
PVC (Tygon)	-	3.7	2.1	-	-	-
Perspex	-	_	-	30	-	-
Mica	-	_	-	58	15	-

Factor tables for thickness calculations of common materials used in high vacuum systems

 $\delta$  = maximum permissible deflection at centre of plate L<sub>c</sub> = critical length for cylinder = 1.11D(D/h)1/2

**Example 2:** Using the ASME Boiler and Pressure Vessel Code (the Code) Section VIII, Division 1 for the safe design pressures for a vacuum vessel is used to calculate the maximum external pressure acceptable for a given wall thickness. The following example uses the same vessel dimensions as in Example 1, with a wall thickness of 3.8 mm as calculated using the VSA method.

The calculations reference the Vacuum Vessel Cylindrical Shell Thickness using paragraphs UG-28, UG-27 of ASME Boiler and Pressure Vessel Code.

Vessel shell thickness t = 3.8 mm  $D_o = 400$  mm (outer diameter of shell)  $D_o/t = 105.26$  Since this ratio is greater than 10, follow UG-28(c)(1) L = 500 mm (length of stiffened shell)  $L/D_o = 1.25$ E = 28 x 10<sup>6</sup> (modulus of elasticity for 304 stainless steel)

From Table G in Sec II, Subpart 3 to determine Factor A

A = 1 x 10<sup>-3</sup> (Factor A, from Table G of Sec. II, Subpart 3)  $P_{ext} = \frac{2AE}{3(\frac{D_o}{t})}$ 

 $P_ext = 177.33$  psi (max allowable working external pressure for given *t*) = 1222 kPa

A wall thickness of t = 3.8 mm results in a maximum allowable working external pressure of P\_ext = 325 kPa, which is greater than the external pressure that the vacuum vessel will see.

**Example 3:** For comparison, calculate the maximum external pressure as in Example 2 but with a wall thickness of 2.5 mm.

 $D_{o} = 400 \text{ mm}$ t = 2.5  $D_{o}/t = 160$ L = 500 L/D\_{o} = 1.25 E = 28 x 10<sup>6</sup> (modulus of elasticity for 304 stainless steel)

From Table G in Sec II, Subpart 3 to determine Factor A

A =  $5.3 \times 10^{-4}$  (Factor A, from Table G of Sec. II, Subpart 3)

$$P\_ext = \frac{2AE}{3(\frac{D_o}{t})}$$

P\_ext = 61 psi (max allowable working external pressure for given t of 2.5 mm) = 420 kPa

Therefore a wall thickness of 2.5 mm has a P\_ext of 420 kPa.

**Example 4:** This is a worked example for a large vacuum vessel from ASME Boiler and Pressure Vessel Code (the Code) Section VIII, Division 1, paragraph UG-28. It is in imperial units.

Calculate the maximum allowed external working pressure following UG-28. The minimum required thickness takes into account the support of the stiffening rings. Note that the actual vessel shell thickness t = 0.375-inch

 $D_o = 38.0$  inch (965 mm) (outer diameter of shell) t = 0.261 inch (6.63 mm) (minimum required thickness of shell for external pressure of 14.5-psia)  $D_o/t = 145.594$  Since this ratio is greater than 10, follow UG-28(c)(1) L 235.7 inch (6057.5 mm) (length of stiffened shell)  $L/D_o = 6.203$ E =29x10<sup>6</sup> psi (modulus of elasticity for SA-516 Gr 60 carbon steel)

From Table G in Sec II, Subpart 3 to determine Factor A A =  $1.108 \times 10^{-4}$  (Factor A, from Table G of Sec. II, Subpart 3)

$$P\_ext = \frac{2AE}{3(\frac{D_o}{t})}$$

P\_ext = 14.709 psi (maximum allowable working external pressure for given t) = 101.35 kPa

A wall thickness of t = 0.261 inch results in a maximum allowable working external pressure of P\_ext = 14.7 psia, which is greater than the external pressure that the vacuum vessel will see. Since the actual wall thickness is 0.375 inch (9.5 mm), the vessel design is adequate for the working external pressure.



Factor A table for Stainless Steels

# **Appendix 2 – Low Outgassing Specialist Non Metal Materials**

#### • Celazole® PBI (PolyBenzImidazole) http://www.boedeker.com/celazo\_p.htm

Celazole® is the highest temperature-capable plastic available. However, it is very brittle (almost ceramic-like) and quite difficult to machine. That said, is it frequently used for bushings, bearings, rollers, and spacers in extreme environments. Its outgassing values are listed as 2.50% TML, 0.00% CVCM, 0.40% WVR.

#### • Vespel® (Polyimide)

http://www2.dupont.com/Vespel/en\_US/assets/downloads/vespel\_gen/E61500.pdf

DuPont Vespel® SP-1 is one of the most-used high-temperature plastic materials used in applications where high-purity and electrical properties are needed. Vespel is frequently used in ultra-clean semiconductor and chemical applications. It is also one of the most expensive materials sold, but is flight-approved for NASA, USAF and other aerospace agencies. Its NASA outgassing values are listed as 1.09% TML, 0.00% CVCM, 0.40% WVR.

#### • Duratron® XP (Polyimide)

http://www.portplastics.com/download/pdf/plastics/highPerformance/highPerformance26.pdf

Duratron® XP is the first real alternative to Vespel ... it was developed specifically to replace Vespel in extreme applications at a slightly lower price. It contains less than 1% metallic impurities as measured using the ICP-MS test standard. Duratron XP is ideal for use in high-energy gas plasma etch and strip processes. Outgassing values for Duratron XP are 0.75% TML, 0.00% CVCM, 0.49% WVR.

#### • Torlon® 4203 unfilled PAI (PolyAmide-Imide) http://www.boedeker.com/torlon-4203.htm

Unfilled Torlon® 4203 has high dielectric properties and low thermal expansion, and is much less expensive than some advanced polymers. Torlon 4203 is typically used for insulators, spacers, and mechanical parts up to 520°F. Its outgassing values are listed as 1.85% TML, 0.00% CVCM and 0.49% WVR.

#### • Torlon® 5530 glass-filled PAI (PolyAmide-Imide) http://www.boedeker.com/torlon-5530.htm

Torlon 5530 (30% glass-filled) is typically used for applications where dimensional stability over a wide temperature range is needed, as with temperature test sockets, nests, and fixtures. Its outgassing values are listed as 0.58% TML, 0.00% CVCM (% WVR is not shown). NOTE: Torlon's moisture absorption is a bit high, so critical dimensional stability can be an issue.

#### • Semitron® ESd 500HR (filled PTFE)

http://www.portplastics.com/download/pdf/plastics/staticControl/staticControl18.pdf

Semitron® ESd 500HR is antistatic/conductive PTFE. This material is relatively clean, readily machinable, dissipates static electricity reliably ... as a result it is used in test handling equipment, fixtures, and other applications where static generation may cause failures and/or errors in production environments. This material has low outgassing values of 0.04% TML, 0.00% CVCM and 0.01 % WVR. PTFE has good mechanical properties up to approximately 500°F.

#### • Neoflon® PCTFE (PolyChloroTetraFluoroEthylene)

http://www.aftonplastics.com/materials/pdfs/neoflan\_pctfe.pdf

PCTFE exhibits high chemical resistance, low and high temperature capability, resistance to most chemicals (including strong acids and bases), low friction, electrical and thermal insulation, and "slipperiness". PCTFE has the lowest outgassing values of any thermoplastic material we sell ... 0.01% TML, 0.00% CVCM, 0.00% WVR.

• **PEEK (PolyEtherEtherKetone)** http://www.dotmar.com.au/ketron-peek-1000/ketron-peek-1000-polyetheretherketone.html

PEEK is pure, easily machinable, chemically resistant, stable, and also has relatively low outgassing values (0.31% TML, 0.00% CVCM, 0.06% WVR). PEEK has good mechanical properties, but will not take temperatures over  $350^{\circ}$ F, so it may not have the mechanical or thermal performance needed.

• **Techtron® PPS (PolyPhenylene Sulfide)** http://www.dotmar.com.au/techtron-hpv-pps/techtron-hpv-pps.html

Techtron® PPS is easily machined to close tolerance, has excellent mechanical, thermal and chemical stability and has one of the lowest outgassing values of any thermoplastic material we offer (0.04% TML, 0.00% CVCM ... % WVR is not shown). Techtron PPS is generally a bit less expensive than PEEK or Torlon, but again, will not take as high temperatures.

• Ultem® PEI (PolyEtherImide) http://www.boedeker.com/ultem\_p.htm

Ultem<sup>®</sup> has good dielectric properties and low thermal expansion, and is considerably less expensive than some other polymers. PEI is also clean and stable, but is not particularly resistant to chemicals or solvents ... it has outgassing values of 0.40% TML, 0.00% CVCM and 0.06 % WVR. PEI has good mechanical properties up to approximately 410°F.

• Semitron® ESd 410C (filled PEI) http://www.boedeker.com/sem410\_p.htm

Semitron® ESd 410C is antistatic/conductive PEI. This material is relatively clean, readily machinable, dissipates static electricity reliably ... as a result it is used in test handling equipment, fixtures, and other applications where static generation may cause failures and/or errors in production environments. This material has outgassing values of 0.46% TML, 0.00% CVCM and 0.17 % WVR. PEI has good mechanical properties up to approximately 340°F.

#### • Ertalyte® PET-P (Polyethylene Terephthalate) http://www.boedeker.com/ertaly\_p.htm

Ertalyte® offers the dimensional stability of acetal with the wear resistance of nylon. Ertalyte® PET-Polyester is clean, chemically resistant, stable, and also has relatively low outgassing values (0.13% TML, 0.00% CVCM ... % WVR is not shown). PET-P is considerably less expensive than most of the other materials listed above, but may not have the mechanical or thermal performance needed for all applications.

#### • Semitron® ESd 225 (filled acetal) http://www.boedeker.com/sem225\_p.htm

Semitron® ESd 225 is antistatic/conductive acetal. This material is relatively clean, readily machinable, dissipates static electricity reliably ... as a result it is used in test handling equipment, fixtures, and other applications where static generation may cause failures and/or errors in production environments. This material has low outgassing values of 1.00% TML, 0.05% CVCM and 0.60 % WVR. Acetal has good mechanical properties up to approximately 180°F.

# Appendix 3 – Materials for use in Vacuum

The choice of materials is limited for vacuum systems to a range that has little impact on vacuum production and ultimate base vacuums.

#### 1.1 Metals

- <u>Stainless Steel</u> is used for the majority of vacuum system designs due to its:
  - o Durability
  - Surface finishing
  - Ability to withstand moderate temperatures for system bake out
  - Low magnetic susceptibility
  - Low thermal conductivity
  - Resistance to oxidisation
  - Low vapour pressure

Type 304 or 316 stainless steel is ideally suited for vacuum vessel construction because of its machining/welding characteristics, excellent corrosion resistance and overall cost effectiveness.

Not all stainless alloys are acceptable. Free-machining 303 steel contains sulphur, which tends to outgas. Alloys with good weldability using TIG or MIG welding are usually chosen.

- 304 or 316 stainless steel is a common choice of a stainless steel.
- $\circ~$  304L stainless steel, a low-carbon variant of 304 steel, is used for ultra-high vacuum systems.
- 347 stainless steel does not accept high polish.
- $\circ$  321 stainless steel is chosen when low magnetic permeability is needed.
- <u>Mild steel</u> is okay for low to moderate vacuums above 10<sup>-4</sup> Pa. Outgassing can be reduced with suitable plating such as nickel. It has high permeability to hydrogen and tendency to rust. Mild steel must not be used for any components or chambers used near the bending magnets
- <u>Aluminium alloys</u> are easily machined and have a low vapour pressure, unless the alloys contain high proportion of zinc. 6061 grade is good for general use. Components used in high vacuums or better must not be anodized, as the oxide layer can trap water vapour and outgas. Aluminium and its alloys have low strength at high temperatures, distort when being welded, and the copper-containing ones are poorly weldable. Aluminium wire rings can be used as cheap gaskets in demountable seals. Soft alloys must be used to ensure wire gaskets do not damage the seal faces. Aluminium has high thermal conductivity, good corrosion resistance, and low solubility of hydrogen. Loss of strength at high temperatures limits its use in bakeable applications, but aluminium is advantageous for large-size systems due to its lower weight and lower cost than stainless steel. Aluminium is not recommended for vacuum chambers.
- <u>Brass</u> is not acceptable for vacuum chambers however it may be suitable for some small beam line inserts for specific applications. Once used widely but copper can cause problems with neutron production if struck by proton beams of particular energies. Brass is for high thermal conduction applications in cooling baffles or sample mounts that may be heated. Although bare

brass has good corrosion resistance the zinc content may cause outgassing problems. This can be reduced by plating with nickel.

- <u>Nickel</u> is widely used in vacuum technology, e.g. as mechanical parts in vacuum tubes. It is relatively low-cost, can be spot welded, can be easily machined, has high melting point and is resistant to many corrosive fluids and atmospheres. Its potential drawback is its ferromagnetism, which restricts applications that would be influenced by magnetic fields.
- <u>Beryllium</u> is used primarily for x-ray windows.
- <u>Oxygen-free copper</u> is okay for high vacuum but it is difficult to outgas completely. Copper is insensitive to hydrogen and impermeable to hydrogen and helium, has low sensitivity to water vapour, but is attacked by mercury. Although, oxygen-free copper is widely used as it is easily machined and has good corrosion resistance. It is unsuitable for bakeable vacuum envelopes due to its tendency to oxidize and create scales. Conflat flange seals are made from copper. Its strength falls sharply above 200 °C. Its vapour pressure becomes significant at above 500 °C.
- <u>Indium</u> wire is used as a gasket in demountable seals. Not suitable for high temperature above  $\sim 100^{\circ}$ C applications.
- <u>Gold</u> wire is used as a gasket in demountable seals for ultra-high vacuum.
- <u>Tantalum</u> is acceptable in vacuum systems but it is difficult to work with due to its hardness. It is expensive so it is usually purchased in sheet form. Typically mounted as a shield where ion beams contact a surface as it has a low production of secondary electrons. Good for apertures and defining slit faces and anywhere that a metal edge is used to intersect the ion beam.
- <u>Zirconium</u> is corrosion-resistant. Like tantalum, it has low production of secondary electrons, so it is used as a shield of areas where reducing their production is important. It is used for neutron windows. It is expensive and rarekly used. Zirconium and zirconium hydride are used for gettering.

#### **1.2 Plastics**

Plastics or ceramics are not to be used as the primary vacuum chamber structure without engineering advice from ANSTO or a specialist scientific vacuum instrument company. Plastic is very good for electrical insulators, bushes or light weight components are necessary in vacuums.

- <u>Polytetrafluoroethylene</u>, PTFE or Teflon as it is commonly known is suitable for use inside of vacuum systems from low to ultra high vacuums. Being soft PTFE can flow (creep) so for applications such as sample positioning systems or other systems requiring high stability then other plastics may need to be considered. PTFE has be far the highest dielectric strength, for extruded PTFE, 19.7 kV/mm 60 173 kV/mm for an insulating film. This compares with air which is around 1 kV/mm. It is self-lubricating, tolerant to fairly high temperatures, and has low outgassing. It is not suitable for barrier between vacuum and atmosphere, due to its permeability.
- <u>Ertalyte</u> offers the dimensional stability of acetal with the wear resistance of nylon. It is stable with relatively low outgassing values and has very low water absorption. It is very good for small mechanical loads requiring electrical isolation in vacuum systems.

- <u>Polyvinyl Chloride (PVC)</u> is acceptable in vacuum systems but not where heat is above 50°C. It is usually an amorphous thermoplastic material with excellent chemical resistance and dielectric properties, good tensile, flexural and mechanical strength, low moisture absorption, exceptional dimensional stability and good flammability characteristics.
- Other plastics can be used in vacuum systems but must have low vapour pressure. Care must be taken to ensure they are not used in applications that may require temperatures that will cause high outgassing rates.
- <u>Perspex</u> (acrylic plastic) is another plastic which has good dielectric properties but a high vapour pressure especially at raised temperatures making it unsuitable in vacuums however it can be used in small amounts in a well pumped high vacuum system.
- <u>Vespel</u>, a polyimide, is very expensive, but machines well, has good electrical insulator properties and is compatible with ultra-high vacuum. It does however absorb moisture and requires a longer pump down time. It also performs well in extremely low cryogenic temperatures. Good for bushes or where a mechanically stable plastic is required.
- <u>Nylon</u> is self-lubricating but has high outgassing rate and relatively high water absorption.
- <u>Polycarbonates and polystyrene</u> are good electrical insulators with moderate outgassing.
- <u>Mylar</u> is used to make thin windows that allow ion beams to penetrate from vacuum to positive pressures with little energy loss. For example gas detectors mounted onto beam lines for AMS have gas inside of the detector separated from the beam line which is under vacuum. Ion beams pass through the window as they move from the vacuum side to the positive pressure side. Other materials in this category include Kapton which is rated for higher temperatures.

#### **1.3 Elastomers**

Some elastomers have sufficient vacuum properties and are used widely as vacuum seals in the form of 'o' rings.

- <u>Viton</u> is the standard seal used throughout the accelerator area as it is long lasting, low vapour pressure as compared with other elastomers and is bakeable to 200 °C.
- <u>Nitrile</u> rubber is used for vacuum seals. Does tend to break down over time. First signs of breakdown are small splits. If used for vacuum seals then they must be inspected regularly.
- <u>Natural</u> rubber is not typically used in vacuum systems other than inside of vacuum pumps usually on the low vacuum side of the system.
- <u>Silicone</u> rubber is not generally used as a seal in high vacuum environments. Silicone is soft when compared with other elastomers. It has a relatively low vapour pressure.

Further reading: Materials for high vacuum technology: an overview, S. Sgobba, CERN, Geneva, Switzerland. http://cdsweb.cern.ch/record/983744/files/p117.pdf

#### 1.4 Ceramics and glass

• <u>Alumina</u> ceramic based ceramics in vacuum systems perform well provided the ceramic is not porous which can trap gas bubbles. Ceramics are readily available bonded onto metal flanges or mounting plates. Ceramic can also be baked if necessary to speed up the outgassing of surfaces.

Glass is similar to ceramics in this application. It is very low vapour pressure but difficult to work with. Glass viewports can be purchased already mounted in a metal flange.

- <u>Borosilicate glass</u> is often used for smaller assemblies and for viewports.
- <u>Porcelain</u> ceramics, when fully vitrified and therefore non-porous, are excellent insulators usable to 1500 °C. it is generally commercially available bonded to metal in electrical insulators and other feedthroughs.
- <u>Mica</u>, although it is neither ceramic or glass it best fits in this category. Mica has been used in vacuum systems for both electrical and thermal insulation but because Mica is a series of laminations it contains trapped air which is not suitable in high to ultra-high vacuums.
- <u>Macor</u> is a machinable glass made by Corning Incorporated with similar properties to ceramics. It has a low vapour pressure and thermally stable up to 1000°C making it bakeable. It is made from mica and borosilicate glass. It is by far the most flexible of the glass-ceramic options.

 Leakage Testing Handbook, Prepared for Liquid Propulsion Section, Jet Propulsion Laboratory, National Aeronautics and Space Administration, Pasadena, California
 Nondestructive Testing Handbook, Volume One, Leaktesting, American Society for Nondestructive Testing.
 Leakage Testing Handbook, Revised Edition, July 1969, General Electric.
 Fluid Flow in Small Passages, Mars Hablanian, J.W.Marr, Varian

#### 1.5 Greases and oils

There are few acceptable greases and oils for use in vacuum systems. Users must ensure they purchase greases and oils specifically designed for use in vacuums. Typically they will have low vapour pressures, extremely low water absorption and easy to remove for cleaning.

- <u>Apiezon family</u> For further information http://www.apiezon.com/ . These have been used throughout the history of accelerators at ANSTO with excellent results. Wherever greases are used remember to use the minimalist amount.
- <u>Greases</u>
  - Apiezon H High Temperature Vacuum Grease, silicone and halogen free
  - Apiezon L Ultra high vacuum grease, silicone and halogen free
  - Apiezon M High vacuum grease, silicone and halogen free
  - Apiezon N Cryogenic high vacuum grease, silicone and halogen free
  - Apiezon T Medium temperature grease, silicone and halogen free
  - o Apiezon AP100 Ultra High Vacuum Lubricating Grease, silicone free
  - o Apiezon AP101 Anti seize Vacuum Grease, silicone free
- <u>Waxes</u>
  - Apiezon® Waxes and Compounds
  - Apiezon Wax W (Hard Vacuum Sealing)
  - Apiezon Wax W40 (Softer Vacuum Sealing)
  - Apiezon Wax W100 (Softest Vacuum Sealing)

- Apiezon Sealing Compound Q
- <u>Vacuum Oil</u> is used in few applications around the accelerator area. With a change to oil free pumps most oil consuming pumps are redundant. However these types of pumps may still in use in other applications. Oil vane roughing pumps and diffusion pumps were the largest consumers of oil. More recent brands of vane pumps were specifically tuned to oils of particular specifications so generic oils may not be compatible. Diff pumps are more forgiving and high quality generic synthetic oils may be used. The list below is a small sample of what is available and previously used.
  - $\circ\,$  Shell Vitrea 68 oil for roughing pumps. Check specification on pump to ensure compatibility with this mineral oil
  - Fomblin PFPE (Perfluorpolyether) oils designed for rotary pumps. Check specification on pump to ensure compatibility with this mineral oil
  - Apiezon® Diffusion Pump Fluids Apiezon AP 201 The Apiezon diffusion pump fluid is known for its exceptional higher degree of oxidation resistance.

# **Appendix 4 – Surface finishing**

In most cases a clean polished surface will yield the least outgassing as there are minimal surface traps for gas. Also, a polished surface is easier to clean and keep clean. Achieving a polished surface may not be practicable due to accessibility into tubes, chambers and small components but a combination of other methods may provide an adequate surface finish that will allow the desired vacuum level to be reached. The best method for a given surface will depend on the material, geometry and accessibility to its surfaces.

- <u>Polishing</u> A polished surface may be either smooth or uneven but the metal will lustre in either case. Polishing is a method of removing a layer of metal to expose clean metal underneath. This can be done with a mechanical buff or by hand with the aid of a polishing compound. Care must be taken to ensure all traces of the polishing compound are removed during final cleaning. All residues must be removed.
- <u>Grinding, sanding and other coarse to fine abrasive methods</u> These methods tear the surface dragging metal over metal. Some metal breaks away exposing clean metal underneath. The problem with this type of surface preparation is that the metal can fold over and not break away leading to voids of trapped gases. It is important that a fine grit is the last grit used to ensure larger metal drifts on the surface are torn away.
- <u>Grit blasting with grit or beads</u> This method propels grit or beads onto the metal surface causing fragments of metal to be removed with the impact. Like the abrasive techniques metal can be layered on metal (peening) if the grit is too coarse. Best to finish with a fine grit then a mechanical polish. Grit blasting can provide a more uniform surface finish and is very good for removing scale that may be present from rolling or extrusions.
- <u>Pickling</u> by far a very good way to clean welds and other heat induced decolourisations in the metal. It will remove a thin layer of metal, scale, oils, etc. Care must be taken to ensure any chemicals used are neutralised and thoroughly cleaned away. Residues cannot be tolerated. Makes general maintenance of the surface easier.
- <u>Passivating</u> a form of chemical polishing and surface protection. Can be stimulated with an electric current. Passivating is the spontaneous formation of a hard non-reactive surface film that inhibits further corrosion. It isn't a preferred method of surface preparation in vacuum systems as damaged surfaces can form micro cracks trapping water and other gases. For example, damage to anodised aluminium may allow aluminium oxide to form which can readily trap water. Passivation is not typically used for stainless steel as the surfaces are self healing, provided sufficient oxygen is available.

Further reading on surface passivation can be found at: http://www.euro-inox.org/pdf/map/Passivating\_Pickling\_EN.pdfGetter surfaces

• Nickel Plating – has been used on vacuum components to provide stability to the surface of normally oxidising metals such as steel, copper and brass. It is important to ensure that the nickel has uniform bonding to the parent metal. Cases have been observed where the plating has lifted creating voids in the vacuum space. Although nickel is resistant to corrosion it will form a thin layer if exposed to moist atmospheres. A periodic clean is recommended to minimise problems with corrosion.

Element	Melting Point °C	Critical Temp °C	Phase at Critical T
Aluminium	660	1124	Liquid
Americium	1176	896	Solid
Antimony	631	455	Solid
Arsenic	84	270	Liquid
Barium	729	574	Solid
Beryllium	1289	1102	Solid
Cadmium	321	226	Solid
Caesium	29	114	Liquid
Calcium	847	538	Solid
Californium	900	1402	Liquid
Cerium	798	1602	Liquid
Chromium	1863	1220	Solid
Cobalt	1492	1418	Solid
Copper	1084	1146	Liquid
Dysprosium	1412	1025	Solid
Erbium	1529	1139	Solid
Europium	822	547	Solid
Gadolinium	1312	1450	Liquid
Gallium	30	954	Liquid
Germanium	938	1260	Liquid
Gold	1064	1291	Liquid
Hafnium	2231	2255	Liquid
Holmium	1474	1146	Solid
indium	157	836	Liquid
Iridium	2447	1199	Solid
Iron	1538	1253	Solid
Lanthanum	918	1586	Liquid
Lead	328	622	Liquid
Lithium	181	465	Liquid
Lutetium	1663	1517	Solid
Magnesium	651	386	Solid
Manganese	1246	887	Solid
Mercury	-39	22	Liquid
Molybdenum	2623	2319	Solid
Neodymium	1021	1219	Liquid
Neptunium	639	1781	Liquid
Nickel	1455	1409	Solid
Niobium	2468	2501	Liquid
Osmium	3033	2721	Solid
Palladium	1555	1348	Solid
Platinum	1769	1800	Liquid
Plutonium	640	1506	Liquid
Potassium	64	165	Liquid
Praseodymium	931	1381	Liquid
Protactinium	1572	2636	Liquid
Rhenium	3186	2817	Solid
Rhodium	1963	1874	Solid
Rubidium	39	129	Liquid

# **Appendix 5 – Critical Vapour Pressures**

Substance	Vapour Pressure (Pa)	Temperature (°C)
Acetaldehyde	98.7	20
Acetone	24640	20
Butane	22	20
Carbonyl sulphide	1255	25
Ethanol	5.83	20
Ethylene glycol	0.5	20
Formaldehyde	435.7	20
Freon 113	37.9	20
Methanol	12800	20
Methyl isobutyl ketone	26.48	25
Nitrogen (N <sub>2</sub> )	63200	20
Oxygen (O <sub>2</sub> )	54200	20
Propane	2200	55
Propanol	2.4	20
Tungsten	0.1	3203
Water (H <sub>2</sub> O)	2.3	20
Xenon difluoride	0.6	25

# **Appendix 5 – Vapour Pressures continued**

Substance	Vapour Pressure (Pa)	Melting point (°C)	
Apiezon Oil J	0.13 at 200°C 1.3 x 10 <sup>-4</sup> at 20°C	-	
Apiezon Oil K	0.13 at 300°C 1.3 x 10 <sup>-7</sup> to 10 <sup>-8</sup> at 20°C	-	
Apiezon Grease L	0.13 at 100°C 1.3 x 10 <sup>-7</sup> to 10 <sup>-9</sup> at 20°C	47	
Apiezon Grease M	0.13 at 200°C 1.3 x 10 <sup>-5</sup> to 10 <sup>-6</sup> at 20°C	44	
Apiezon Grease N	0.13 at 200°C 1.3 x 10 <sup>-6</sup> to 10 <sup>-7</sup> at 20°C	43	
Apiezon Grease T	About 1.3 x 10 <sup>-6</sup> at 20°C	125	
High Vacuum Grease Dow Corning	< 1 x 10 <sup>-4</sup> at 20°C	-	
Nylon	~ 1 x 10 <sup>-3</sup> at 20°C	-	
Teflon	< 1 x 10 <sup>-4</sup> at 20°C	-	
Viton	1 x 10 <sup>-6</sup> at 20°C	-	

# Appendix 6 – Outgassing tables for various materials Reference site: http://home.fnal.gov/~mlwong/outgas\_rev.htm

					i sampies.	
Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	(hours)	Test method	Reference	Year
Aluminium	None	1x10 <sup>-6</sup>	1h		Schamus (ref Markley, et al)	1999
Aluminium	Degassed	$1.7 \times 10^{-7}$	1h		Schmaus (ref Markley, et al)	1999
Aluminium	Degassed	2.7x10 <sup>-8</sup>	10h		Schmaus (ref Markley, et al)	1999
Aluminium 6061-T6	Baked 13.5h @ 300°C	1.4x10 <sup>-8</sup>	10h		Schmaus (ref Das)	1999
Aluminium	Cleaned	8x10 <sup>-9</sup>	10h		Schmaus (ref Blears, et al)	1999
Aluminium	Fresh, degreased w/ trichloroethylene & cleaned w/ ethyl alcohol	6.3x10 <sup>-9</sup>	1h	conductance	Elsey (ref Schram)	1975 (1963)
Aluminium	Degassed	$4.6 \times 10^{-9}$	100h		Schmaus (ref Markley, et al)	1999
Aluminium 6061-T6	Baked @ 200°C	4.5x10 <sup>-9</sup>	10h		Schmaus (ref Das)	1999
Aluminium	Degassed 24h, degreased w/ trichloroethylene & cleaned w/ ethyl alcohol	4.14x10 <sup>-9</sup>	1h	conductance	Elsey (ref Schram)	1975 (1963)
Aluminium 6061-T6	None	2.5x10 <sup>-9</sup>	10h		Schmaus (ref Das)	1999
Aluminium 5083-O	Bell jar, as received, room temp.	2.18x10 <sup>-9</sup>	10.3h	Rate-of-rise	Schrank, et al	1964
Aluminium 5083-O	Bell jar, as received, room temp.	1.27x10 <sup>-9</sup>	8h	Conductance	Schrank, et al	1964
Aluminium 5083-O	Bell jar, as received, room temp.	6.6x10 <sup>-10</sup>	24h	Conductance	Schrank, et al	1964
Aluminium 5083-O	Bell jar, as received, room temp. after baking @ 220°C	$4.6 \times 10^{-10}$	50h (baked @27-31h)	Conductance	Schrank, et al	1964
Aluminium 6061-T6	Baked 15h @ 300°C	$1.6 \times 10^{-10}$	10h		Schmaus (ref Das)	1999
Aluminium	Degassed 24h, degreased w/ trichloroethylene & cleaned w/ ethyl alcohol	3.06x10 <sup>-10</sup>	1h	conductance	Elsey (ref Schram)	1975 (1963)
Aluminium 5083-O	Bell jar, no. 4 finish (lapped), room temp.	$2.87 \times 10^{-10}$	6.0h	Rate-of-rise	Schrank, et al	1964
Aluminium	Fresh, degreased w/ trichloroethylene & cleaned w/ ethyl alcohol	6.0x10 <sup>-10</sup>	10h	conductance	Elsey (ref Schram)	1975 (1963)
Aluminium, type 1100	Cleaned w/ detergent, rinsed w/ acetone, pumped 24 hours	~10 <sup>-10</sup>	0	conductance	Young	1968
Aluminium	LEP vacuum chamber, chem clean, baked in situ @ 150°C	$2.3 \times 10^{-11}$	24h		Mathewson, et al	1988
Aluminium 6061-T4	Degreased in acetone w/ methanol rinse; baked 100°C	6x10 <sup>-12</sup>	24h	Rate-of-rise & conductance	Halama, Herrera	1976

Outgassing rates of aluminium Note the different methods of measurement and treatment of samples.
Aluminium 6061-T4	Degreased in acetone w/ methanol rinse, chem polished, baked 100°C	3x10 <sup>-12</sup>	24h	Rate-of-rise & conductance	Halama, Herrera	1976
Aluminium 6061-T4	Degreased in acetone w/ methanol rinse, chem polished, glow discharge in Ar, baked 100°C	5x10 <sup>-13</sup>	24h	Rate-of-rise & conductance	Halama, Herrera	1976
Aluminium, type 1100	Above plus baked 15h @ 250°C under vacuum	$4x10^{-13}$	24h @ room temp	conductance	Young	1968
Aluminium	PETRA vacuum chamber, glow discharge@145°C	$1 \times 10^{-13}$	Up to 200h	In situ glow discharge, conductance	Mathewson, et al	1977

# Outgassing rates of stainless steels, along with the surface treatment and pumping time.

Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	Time (hours)	Test method	Reference	Year
Stainless steel	None	6.4x10 <sup>-7</sup>	1h		Schamus (ref Markley, et al)	1999
Stainless steel	degreased	4x10 <sup>-7</sup>	1h		Schamus (ref Markley, et al)	1999
Stainless steel	None	2x10 <sup>-7</sup>	1h		Schamus (ref Blears, et al)	1999
Stainless steel	None	2x10 <sup>-8</sup>	10h		Schamus (ref Blears, et al)	1999
Stainless steel NS22S	Fresh	1.4x10 <sup>-8</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel ICN 472	fresh	1.3x10 <sup>-8</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel	None	1.2x10 <sup>-8</sup>	10h		Schamus (ref Das, et al)	1999
Stainless steel ICN 472	sanded	8.3x10 <sup>-9</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel NS22S	electro polished	4.3x10 <sup>-9</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel NS22S	mechanically polished	1.7x10 <sup>-9</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel	fresh	1.5x10 <sup>-9</sup>	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel ICN 472	Polished & vapor degreased	1.4x10 <sup>-9</sup>	10h		Schamus (ref Dayton, et al)	1999
Stainless steel	None	1.4x10 <sup>-9</sup>	10h		Schamus (ref Das, et al)	1999
Stainless steel NS22S	fresh	1.3x10 <sup>-9</sup>	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel ICN 472	sanded	1.0x10 <sup>-9</sup>	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel	None	7.6x10 <sup>-10</sup>	1h		Schamus (ref Das, et al)	1999
Stainless steel NS22S	mechanically polished	$4.6 \times 10^{-10}$	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel NS22S	electro polished	$4.3 \times 10^{-10}$	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel	Baked 24h @ 200°C	$1.5 \times 10^{-10}$	1h		Schamus (ref Das, et al)	1999

Stainless steel	None	$1.1 \mathrm{x} 10^{-10}$	100h		Schamus (ref Das, et al)	1999
Stainless steel 304	Degrease + water rinse	$4.0 \times 10^{-11}$	40h	Conductance	Strausser	1973
Stainless steel U15C	Baked 25h @ 300°C	$4.5 \times 10^{-12}$			Elsey (ref Calder, et al)	1975
Stainless steel 304	Degrease + water rinse, baked in vacuum 150°C for 12h	4.0x10 <sup>-12</sup>	5h after bakeout	Conductance	Strausser	1967
Stainless steel 304	Electropolished, baked 30h @ 250°C	$3.0 \times 10^{-12}$			Elsey (ref Young)	1975
Stainless steel U15C	Baked 45h @ 360°C	$2.6 \times 10^{-12}$			Elsey (ref Calder, et al)	1975
Stainless steel	Baked 24h @ 200°C	$9.3 \times 10^{-13}$	100h		Schamus (ref Das, et al)	1999
Stainless steel U15C	Baked 3h in vacuum @ 1000°C + 25h in situ @ 360°C	1.6x10 <sup>-14</sup>			Elsey (ref Calder, et al)	1975

Other cleaning methods although outgassing rates are not measured:

Material	Treatment	Results	Reference	Year
Aluminium	Quench a hot aluminium extrusion in an Ar- $O_2$ atmosphere	Dense, thin (~20Å thick) oxide layer	Sasaki, Y.T.	1990
Aluminium 6063	Clean with an alkaline detergent (Almeco 18)	Removes MgO, C, and Al <sub>2</sub> O <sub>3</sub>	Sasaki, Y.T.	1990
Aluminium	Vapour degreasing, pump for 120h before and after bake	Dominant residual gases: H <sub>2</sub> O, H <sub>2</sub> , CO, CO <sub>2</sub> , & CH <sub>4</sub> ; after baking @ 150°C and pumping, H <sub>2</sub> O & CH <sub>4</sub> gone, outgassing rates of other gases reduced 1 order of magnitude except H <sub>2</sub>	Mathewson, A.G., et al	1989
Aluminium	Vapour degreasing, then light alkaline etch (CERN basic procedure), pump for 120h before and after bake	Dominant residual gases: H <sub>2</sub> O, H <sub>2</sub> , CO, & CO <sub>2</sub> ; after baking @ 150°C and pumping, only H <sub>2</sub> exists	Mathewson A.G., et al	1989
Aluminium	Strong alkaline etching in NaOH, pump for 120h before and after bake	Dominant residual gases: H <sub>2</sub> O, H <sub>2</sub> , CO, CO <sub>2</sub> , & CH <sub>4</sub> ; after baking @ 150°C and pumping, only H <sub>2</sub> exists	Mathewson A.G., et al	1989
Aluminium	Vapour degreasing, Ar glow discharge @ 150°C	On initiation of discharge, large pressure increase (normal in Ar glow discharge) was too large for accurate measurement	Mathewson A.G., et al	1989
Aluminium	Vapour degreasing, then light alkaline etch (CERN basic procedure), Ar glow discharge @ 150°C	H <sub>2</sub> , CO dominant gases desorbing from surface during discharge cleaning	Mathewson A.G., et al	1989
Aluminium	Strong alkaline etching in NaOH, Ar glow discharge @ 150°C	H <sub>2</sub> , CO dominant gases desorbing from surface during discharge cleaning at order of magnitude less than light alkaline etch	Mathewson A.G., et al	1989
SS	H <sub>2</sub> glow discharge - 22°C	Dominant residual gases: $CH_4$ , $CO$ , & $C_2H_4$ ; water production insignificant; frequent arcing	Dylla, H.F.	1988

		on all exposed surface noted during initial discharge period		
SS	H <sub>2</sub> glow discharge – 150°C	Dominant residual gases: $CH_4$ , $CO$ , & $C_2H_4$ , and $H_2O$ ; frequent arcing on all exposed surface noted during initial discharge period	Dylla, H.F.	1988
SS	Ar glow discharge – ambient temp	Dominant residual gases that were significantly removed: $CO_2$ , $H_2$	Dylla, H.F.	1988
SS	Ar/O <sub>2</sub> glow discharge	Improved removal of CO and CO2; minor amts of implanted Ar that can be removed by baking @ 350°C	Dylla, H.F.	1988
	Ar/O <sub>2</sub> glow discharge 2h, exposed to air, then Ar/O <sub>2</sub> glow discharge 2h	Some degree of passivation: residual gases were removed with 1 <sup>st</sup> cleaning & did not show up during the 2 <sup>nd</sup> cleaning	Dylla, H.F.	1988
	O <sub>2</sub> glow discharge	Rapid removal of carbon, not of hydrocarbons (which can be baked out initially), increased surf. Oxidation, sputtering of the base metal, and possible enhanced outgassing of O <sub>2</sub> - containing molec.	Dylla, H.F.	1988

# Different outgassing measurements of epoxies

Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	Time (hours)	Test method	Reference	Year
NMA w/ solid alumina filler	Cured 300°F	$1.6 \times 10^{-7}$			Hanson, Patel	1970
Polyimide polymer	Thermosetting; measured @ 40°C	~1.1x10 <sup>-7</sup>	4h+9min	conductance	Kendall, Zabielski	1965
Polyimide polymer	Thermosetting; measured @ 155°C	~5x10 <sup>-8</sup>	4.5h increase temp + 6h	conductance	Kendall, Zabielski	1965
Silicone resin	Needs dicumyl peroxide catalyst; thermosetting, measured @ 35°C	~5x10 <sup>-8</sup>	4h	conductance	Kendall, Zabielski	1965
Araldite epoxies	Cured ~150°C for 15h	~10 <sup>-8</sup>	51h pumping	conductance	Barton, Govier	1965

# Kapton is a newer material and little data exists about its outgassing rate. Below are 3 references

Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	Time (hours)	Test method	Reference	Year
Kapton cable		1.0x10 <sup>-5</sup>	0h	conductance	Postma	1999
Kapton foil		$1.0 \times 10^{-7}$	40h		Ferro-Luzzi	1999
Polyimide	Bake @ 300°C	$4.0 \mathrm{x} 10^{-8}$	12h		Weston	1970
Kapton cable		$2.4 \times 10^{-8}$	168h	conductance	Postma	1999
Kapton cable		2.7x10 <sup>-9</sup>	336h	conductance	Postma	1999
Kapton cable		$6.5 \times 10^{-10}$	504h	conductance	Postma	1999

# Outgassing rates of several types of plastics

Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	Time (hours)	Test method	Reference	Year
Nylon		$1.2 \times 10^{-5}$	1h		Elsey (ref	1975
					Power, et al)	
Nylon		6.0x10 <sup>-6</sup>	1h		Elsey (ref	1975

					Power, et al)	
PVC	24h @ 95% relative	8.5x10 <sup>-7</sup>	1h	conductance	Elsey (ref	1975
	humidity				Santler)	
PTFE	Fresh	$1.7 \mathrm{x} 10^{-7}$	1h	conductance	Elsey (ref	1975
					Santler)	
Teflon		6.5x10 <sup>-8</sup>	1h	conductance	Elsey (ref	1975
					Santler)	
PTFE	Fresh	3.3x10 <sup>-8</sup>	10h	conductance	Elsey (ref	1975
					Santler)	
Teflon		$2.5 \times 10^{-8}$	10h	conductance	Elsey (ref	1975
					Santler)	
PVC	24h @ 95% relative	$2.0 \times 10^{-8}$	10h	conductance	Elsey (ref	1975
	humidity				Santler)	
G-10		~10 <sup>-8</sup>			Beams	2001
					Division -	
					FNAL	

Reference to the following has been lost but it is believed to have originated in an early Vacuum Catalogue from the 1990's

 $K_1$  is the outgassing rate (air equivalent) after 1 hour pumping  $\alpha_1$  is the slope of the (log K - log t) curve at 1 hour

Material	K <sub>1</sub> Torr litre/sec cm <sup>2</sup> (x 10 <sup>-10</sup> )	α1	K <sub>10</sub> Torr litre/sec cm <sup>2</sup> (x 10 <sup>-10</sup> )	a <sub>10</sub>
Aluminium Alloy (fresh)	63	1	6.0	1
Aluminium Alloy (degassed 24 hours)	41.4	3.2	3.06	0.9
Aluminium Alloy (3 hours in air)	65.5	1.9	4.75	0.9
Aluminium Alloy (anodised -2µ pores)	2760	0.9	322	0.9
Aluminium Alloy (bright rolled)	-	-	75	1
Duralumin	1700	0.75	350	0.75
Brass (wave guide)	4000	2.0	100	1.2
Copper (fresh)	400	1	41.5	1
Copper (mechanical polish)	35	1	3.56	1
OFHC Copper (fresh)	118	1.3	12.6	1.3
OFHC Copper (mechanical polish)	19	1.1	1.63	1.1
Gold (fresh wire)	1580	2.1	5.1	1
Mild steel	5400	1	500	1
Mild steel (slightly rusty)	6000	3.1	130	1
Cr plated steel (fresh)	70.5	1	6.8	1
Cr plated steel (polished)	91	1	8.0	1
Ni plated steel (fresh)	42.4	0.9	4.84	0.9
Ni plated steel	27.6	1.1	2.33	1.1
Chemically Ni plated steel (fresh)	83	1	7.05	1
Chemically Ni plated steel (polished)	52.2	1	4.6	1
• • • • •				

Araldite (moulded)	116	0.8	35.2	0.8
Araldite D	800	0.8	220	0.78
Araldite D	190	0.3	125	0.5
Araldite F	150	0.5	73	0.5
Celluloid	860	0.5	430	0.5
(PTFE) (fresh)	16.6	0.8	3.31	0.9
Kel-F Oak ridge	4	0.57	1.7	0.53
Methyl methacrylate	420	0.9	140	0.57
Mylar V-200 (24 hr at 95% RH)	230	0.75	40	-
Nylon	1200	0.5	600	0.5
Pertinax	620	0.18	290	0.5
Perspex	72	0.44	27	0.44
Perspex	310	0.4	180	0.4
Polyamid	460	0.5	230	0.5
Polyester - glass laminate	250	0.84	80	0.81
Polyethylene	23	0.5	11.5	0.5
Polystyrene	2000	1.6	200	1.6
Polystyrol	56	0.6	12	0.61
Polyvinylcarbazol	160	0.5	80	0.5
PTFE	30	0.45	15	0.56
P.V.C. (24 hr at 95% RH)	85	1.00	2	-
Teflon	6.5	0.6	2.5	0.2
Terephenil (fresh)	62.2	0.5	16.8	0.5
Neoprene	3000	0.4	-	-
Viton	114	0.8	-	-
	1	1		

# **Appendix 7 – Local Plant and Safety Regulations**

Regular maintenance inspections are carried out on ANTARES and STAR accelerator vacuum equipment to ensure operational status, vacuum quality, vacuum pump performance, and the mechanical and electrical integrity of the systems.

Generally, the integrity of the vacuum systems can be measured as a direct function of the known static vacuum pressures in a given vacuum system. That is, a lowering of vacuum from the known average vacuum pressure in a given system will indicate either/and, a leak in a seal, a failing vacuum pumping system or a structural failure.

The following is an overview of the application of the ANSTO OHSE Plant and Safety Standard, AS 2309, to vacuum systems in the accelerator facilities. *Reference: http://docushare.ansto.gov.au/Get/File-32340/Plant\_Safety\_Standard.pdf* 

Risk	Hazard	Risk rating	Controls	Risk rating (with controls in place)
Catastrophic failure vacuum vessels	Implosion – scattering of material fragments and loose fittings becoming missiles	Very low	<ul> <li>Use of vacuum standards to design equipment for safe usage.</li> <li>Protective barriers around equipment and fittings that may be susceptible to knocking</li> </ul>	Very low
Cotostrophia failura	• Implosion – scattering of material fragments and loose fittings becoming missiles	Very low	<ul> <li>Use of vacuum standards to design equipment for safe usage.</li> <li>Purchased equipment from reputable companies/designers only</li> </ul>	Very low
Catastrophic failure vacuum beam lines and associated equipment	• Electric shock from fault in high vacuum gauge	Very low	• Vacuum gauges using high voltages in key areas will automatically switch off at low vacuums/atmospheric pressure	Very low
	• Failure of welded joint	Very low	• System will leak to atmospheric pressure at a rate commensurate with the conductance of the failed joint. No control is deemed necessary.	Very low
Electric shock from a failed vacuum pump or powered fitting	Electric shock from frayed lead or failure of earth inside of equipment	Low	<ul> <li>All equipment is protected from short circuits by circuit breaker protection.</li> <li>Most circuits now have RCD protection.</li> <li>A program is in place to put all circuits onto RCD protection</li> </ul>	Very Low
Failure of support structures	Crush, fall, bump, trip hazards during and after failure	Low	<ul> <li>Use of vacuum standards to design equipment for safe usage</li> <li>Access to equipment at height via approved ladders or dedicated platforms</li> <li>Non acceptance of standing on beam line stands and mounts</li> </ul>	Very Low
breakages to	<ul> <li>Fragments of broken</li> </ul>	LOW	<ul> <li>Barriers around exposed equipment</li> </ul>	LOW

1) Risk assessment of plant – Accelerator Facilities Only

fittings and equipment protruding from vessels and beam lines	<ul> <li>parts become missiles.</li> <li>Cuts to personnel bumping into fittings</li> <li>Secondary injuries including electric shock from exposed wiring</li> </ul>		<ul> <li>that is deemed to be hazardous</li> <li>Access control into accelerator areas restricted to accelerator personnel and visitors who are escorted</li> <li>See electric shock hazard controls above</li> </ul>	
Over pressure of vacuum systems during venting	Positive pressure in beam lines when venting with gases from high pressure storage	Very Low	<ul> <li>Pressure relief valves in use at systems where regular vacuum cycling is necessary</li> <li>Use of portable pressure relief fittings where local venting is required.</li> </ul>	Very Low
Implosion of glass view ports	Shards of glass scattering towards operators	Low	<ul> <li>Use purpose designed commercial vacuum windows</li> <li>Use small diameter (&lt;50mm x 6 mm thick) quartz windows on vacuum systems where ion beams can strike. Quartz is not known to crack through but to develop small surface anomalies</li> <li>Use double rubber seals to support windows to minimise mechanical stresses</li> </ul>	Low
Broken film windows	Sudden venting of vacuum to atmospheric pressure	Very Low	<ul> <li>Damage to equipment – high vacuum gauges must have auto shutdown at low vacuums</li> <li>Vacuum system isolated with gate valve to minimise loss of vacuum in adjoining systems</li> </ul>	Very Low
Breach of accelerator tubes with SF6	Vacuum systems pressurised with SF6 gas	Low	<ul> <li>Large gate valves either end of the accelerator vacuum tubes will shut if pressure rises to maintain SF6 within the vacuum vessels</li> <li>Some gas will enter the vacuum systems outside of the isolated section. The pressure will be relieved through blow off venting ports at each end of the vessel.</li> <li>Oxygen depletion alarms will sound if dangerous levels of SF6 are released. Personnel will be ordered to leave building through PA announcement</li> </ul>	Low

- 2) Risk control measures for eliminating or reducing the assessed risks *See table above*
- 3) Registration or notification of design of plant *Not deemed necessary. See AS 4343-2005*
- 4) Licensing of plant Not deemed necessary. See AS 4343-2005
- 5) Maintenance of plant

*Regular maintenance applied via Accelerator Maintenance system. See ENV-I-076-004 Vacuum Systems Maintenance manual* 

- 6) Plant operations and risk controls *See table above*
- 7) Requirements in relation to specific types of plant *Only vacuum system plant*
- 8) Training and supervision Vacuum technology training is provided in-house to world's best practices. Additional training has been provided by the Vacuum Society of Australia through their training courses.
- 9) Monitoring and evaluation Regular maintenance applied via Accelerator Maintenance system. See ENV-I-076-004 Vacuum Systems Maintenance manual

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# Vacuum Technology

# and

# Vacuum Design Handbook

# for

# **Accelerator Technicians**

Prepared by David Garton November 2011 Revision 0 Vacuum Technology and Vacuum Design Handbook for Accelerator Technicians

This handbook is a compilation of information gathered from over 50 years of direct hands-on experience to applicable information widely available from the vacuum technology industry. It seeks to address common and specific vacuum technology problems whilst clarifying the design standards and philosophies adopted for use in the ANSTO accelerator facilities. The author wishes to thank the reviewers and the many technicians from ANSTO that have contributed directly and indirectly to this booklet.

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# Chapter 1

# Vacuum design standards and good practises for the Accelerator Area

- \* This chapter must be read in conjunction with the detailed vacuum information in this document.
- \* Based on best practises developed at ANSTO and other local Australian accelerator laboratories in conjunction with Australian Standards and industry technical guides, referenced throughout this document.
- \* Where a standard is unavailable for a specific design, best practices are used from industry's best practises.
- \* *Refers to all vacuum chambers, beam lines and other enclosures used on the accelerators or stand alone.*

## 1. Basic design

- All vacuum vessel designs must be consistent with the design standards in Australian Standard AS 1210-2010 and maintain a Hazard Level of E as per the design conditions tabled in "Hazard Levels of Pressure Equipment" in Section 2, Hazard Levels of Pressure Equipment in Australian Standard AS 4343-2005.
  - The vacuum equipment designed by/for the accelerator area maintains a Hazard level of E. All pressure equipment with a pV  $\leq$  30, pressure (p in MPa) x volume (litres)  $\leq$  30 is classed as Hazard level of E. For vacuum equipment in Hazard Level E the derating value of 0.1 is applied, ie 0.1pV  $\leq$ 30. Note the amount of stored energy at this level is very small. For example a Ø 1000 mm chamber, 1000 mm high will have a pV for vacuum equal to 7.85.
  - AS 4343-2005, Section 2.1.2 Typical hazard levels, part (d), states for Hazard Level E, "This equipment is usually exempt from special regulatory control but is <u>covered</u> by general plant safety regulations". See Appendix 7.
- All designs of chambers excluding general beam lines and fittings should be checked for structural integrity using finite elements analysis. Sound designs with acceptable structural integrity will be released for manufacture. A copy of the design acceptance tests must be filed with the project file. The standard finite element analysis can be performed utilising Solidworks. (Modelling for irregular shaped chambers, calculations Appendix 1 where appropriate for regular shapes)
- Stainless steel (304 or 316) will be used to construct chambers, beam lines and general fittings unless it is a *special* requirement to use other metals. Do not choose substances that have high outgassing rates or can contaminate vacuum systems.
- Measurement chambers and other large volume chambers should be cylindrical unless the chamber is an enclosure between pole faces (eg magnet box) or is an odd shape to accommodate steering plates or devices. When non-cylindrical chambers are required deflection of chamber walls should be modelled and taken into consideration.
- Other chambers including magnet boxes, ESA and deflector coffins to have suitable rib strengthened rectangular or curved sides where it is not practicable to increase the wall thicknesses.
- Minimum wall thickness calculation methods can be found in Appendix 1. As a general rule care must be taken when using formulas for vacuum calculations as historically many units other than SI units have been used to form some equations. For example length in cm, and pressure in torr is common in old notes.

- Top and bottom flanges to have integrated structural support rim which becomes the flange and seal for chamber base and top (user) flanges.
- Ports must be a suitable length to ensure unobstructed insertion for screws into flanges. Suitable weld preparation must allow for adequate weld penetration. See Welds below.
- All internal edges must have a smooth radius. No sharp edges to be left after welding or surface finishing.
- All machined finishes other than vacuum seals to be equal to or better than 1.6 microns
- No sections within the vacuum space will have metal to metal contact where gas can be trapped unless it is at the edge of a seal joint. When this is necessary, pump out grooves must be incorporated in the design.
- Vacuum windows shall be made from toughened glass or quartz. No plastic to be used for windows unless the window is a flange on a chamber that is designed to withstand more than 100 kPa. The large plastic flange/windows should be made from >18 mm thick up to Ø200 and >25 mm thick for up to Ø400 in clear Perspex or polycarbonate.
- For ANTARES beam lines the preferred flanges are Dependex, nominal diameter 100 mm. See section 8 for detail on Dependex flanges.
- For STAR there is no preferred flange type. It must be selected depending on the desired base vacuum and interface to other adjoining flanges.
- As a guide, the MDC catalogue from Vacuum Products Corporation 2003 onwards, Building Blocks for Vacuum Science and Technology, provides vacuum tubing dimensions for most common sizes. The dimensions must be assumed to be minimum dimensions. Other tubing sizes should be calculated using the formulas in Appendix 1.
- Vacuum pumping ports leading to the vacuum pumps must be designed to optimise conductance.
- Pressure relief valves to be incorporated where a vacuum system can be vented to >10 kPa above atmospheric pressure.
- ASME state that the design of an external pressure vessel must consider Material type, diameter of chamber, unstiffened length, temperature and wall thickness

# 2. Welds

- TIG is preferred for vacuum sealing welds as minimal scale and flux is produced. Electron beam welding is suitable for thin walled materials. TIG produces high quality welds with or without filler rods to produce flat, smooth well penetrated welds. TIG comes into its own where the weld preparation allows for melting together of the parent materials without filler rods. MIG has a higher deposition of filler material so care must be taken to ensure welds are kept smooth and flat.
- No standard exists for vacuum welding of compact scientific vacuum equipment where pressures approaching zero are necessary. Equipment designed in the accelerator area and welded in the ANSTO workshops since 1990 has had penetrations of ≥1mm but ≤2mm on a butt weld and ≥2 mm on a fillet weld. Experience has demonstrated the adequacy of these figures. No failure reported.
- For thin walled materials ie < 1 mm, such as bellows, manufacturers' will use their industry standard. ANSTO does not weld thin wall materials.
- Weld preparation must be provided where possible to ensure even heat penetration.

- Electric arc with rods is not recommended due to flux trapping. This can cause pits which have long lasting outgassing periods.
- All welds that form a seal against atmospheric or other positive pressures must be made to the internal side (vacuum side) of the joint.
- Tack (stitch) welding is recommended on the external side only with less than 50% of the diameter covered in small intervals. This minimises the chance of trapped voids and facilitates He leak testing post welding.
- All welds must be free from pitting and scale. Must be cleaned to bare metal. If chemicals are used to clean welds care must be taken to remove all traces of chemicals.
- Some chambers such as magnet boxes will need to have some joints welded externally due to access problems on thin chambers. In this case, good penetration must be achieved to ensure minimal gas trapping points.

## 3. Surface finishes

- The inside of the chamber and ports must be 1.6 micron or better for mechanical finishes.
- Can be chemically cleaned (polished) but all traces of chemicals must be neutralised and removed prior to use in vacuum.
- All machining oil to be removed with an alcohol scrub then acetone rinse, minimum. The use of kerosene, turpentine or other oily solvents is not encouraged however where they are used the final clean must be with acetone followed by alcohol. Water based cutting fluids can be used as they are more readily removed therefore having less effect on outgassing.
- Nitrile gloves to be used when assembling cleaned components. Other un-powdered gloves may be used but only those that do not react with the solvents being used.
- Care must be given to cleaning and handling surfaces that will be exposed to vacuum. Gloves should be used to minimise the transfer of dirt and oils from the skin to vacuum surfaces.
- A suitably clean area should be used for the preparation of vacuum systems and all tolls used should also be cleaned (degreased).
- Stainless steel can be cleaned in nitric acid to remove scale that remains from rolling or wire cutting. It is may also be used to prepare stainless steel components for use in ultra-high vacuum systems.

# 4. Flanges

- Flanges must be standard flanges selected from the group referenced this document for compatibility reasons. Exceptions may be made but all 'o' ring seals must be to Australian Standard AS 2842-1986. External flange rings not in contact with the vacuum space may be aluminium alloy. Stainless flanges are not economical or necessary unless the seal requires forces to crush metal seals such as copper ConFlat, aluminium or indium wire.
- Minimum thicknesses for top and bottom flanges to be calculated using the standard in Appendix 1 Structural Calculations for Scientific Vacuum Vessel Design.
- All other flanges to have threads consistent with their design standard.
- ANTARES Primary flanges will be Dependex unless interface is necessary to accommodate other standard flanging.
- For Dependex, screws to be imperial sizes consistent with UNC series.

• STAR utilises many of the major vacuum flange/seal designs. The most appropriate design must be chosen that meets the specific application.

## 5. Seals

- 'O' ring sizes and sealing groove dimensions must comply with the Australian Standard AS 2842-1986. Adjustments to groove sizes can be made where specific 'crush' is necessary for special applications.
- Viton rubber 'o' rings to be used unless a special requirement for an alternate elastomer is identified.
- Standard flange and seal sizes to be used unless an application prohibits their use.
- It is recommended that vacuum sealing surfaces are cleaned just before a seal is made to minimise the possibility of dust and hair settling on sealing surfaces.
- Metal seals may be used in specifically designed sealing surfaces including, aluminium and indium wires. They are very good as ultra-high vacuum seals.

## 6. Screws

- All screws for flanges other than Conflat will be made from stainless steel in either 304 or 316 grades.
- Screws used in Conflat flanges will be made from A2-70 or A4-70.
- All screws to be socket head unless access necessitates hexagon or other special head. Slotted or Philips head must not be used.
- Stainless steel flat washers to be used under all screws where practicable. Do not use lock or star washers on standard flanges.
- In the vacuum space, no screws to be used in holes unless the holes are through holes and can be pumped from both sides. Screws can be used in blind holes if they have a minimum Ø1 mm hole drilled through the centre to allow trapped gas/air to escape.
- Unless a standard flange uses non metric thread, all screw threads must be within the metric series. Unless there is a specific need, avoid using intermediate metric sizes, that is, 0.5 mm steps such as M1.5 mm, M2.5 mm etc.
- Anti-seizing compounds can be used on threads but extreme care must be taken to ensure they do not enter the vacuum space. Graphite powder can be used as a lubricant inside the vacuum space. It can be applied using ethanol to wet the surface to be lubricated. Allow solvent to evaporate prior to pumping.

# 7. Feedthroughs

- Sliding seals should have dual 'o' ring seals with a gland formed seal at least at one end. The shaft finish must be 0.8 micron or better.
- All sliding shafts must have retainers to stop them being pulled through into the vacuum space.
- All coax feedthroughs to be glass or ceramic hermetically sealed. No plastic.

#### 8. Lubricants, vacuum greases and epoxies

- Only vacuum specific grease and oils to be used in vacuum systems.
- Silicone based or particular Apiezon family lubricants design for vacuum use, are recommended on sliding seals. Thin smears only.
- No vacuum greases should be used unless there is a small leak through a damaged sealing surface and all other options are not feasible. If grease must be used it must be designed for high vacuum use and only a very thin smear to be used.
- Do not use grease to fill suspected leaks in welds or fittings.
- Where a vacuum epoxy must be used the surface must be well prepared to ensure appropriate bonding. Care must be taken to ensure the epoxy seal doesn't create a trapped void of air which may become an outgassing problem. 'Torr-Seal' is commonly used due to its relatively low vapour pressure, and thermally stable to 120°C. (this is a Varian product)
- Vacuum oils are not recommended for general lubrication as they can creep on surfaces spreading contamination and increasing the outgassing surface.
- See Appendix 3 section 1.5.

#### 9. Mounting

- The load of the chamber assembly must rest on a standard accelerator beam line table ANSTO, HVEC, HVEE or NEC or a purpose built stand that minimises vibration.
- Vibration decoupling will be achieved by supporting the chamber separately from the vacuum pump and using an edge welded bellows between the chamber and pump.
- For ANTARES beam line vacuum equipment the beam axis alignment is nominally 1750 mm from floor height.
- For STAR beam line vacuum equipment the beam axis alignment is nominally 1193 mm from floor height.

#### 10. Access

- The working face of the chamber must face an area that is easily and comfortably accessible. The floor space immediately in front of the working face should be raised using steps to facilitate access.
- Heavy or awkward shaped vacuum equipment must be designed to be lifted with the aid of over cranes for example lifting lugs.

#### 11. Testing

- Vacuum equipment must be tested prior to routine use to minimise time spent chasing leaks in completed systems.
- Vacuum vessels must be helium leak tested to ensure all seals are better than  $1 \ge 10^{-10}$  Pa.m<sup>3</sup>/s ( $1 \ge 10^{-9}$  mbar.l/s). As a standard, during a leak test there should be no deviation from this baseline level for a leak-tight system. A record of the test is to be noted on the engineering drawing showing, date, maximum leak rate, leak location/s, leak detector used and operator name.

- For all other vacuum equipment in-service testing may be necessary to allow complete operation of various feedthroughs and positioners.
- Caution must be taken before positive pressure testing vacuum equipment as the equipment may be irreversibly damaged. Warning Do not use water testing on any vacuum equipment.
- A Residual Gas Analyser (RGA) can be used to give more detailed information about the gas composition inside a vacuum volume. An RGA can be used as a substitute for a helium leak detector with the benefit of providing more information about outgassing, or virtual leaks inside the vacuum volume. The pressure of the vacuum system must be  $< 1 \times 10^{-2}$  Pa to prevent damage to the filament inside the instrument. Instruction manuals must be checked before using the RGA head to ensure safe and effective use.

## 12. Operation and Continued Monitoring

- All chambers should have a high vacuum gauge sensing within the chamber space. This includes all adjoining compartments that may be isolated by a valve.
- The chamber must have valves attached that allow complete vacuum isolation from the beam lines and other equipment.
- To protect high vacuum gauges against the effects of operating in low vacuum, high vacuum gauges should be automatically isolated if the vacuum pressure rises above  $1 \times 10^{-2}$  Pa.
- High vacuum gauges must be cleaned at minimum every 3 years or whenever a gauge develops instability as compared with adjacent gauge readings.
- Systems indicating higher than normally observed pressure must be isolated and repaired. A helium leak test is recommended if the leak location is not clear.
- To avoid undesired contamination, vacuum chambers should be vented with dry nitrogen or argon instead of air. This minimises water vapour and oil from depositing on the vessel walls.

# Chapter 2

# Design Criteria, Information and Philosophies for Vacuum Systems used in the Accelerator Area

#### Overview

In an accelerator system ion beams are transported from ion sources through to end stations (or detectors) through vacuum space maintained inside of beam tubes (lines). The relatively low number of gas molecules in the vacuum ensures a higher mean free path for the accelerated ions and hence less loss of ions through collision which is crucial especially for AMS measurements when counting a very small number of ions. A high vacuum also minimises the formation of plasmas in an electric field at high voltages which can avalanche into spark breakdown damaging sensitive equipment.

The vacuum systems used in the accelerator area are designed to be fail-safe to protect people and equipment. A very high proportion of problems with vacuum systems relate to, seals leaking on first use, being incorrectly mounted or failing due to aging 'o' rings. Other problems, however less frequent, include mechanical damage to sealing surfaces or fatigued parts such as bellows. The problems are all characterised by a poor vacuum or a previously steady vacuum that begins degrading slowly as observed during the operation of the vacuum system over its life time. The remaining problems are due to poor welds or vacuum fittings breaking. Poor welds are usually found during the initial helium leak test. They are remedied by either removing and re-welding the section or using a commercial vacuum epoxy. The choice is dependent on whether the weld is on a structural component or just a seal. Broken vacuum fittings are replaced or repaired.

Within the accelerator area. physicists work directly with technicians who apply broad knowledge and experience in vacuum technology to develop their concepts for use in the accelerator environment. This mix has been successful for 50 years. However not all designs are completed in the accelerator area. There are cases where commercial projects call for a more production based design or designs of chambers with complex noncylindrical shapes. These may go to the ANSTO drawing office for completion or a specialist vacuum design company.



Fig 1. ANSTO designed and developed beam lines on the STAR accelerator in building 22

It is not possible and often not necessary to standardise the design requirements for all components used in the accelerator area so the following information is intended to help designers of vacuum equipment to comply with accelerator specific and Australian standards. As of 2011, there are no known commercial workshops in Australia who specialise in the production of vacuum vessels. Local representatives from vacuum suppliers can provide access to overseas specialist workshops.

# 1. Terminology

**ANTARES** – Australian National Tandem for Applied RESearch. The HVEC FN Tandem accelerator in B53.

Backing – To pump on the exhaust (back) of a high vacuum pump

Beam line – The evacuated tubes that carry the ion beams from the ion sources to the end stations

**Cryopump** – Cryogenic vacuum pump. Uses a helium cooling interface to the vacuum space to freeze gases.

**Dependex** – A type of flange which is the standard flange used on ANTARES and ANSTO built beam lines.

**Fore line** – The vacuum line, tube, pipe, whatever between the high vacuum pump and the backing pump

High vacuum – See details in Chapter 3, para 1.3

ISO - International Standards Organisation

KF – Klein (small) Flange

LF – Large Flange

Low Vacuum – See details in Chapter 3, para 1.3

Outgassing - the liberation of gaseous vapours from the surfaces within a vacuum space

Pa – Unit of pressure, Pascal. 1 Pa = 1 N/m<sup>2</sup> = 0.01 mBar = 7.5 mTorr = 1.45 x 10<sup>-4</sup> PSI

**Pump down = Pump out** – evacuating gas from a vacuum space

**Roughing or Roughing-out** – Usually used to describe the initial transfer of gas from atmospheric pressure to low vacuum.

STAR – Small Tandem for Accelerator Research. The HVEE Tandetron accelerator in B22.

Turbo – an abbreviation for turbomolecular vacuum pump

**Ultra-high vacuum –** See details in Chapter 3, para 1.3

Venting – the equalisation of negative pressure to atmospheric pressure in vacuum equipment

Vessel – generic for a vacuum chamber where an instrument is mounted or interfaced into the vacuum space.

# 2. Standard Units

#### 2.1 General

Although metric standard is adopted across all ANSTO accelerator facilities it does not mean that all equipment is metric as the US built FN Tandem is mostly imperial, conversely, the Dutch built HVEE Tandetron is all metric. There is a combination of metric and imperial devices used throughout the facilities but designers must ensure that where practicable all new designs are metric.

#### 2.2 Threads

New equipment for use in the accelerator area, where practicable will be designed with metric standard threads.

• Contrary to the standard, on ANTARES there is a combination of threads in use. The accelerator high voltage generator and parts that have originated from the United States are

mostly imperial. Typically, UNC and some UNF. Most beam lines and endstations are metric but all Dependex and most Conflat flanges are imperial.

• ON STAR all threads are metric. ANSTO designed and built beam lines are metric except for the Dependex flanges which are UNC.

Care must be taken to identify threads and use the correct screws. Where practicable, metric threads are to be used other than on Dependex flanging.

#### 2.3 Pressure

All pressure indicating devices shall be in the SI unit, Pascal. Historically, Torr was used but this was replaced by Pa in the late 1980's. Most international accelerator labs use mbar. The conversion is as simple as multiplying mbar by 100 to get Pa. Pascal refers to a physical force of newtons per metre squared.

# 3. General Design Philosophies

There are many varieties of vacuum chambers and equipment in use in the accelerator area. Most have been designed and made at ANSTO. Examples include vacuum equipment measurement chambers where samples are measured, beam line transport components, ion source components or sensitive detectors, to name a few. Each has its own unique characteristics and vacuum design considerations.

In order to maintain a suitable mean free path for ion beams whether being transmitted from a source or scattered from a sample into a detection system, high to ultra-high vacuums are necessary. In a high vacuum the density of gas is low enough to minimise electron flow from surfaces at high voltages to surfaces at lesser potentials. It is therefore prudent to ensure designs of vacuum equipment that directly couple to the accelerator are designed for a minimum of high vacuum operation.

In every case the designers must ask the following questions:

- What shape? How big? Where will it fit? How will it be mounted? What level of vacuum?
- Will it be ridged enough and have low enough distortion to mount ion optical components?
- Is the design flexible enough to enable small scope changes in the future? Is it designed with consideration for reuse in another application when the project finishes?
- Does the design use vacuum technology best practises?
- Does it meet Australian and local standards where applicable?
- Is it a safe design?
- Is it achievable with resources available?
- Is it cost effective. Can we modify existing equipment to achieve a similar result?
- Does it have low environmental impact?

#### 3.1 The Shape

The first consideration is to define the actual use of the vacuum equipment relative to the experiment. Where possible cylindrical shapes should be considered first as they are stronger under vacuum and consequently being round, ports can be welded into the chamber aligned radially to the centre which ideally aligns instruments including detectors and cameras with the intersection point of the ion beam and the sample being measured.

Non-cylindrical chambers must be carefully designed to included adequate strengthen ribs to minimise distortion and hence misalignment of instruments.



Fig 2. A few examples of vacuum vessels used in the accelerator area

# 3.2 The size

The size is dictated by the space needed inside of the vessel or equipment in order to meet the operational demands. In the case of measurement chambers too big may mean problems with the distance between samples and detection systems and too small may mean manual handling of samples may be inhibited. Large chambers may have to be certified as per AS 4343-2005, Hazard Levels of Pressure Equipment in Australian Standard.

#### 3.3 Location

It will be an agreement between the accelerator user groups as to where equipment and beam lines will be located. This agreement will endeavour to minimise conflicts in beam line space, usage, accessibility and resourcing.

#### 3.4 Mounting

In general equipment must be secured onto the floor unless it is designed as mobile. An accelerator compliant beam line table should be used with standard beam line supports. Vibration must be identified during the design stage and decoupling stages provided.

Insertion bellows must be provided in long sections of beam lines to enable compression of the beam lines to facilitate dismantling and to ensure minimal strain on vacuum joints.

Designs must consider trip, bump and fall hazards. Structures that support platforms or heavy equipment that may otherwise fall must not be reliant on the structure supporting the vacuum chambers and beam lines. For example, ladders, walkways, etc.

- For ANTARES beam line vacuum equipment the beam axis alignment is nominally 1750 mm from floor height.
- For STAR beam line vacuum equipment the beam axis alignment is nominally 1193 mm from floor height.

## 3.5 Flexible design

It is prudent to ensure where practicable designs ensure some redundancy for feedthroughs, access ports and internal space. A major problem with early chambers was the lack of space allowed for inserting samples by hand or manually manipulating internal equipment and wiring. It is easier to design flexibility into the chamber or equipment at the design stage rather than having to dismantle it and have it reworked in the workshops later.

## 3.6 Can it be made?

When designing any equipment seek advice from a qualified machinist. Often designs include machining that may be very expensive or not possible. Early in the design phase ensure that the resources are available to service the vacuum equipment. It is recommended to consider the following early in the design stage:

- Power
- Cooling water
- Air cooling
- Signal lines
- Control lines
- Add-on electronic and mechanical instrumentation

## 3.7 Safe design

There are no cases that the author has been able to find of scientific vacuum chambers or equipment, like those used in accelerator facilities, failing and causing injury. Vacuum pressure is relatively low, -100 kPa, or about half the equivalent magnitude in a car tyre.

For safe designs the structure must meet the minimum dimensions as calculated in Appendix 1 - Structural Calculations for Scientific Vacuum Vessel Design.

Care must be taken with the design of the following:

- Glass view ports (most glass breakages are in the form of a crack which leak to atmospheric pressure quickly without the spread of shards)
- No internal sharp edges; in the case where a person must insert their hands blindly to access components.
- Venting limits pressure relief for positive pressure?
- Evacuation rate regulation; especially in the case where very thin material windows separate different pressure systems within a single vacuum space for example beryllium windows on x-ray detectors.
- Equipment mounts/stands; where an operator must climb up high to operate devices on the system
- If oil type vacuum pumps are used then mist filters must be fitted to the exhausts.

# **3.8** Cost effective design

All *bells and whistles* may not be necessary even after considering some design redundancy for future changes. Where possible, designs should consider integrating components that are available off the shelf. Designing what has already been designed by someone else is wasting time and effort.

It may be cheaper to modify an existing chamber for use rather than start from the beginning. Many chambers used around the accelerator area have successfully had one or more lives.

## **3.9** Design by numbers

This is a matter of ensuring structural integrity and being able to prove it. Likewise, designing the vacuum pumping system requires calculating the most appropriate size pump rather than going for the "biggest"!

There are formulas in Chapter 3 that allow must crucial calculations to be made from wall thicknesses to conductance. Good enough is not acceptable. Replication of previously designed equipment is acceptable.

#### **3.10** Australian Standards

Compliance is mandatory however there are few standards that are relevant to scientific vacuum vessels and equipment. The industry has developed its own standards for flange and seal designs which can be seen in the catalogues of most leading manufacturers of vacuum equipment. It is the responsibility of the designer to ensure compliance with all relevant Australian Standards. Chapter 1 discusses what standards are included.

#### 3.11 Environmental impact

Most vacuum systems used in the accelerator area today are designed to be oil free. That is, they do not use oils for lubrication of vacuum seals. There is however some oil vane and diffusion pumps still in use but the oils are responsibly disposed of through waste management at ANSTO. Some old diffusion pumps had been used with mercury as the pumping agent. Personnel must be cautious of using old diffusion pumps and their associated backing pumps as there may be residual mercury on surfaces and in the backing pump oil.

Solvents used for cleaning are used in small amounts and usually for wiping rather than immersing. Where baths are needed the solvents are reused when possible. The solvent bath used for degreasing, self recycles the solvent by design however there is a small loss of solvent over time into the atmosphere.

Where possible solvents used are selected with the environmental impact in mind.

#### 3.12 Manufacture

If ANSTO cannot provide the level of manufacture quality in-house then scientific vacuum equipment is best manufactured by companies that specialise in high tolerance machining and welding. A general metal fabricator will not suffice.

Attention must be paid to considering standard sized tooling during manufacture. Some companies will charge for the purchase of tooling that they consider to be non-standard.

It is imperative that the drawings for manufacture state all tolerances and qualities for the finished product.

On all design drawings, where the angle of the welded ports is crucial, ensure angular tolerances are clear and achievable. If necessary, specify the use of mandrels to hold ports in place during welding.

Some success has been made outsourcing the manufacture through vacuum companies to overseas manufacturers who specialise in high quality small runs.

#### 3.13 Testing

Testing the vacuum equipment or chamber is essential to establish compliance with the design and the all important ultimate base vacuum. Helium leak testing is the standard method used for chambers and most equipment however on some equipment in-service testing may be the only option. This is where add on vacuum equipment doesn't change the original base vacuum of the system it is connected to.

#### 3.14 Inspection

All parts should be cleaned prior to inspection. This doesn't need to be the final clean but enough to ensure all welds and surfaces can be clearly inspected. Welds must be checked for pits and alignment along the weld lines and seals must be checked for surface finishes. All crucial dimensional must be checked. It is recommended that any non-complying findings are photographed and documented in the project file. All non-complying parts should be reworked rather than *patched up*.

#### 3.15 Certification

As most vacuum vessels and equipment in the accelerator area fall within Hazard Level E of AS 4343-2005, certification is a local process. For large measurement vacuum vessels, results from the inspection and leak testing will be recorded on the engineering drawing and registered in the local accelerator register and/or ANSTO drawing register as appropriate.

#### 3.16 Documentation

For all large measurement chambers requiring any level of design calculations, design documents must be kept with project files.

#### 3.17 Maintaining

Vacuum chambers and beam lines do not require high levels of maintenance other than a general periodic clean and occasional seal replacement. Vacuum fittings and devices will have their own level of maintenance so access to these components must not be difficult.

The design must be easy to maintain especially moving parts, sliding or rotating seal and other parts in the vacuum space that may wear. Also vacuum pumping systems that may be mounted under or near the vacuum chambers. Generally any part that will require any level of routine maintenance must be accessible without the need for a major disassembly.

Vacuum pumping systems other than oil free systems, connected onto beam lines and chambers must include suitable oil traps between the backing and high vacuum pumps and where mist filters on all exhausts.

Some high vacuum gauges will require periodic cleaning. On ANTARES high vacuum cold cathode gauges are scheduled for cleaning every 12-18 months or max 3 years for difficult to service or less crucial instrument locations. Otherwise gauges will be cleaned on demand within the periods.

# 4. Performance and Operations

#### 4.1 Ultimate Vacuum

The ultimate vacuum is the maximum vacuum or lowest pressure the vacuum equipment typically reached after 3-5 days of uninterrupted pumping. This is a benchmark value which is used to assess the long term quality of the vacuum system. It is sometimes referred to as the *base vacuum or static vacuum*.

Generally, vacuums in the beam lines and chambers are well within the high vacuum range. Vacuums lower than this range will indicate a higher than normal gas load, a failing seal or a pump malfunction. On the accelerators the two main types of high vacuum pumps are turbomolecular and cryogenic pumps. For a system designed for high vacuum or better with no leaks and low outgassing then vacuums better than  $4 \times 10^{-6}$  Pa should be readily achieved.

The ultimate vacuum is improved by the correct choice of materials, seal types, high vacuum pumps used cleanliness and correct operation. All fittings must be designed or selected deliberately for high to ultrahigh vacuum use. Surface preparation and cleanliness will contribute to improved vacuums. A finger print can outgas for long periods of time. A bigger (higher speed, higher ultimate vacuum) vacuum pump does not mean a better vacuum!

#### 4.2 Working Vacuum

The working vacuum may be lower than the ultimate vacuum due to sample cycling (changing), introduced gas loads from adjoining systems or outgassing samples. A system must be designed to ensure the high vacuum pumping system has the optimum pumping speed and ultimate vacuum. Once a vacuum system reaches the "working vacuum" little operator intervention is necessary to maintain it but there are some important points regarding ongoing monitoring to ensure a failure is not immanent.

For sections of beam lines that are holding a static vacuum a cursory glance at the local vacuum gauge will indicate the stability of the vacuum. Generally, the (vacuum) pressure will change slightly throughout a day for example due to diurnal changes in ambient temperature and perceived changes due to ionisation gauges "wandering" as they become dirty.

In sections of beam lines that are in the vicinity of operating beam lines or near end stations the vacuum will be continually changing. Factors that influence these changes include increased gas loads from samples outgassing or surfaces that have just be exposed to high humidity, outgassing due to temperature changes within the vacuum space (including bake outs), outgassing from ion beams striking plastics, etc. Gas molecules don't have a preference to whether they travel towards or away from a pump during molecular flow so the source of gas can come from just about anywhere in the open system. These factors must be considered when monitoring vacuums throughout an entire system.

If a vacuum system's pressure rises slowly over many days or weeks if it is not related to the cryopump loading then it may be a seal failing. In this case a helium leak test is recommended.

A fundamental flaw in many vacuum system designs is the neglect for monitoring the backing pressure between the backing pumps and turbo pumps. Pirani gauges which are best suited for this role are relatively cheap and are the best diagnostic tool for determining overall vacuum system performance. The backing pressure should be consistent with changes in the high vacuum pressure. By monitoring the backing pressure over time, the base vacuum will be realised relative to the high vacuum so a pending failure of the backing pump can be determined, hopefully, well before it happens.

Care must be taken to maintain systems that have the potential to degrade over time for example an oil vane roughing pump will back stream oil mist that may accumulate in the high vacuum side of the system over time. This will contribute to lower vacuums due to higher gas loads from the partial pressures of contaminants.

#### 4.3 Sustainable Operation

Now that the vacuum system is designed, manufactured and commissioned for routine service, operating procedures have to be defined and adhered to, to maintain reliable trouble-free service. There are 3 processes that summarise the continuous cycle of operating a vacuum system:

- Pumping down from atmospheric pressure
- Using the working vacuum
- Venting to atmospheric pressure

The accelerator beam lines are divided into stand alone vacuum systems but in essence function as one. Each system can impact on a neighbouring system if things go wrong. In the case of end stations which cycle up and down in pressure more than any other devices an operator must be 100% sure of the pump down sequence in order to avoid damaging, pumps, samples, high voltage devices, detectors, etc. It is possible to shut down the accelerators as well. Most sections of the accelerator vacuum systems are interlocked to isolate a pressure surge to small area. Beam line vacuum systems are design so that both the vacuum pump and the beam line are isolated via gate valves. This is a typical arrangement for endstations as well.

The following must be read remembering that all 3 processes work in a cyclic fashion in a continuous loop.

#### 4.4 **Operational Protocols**

Key vacuum systems operate continuously, 24 hours per day, seven days per week. There are several vacuum systems that are cycled on and off as usage demands. All vacuum systems have one or more high vacuum gauges, some of which are interlocked to make the systems fail-safe. Any slow leaks or major catastrophic failures will close valves and isolate the system. In the few cases where no interlocks are used, a catastrophic failure will result in the high vacuum pump shutting down.

- For a <u>turbo pump</u>, as the pressure rises the gas load on the pump will cause the pump to draw more current to maintain it's speed. It will eventually fail when the current trip level is reached in the controller.
- For a <u>cryopump</u>, as the pressure rises the inflowing gas will condense or freeze onto the in the pump until it begins to warm up. Eventually there will be a thermal trip activated o the compressor which will shut is down. In both cases it is fail-safe.

As part of the accelerator's maintenance system, daily monitoring of all vacuum pressures is carried out. Where a pressure is seen to rise by a few percent without a particular reason, further monitoring will occur and corrective action started. Instantaneous failures are dealt with immediately. All observations and whether any maintenance carried out is noted on the maintenance log.

Where a vacuum system is isolated for service or where it has failed the appropriate tags are placed on the power leads.

Unattended out of hour's operation notification is not normally used as vacuum systems run continuously.

#### 4.5 Pump down

#### General

Pump down time is when most mistakes are made and significant time lost. Before a pump down begins the operator must ensure that all seals that were disturbed had been cleaned and replaced if damaged. The system must then be sealed ready for pump down and the roughing-out valve on the rough-out port, closed.

If a dry pump is to be used, such as a scroll pump, for rough-out then the tube is connected to the pumping port. If an oil vane rotary pump is used then ensure the cold trap has been filled beforehand.

#### Rough-out

Start the roughing pump and allow it to reach base vacuum. This is your first indicator of whether you have a leak in the system being pumped and when you have reached the pumping limit of the

pump. Slowly open the pump out valve on the system. This is where mistakes are made and damaged caused. Relatively speaking the volume in most sections of beam lines and endstations is small and the bulk of the gas can be removed very quickly. The problem is that a reduction in pressure too quickly can cause mechanical shock to fragile parts that must adjust slowly in order to minimise stress such as thin windows on detectors and detectors or devices that contain carbon foils.

Operators must always open roughing out valves slowly then gradually increasing as the gas load reduces. The change in the sound of the loaded pump is a very good indicator. A good design for more sensitive pump outs is to use a metering valve in line with the rough-out port to act as a regulating orifice.

#### Cryogenic Vacuum Pumps

For systems using cryopumps as the primary high vacuum pump it is essential to bring the base pressure down to the limit of the roughing pump before changing over pumping to the high vacuum pump. This reduces the gas load to be trapped (captured) on the cryo pump and extends the service life of the cryopump. Cryopumps are widely used on ANTARES as the primary high vacuum pumps but their limitation is that the warm up as they trap more gas causing a rise in base pressure. That is, when a cryopump is reaching saturation the temperature on the surface increases. More energy is required to hold the molecules on the surface and since it is almost fixed by design the temperature increases. A cryopump's effective pumping speed depends on the freezing and boiling points of the gases being pump relative to the cryopump's temperature. As the temperature increases more molecules leave the surface (boil away) raising the pressure. Some molecules are re-trapped as they hit the surface of the arrays and loose kinetic energy but they have a short residency time and soon add to the rising pressure in the system. Eventually the base pressure in the system becomes unusable so the pump must be regenerated.

#### Turbomolecular Vacuum Pumps

The other type of high vacuum pump widely used in the accelerator area is a turbo pump. Unlike a cryo pump that traps or captures gas molecules a turbo pump transfers the gas by conducting it almost one-way out of the vacuum space. The lowest roughing pressure before changing to a high vacuum pump is not as crucial when a turbo pump is being used. In saying this, the bulk of the gas must be removed before changing from roughing out to high vacuum pumping otherwise the vanes on the turbo pump may be overly stressed due to the pressure surge. A typical changeover pressure range is 5 - 50 Pa.

The optimum changeover time to minimise pump down times can be estimated with the assistance of the formulas found within the document. Letting the roughing pressure reduce to the limit of the roughing pump before changing over to the turbo pump is an inefficient method of system pumping. A turbo pump is more efficient at removing gas molecules as the flow moves towards molecular flow. Changing pumping to a turbo pump just before molecular flow is reached will increase the pumping speed and base vacuum will be reached sooner.

#### Pumping Speed

Each type of gas will pump away at a different speed depending on the mean velocity of the molecule which is a function of its mass. A hydrogen molecule of 0.02 kg/mol and mean velocity of 1762 m/s will reach a pump much quicker than say a Xenon atom of 0.13 kg/mol with a mean velocity of 217 m/s.

#### **Limitations**

In rare circumstances roughing pumps may be left pumping high vacuum systems by mistake. It is unlikely to damage either pump but the ultimate vacuum will not be reached as the roughing pumps are ineffective at conducting away molecules in molecular flow. Water vapour and residual solvents are difficult to remove from a vacuum space with *normal* vacuum pumping. Most low vacuum backing pumps provide gas ballast which can speed up the removal of water vapour and solvents that may otherwise condense inside of the pump during the compression stage. By introducing a small amount of air near the outlet of the compression stage the small increase in pressure helps carry water vapour and solvents to the exhaust before they can condense.

Some vacuum systems have been designed to allow chambers and beam lines to be heated. Heating causes contaminants to be removed from surfaces at lower than normal vacuums. The higher the temperature the faster the removal will be. Care must be taken to ensure heating doesn't inadvertently cause plastics and elastomers used within the vacuum space to outgas. Most bakeable systems use ceramics and metals only.

It is important that where vacuum systems are permanently connected to high voltage devices for roughing out, the insulated (plastic) pump out lines between the roughing pump and high voltage device must be brought up to atmospheric pressure before high voltages are reapplied. Otherwise the low molecular density of gas in the tube may ionise causing burning of the tube or even spark damage that can puncture the tube causing a leak.

# 4.6 Venting

#### General

Venting can cause damage to vacuum components just like pumping out a system from atmospheric pressure too fast.

#### Venting Speed

As discussed earlier the volume of vacuum systems on the accelerators is measured in litres to a several tens of litres. A catastrophic loss of vacuum will be more of a brief audible event rather than a dangerous mechanical failure. Little energy is required to change these systems by 100 kPa. There are a few devices in the vacuum space that may be damaged due to pumping out too fast but none that are considered a danger to personnel.

Other than venting too fast the second point to consider is the introduction of water vapour if venting with air. Key vacuum systems on the accelerators are vented with either dry nitrogen or argon both of which are delivered through a closed reticulation system from gas bottles. The choice of gas is dependent on molecular contamination within the system for example nitrogen is not preferred to vent the 846 ion source as nitrogen is an element that is known to bond with other elements causing molecular ion beams.

#### Regulation of Flow

There are many low flow regulators and rotameters (tubular flow meters) available that provide adequate control over venting. A simple in-house gas regulating system which works well is a tee piece connected between the venting port and the venting gas supply line. The centre port of the tee piece faces upwards and a ping pong ball sits over the hole with a cage over it so that it cant be blown away. Under the ball is an 'o' ring seal. Before the venting valve is opened the venting gas is allowed to flow. The pressure of the gas lifts the ping pong ball of its seal. The gas flow is adjusted depending on the height of the ball above the seal. The ball can only travel a centimetre or so. Once the flow is set the venting valve is opened and the now regulated gas flows into the vacuum space and the balls drops onto the seal stopping air from entering the vacuum space. As the pressure reaches equilibrium the ball begins to rise off the seal until eventually it rises to the original set height indicating the vacuum space is now at atmospheric pressure.

#### Precautions

- Before venting a vacuum system it is wise to isolate all ports and beam lines that must remain under vacuum. To save stressing a beryllium window on an x-ray detector due to the changes in pressure the system should be designed so that the x-ray detector can be isolated with a gate valve. The same logic can be applied to all equipment that may be effected. It is also important to ensure all power supplies and high voltages that may be hazardous to personnel or equipment are isolated prior to venting. This can be done either by administrative protocols or using a vacuum switch such as programmable relays in a vacuum gauge controller.
- Some vacuum gauge controllers use the signal from a Pirani gauge measuring at the same location as a high vacuum gauge to switch off the high vacuum gauge when the pressure becomes too high. This saves the gauge from ionising air when the system is at atmospheric pressure which will significantly reduce its life time.
- Another problem with venting too quickly is the disturbance of dust. Accelerator tubes must be vented slowly to minimise equalisation stresses and also the disturbance of dust. In this case a metering valve is used to limit the vent flow rate.
- Venting can cause loud high frequency noise so care must be taken to dampen the noise to a comfortable level by controlling the air admittance rate, or use hearing protection.
- Most beam line vacuum pumping systems are interlocked to isolate either the beam line or pump or both. In the event where an accidental venting occurs the gate valves will close minimising the effect on surrounding systems.

# 5. Safety

#### 5.1 General

Catastrophic failure causing injuries are not common and no reference is known from overseas accelerator facilities. This is almost certainly due to the low pressures involved in vacuums and the size and nature of the scientific apparatus. There are of course many failures reported for industrial sized tanks and equipment.

The vacuum systems are essentially fail-safe from absolute vacuum to atmospheric pressure. The worst case has been a number of beam lines and measurement chambers developing leaks and thin windows that have ruptured. In each case there has been a gradual rise in pressure to atmospheric pressure.

However, a few rules do apply.

- All vacuum systems must have one or more vacuum gauges attached with the exception to some static chambers used for storing moisture sensitive items or radiation detectors.
- High vacuum gauges must be switched off before vacuum systems are vented to atmospheric pressure.
- Only nitrogen, argon or other inert gas to be used for venting. No flammable gases to be used such as hydrogen.
- Turbomolecular pumps must not be vented from full speed. See manufacturer's recommendations.
- All designs must be consistent or better than the best practises outlined in this document. Inferior equipment must be isolated and reported to the Leader of Accelerator Operations.

- Cryopumps must not be operated without a serviceable pop off valve.
- Cold traps must be warmed and blown dry at least every 6 months to minimise  $O_2$  concentrating (and for de-icing as necessary).

# 5.2 Interlocks

Nearly all vacuum systems on the accelerators are interlocked via a set of integrated relays in a vacuum gauge controller, to protect equipment. Generally, if a rise in pressure is detected above a particular set level then the interlock relays will activate to close corresponding gate and line valves. This is designed to minimise the number of vacuums that must be regenerated in a fault. The interlock system also protects accelerator tubes from exposure to moisture and in the event of a catastrophic failure of the accelerator tubes; the vacuum system will close the main accelerator gate valves to trap SF6 gas.

If an interlocked failed causing a gate valve to stay open in the event of a pressure rise then the next interlock should activate the next gate valve. If the next interlock fails it is possible that the two interlocks share the same vacuum gauge controller which is at fault. In that case the next interlock should activate. If no interlocks activated there would be no increased risk of injury to personnel but all vacuum systems will require regeneration or a restart after the interlocks were repaired and tested.

## 5.3 Fumes from exhausts

There are a number of oil vane pumps in use on the accelerators. In the past, the exhaust on the pumps has released oil mist into the atmosphere inside the buildings. Now, on nearly all oil vane pumps in continual service there are mist filters attached or the exhaust is plumbed into an extraction system that vents the exhaust fumes outside of the building.

#### 5.4 Earthing

Some vacuum gauge controllers are prone to high voltage transient surge damage. These sometimes cause unnecessary isolation of systems requiring resetting. It is very important that all vacuum gauge controllers are well earthed.

# 6. Serviceability

#### 6.1 Access

Most beam lines with the exception to some measurement chambers are static volumes of vacuum requiring little operator intervention other than during preventative maintenance. Vacuum pumping systems do require a higher level of intervention during maintenance and operation. With these points in mind the designer must ensure that all controls and monitoring devices are localised at the working face of the beam line or chamber.

Due to beam lines being so high above floor level, working on them usually requires some form of ladder or steps. When designing beam lines, be sure to have pump out ports facing the passageway beside the beam line. The same applies to vacuum chambers and other large volume vessels.

Measurement chamber will need to have the access port accessible from a location near the control side of the chamber. A platform is recommended to allow easy reach and eay viewing into the ports.

#### 6.2 Maintenance

A well designed vacuum system will require routine maintenance including:

- Changing faulty seals cleaning seals that are regularly opened
- Cleaning vacuum gauges especially cold cathode type high vacuum gauges
- General cleaning inside of measurement chambers and equipment that is; regularly opened and touched, whenever it looks dirty, when the base vacuum degrades or when the surfaces are exposed to the atmosphere for long periods at a time (weeks).
- Servicing of vacuum pumps the group of the most routine maintenance tasks includes; cryopump regeneration, turbo pump bearings, scroll pump tip seals and oil vane pump oil changes to name a few
- Actuator service sliding shafts that penetrate into the vacuum space

#### 6.3 Cleaning

Cleaning is by far the most essential part of general maintenance of a vacuum system. Poor cleanliness will lead to poor vacuums and possibly sample contamination. It is a general philosophy that cleaning is done with solvents, wiped over and/or scrubbed and thoroughly dried.

#### **Solvents**

Typically ethanol, methanol and acetone are used. These solvents remove most greases and oils that are encountered. What ever solvent is chosen the most crucial point to remember is that it must not leave any residue.

For surface finishing see section on 'Surface Finishes'.

Chemical polishing is not commonly used in the accelerator area due to the size of the baths that are needed for large parts, the problem of cleaning or neutralising residual chemicals and not having a suitable facility for handling the types of chemicals used. Although once used electro-polishing is no longer encourages for the same reasons. If suitable facilities were available both chemical and electro-polishing are real options for very good cleans.

A general clean will consist of a number of different levels of cleaning. A single clean with the same solvent will not remove some oils and greases therefore a progressive approached is used using a variety of solvents and wiping materials. The following is a guide only of some such techniques.

Using a dry gas or filtered compressed air, most loose dust can be blown away. This is sometimes the best starting point for cleaning. A final blow down is also useful to ensure complete removal of lint.

The first clean will take away coarse particulates or chemicals. Ethanol dampened tissues if good for this level of cleaning. The ethanol helps remove some oils and greases and mixes with water to reduce the evaporation time. It is also good for mopping up dirt and dust. Scour pads (or 1200 wet or dry paper) moistened with ethanol is good for removing stubborn dirt. This must be continued until better than 95% of dirt, dust, oil and grease is removed. For stubborn dirt it may be necessary to use a petroleum based solvent followed by an ethanol rinse and wipe. Wiping is key to cleaning. The light mechanical brushing dislodges most dirt.

The second clean starts with ethanol but finishes with acetone. Only lint free wipes are used such as Kimwipes. At the end of this level of cleaning the equipment must look ready for use in the vacuum. That doesn't mean it is. It will more than likely have residues remaining on the surface.

Final clean. It is recommended that methanol is used for the final clean or another low residue solvent. The final clean must use lint free wipes dampened with methanol and lightly rubbed over the entire surface. An alternative final clean is to use the solvent bath and suspend the equipment in the hot vapour for a few minutes. Care must be taken with the bath as the hot vapour will degrease skin very quickly.

If a shiny (healthy looking) clean is desired then a metal polish such as Brasso can be used. Whenever Brasso is used the residue must be cleaned off with ethanol. Care must be taken not to get metal polish pastes into gaps that cannot be thoroughly cleaned out. Brasso is an enemy of vacuum, it traps water.

#### 6.4 leak detection

In the accelerator area, a leak is characterised by a flow of gas molecules into a space that is below atmospheric pressure. The pressure of the gas entering the space will restrict the vacuum system from reaching its design pressure which is typically in the high vacuum range. As a benchmark, if a vacuum is  $1 \times 10^{-5}$  Pa or more then disregarding the effects from outgassing, there may be a leak. In system designed for ultrahigh vacuum the benchmark will be in the order of  $1 \times 10^{-7}$  Pa.

Leak detection is usually only necessary for new chambers or equipment. It can be done offline or after the part has been mounted into a beam line. The most common method of leak detection is a helium leak test. Helium atoms being so small will find their way through openings that are too small for most other molecules. Generally, the smaller the leak, the less helium that can make its way along a leak path. The leak detector is usually a mass spectrometer tuned to have highest sensitivity for helium. The detector is connected directly to the device being tested. Any helium that makes its way through the leak will eventually be measured in the mass spectrometer. Residual Gas Analysers (RGA), although not specifically leak detectors, can be used for just that. Working on the same principle, mass spectrometry, the RGA can detect helium as well as other gases.

In some cases leaks can be too large for a helium leak test, that is, the amount of gas in the device being tested is too high diluting the helium to an ineffective percentage. Also, if the pressure is too high the leak detector cannot be opened to the device as the pressure gradient will be too high. Leak detectors work best from the higher end of the low vacuum range.

Often there are multiple leaks in systems. It may mean repairing large leaks in order to find small leaks. Where a leak is too large for the helium leak detector a few other methods can be used to locate leaks.

- Solvents have a very low viscosity and vaporise very quickly in air. Wetting surfaces with small amounts of acetone or ethanol can show small changes on Pirani and Penning gauges.
- Isolation of seals can, by process of elimination, restrict the testing area allowing large leaks to be isolated. For example, if a chamber with many ports and devices attached has a leak. It may be prudent to close all valves leading away from the chamber except for the leak detector valve then sequentially blank off the ports one at a time (using a sound flange and new seal) and test for a leak.

# 7. Soldering wires vs mechanical joints

It is preferable that wires in a vacuum space are joined together with a mechanical clamping type joint. The clamp must allow for gas to escape. Where a direct joint is necessary then soldering is permitted. Standard lead/zinc with resin core is okay provided that ALL flux is removed with ethanol. This may require some soaking in solvent with gentle agitation. Solder without resin core

can be used on stainless steel with a zinc chloride based eutectic flux, used sparingly. Ensure all flux is removed especially between the wire strands.

Be aware that in Faraday cups the continual flexing of wires can work harden the wire causing it to break. These are typically soldered joints. If soldering, be sure not to melt the insulation. It is highly recommended to replace normal plastic insulation with loose fitting PTFE (Teflon) spaghetti.

# 8. Flanges and seals

## 8.1 Common vacuum seals used in the accelerator area

A variety of standard vacuum seals are used on all vessels depending on the degree of vacuum to be achieved and the type of fitting that will be used. The types are widely used in the vacuum industry. ANTARES uses the Dependex style seals and flanges. This was developed for use with HVEC accelerators over 50 years ago. Other types in use include Conflat, ISO, and KF are used as well.

Туре	Vacuum level	Flange type	Seal type
KF	Low to High	Clamp (typically)	Viton 'o' ring, Al, PTFE
Edwards	Low	Threaded retaining nut	Viton 'o' ring
Dependex	High	Ring retained by spring clip	Viton 'o' ring supported by a ring
LF or ISO	High	Ring or clamps	Viton 'o' ring supported by a ring or in a groove
Conflat	Ultra high	Ring fixed or rotating	Copper ring, Square section 'o' ring
Specials	Various	Various	Aluminium or indium wire

Table 1. Common types of vacuum flanges and seals. Each type is available in multiple sizes

# 8.2 KF – Klein Flange

A common flange and seal used for general low vacuum applications due its ease of assembly and vacuum performance. It is common to have KF flanges and seals within the high vacuum side of a system. Vacuum performance can be enhanced by the use of aluminium or PTFE seals which directly replace the Viton 'o' ring.

Many general and specialised vacuum equipment is fitted with KF fittings as the range of adaptors from KF to other major types is readily available.





Fig 3. Assembly drawing for a typical KF flanges seal and a photo of the flange, seal and clamp
# 8.3 Edwards

This is no longer a preferred type that is used in the accelerator area however some equipment still uses this type.

One end of the fitting is shaped to retain an 'o' ring while the other end has an angled sealing surface that mates and crushes the 'o' ring to make a seal. They are connected by a threaded nut which when tightened squeezes the two halves together. It is normally for low vacuum applications.

# 8.4 Dependex

This was designed for use on HVEC Van der Graaff accelerators and beam lines. It was introduced to ANSTO in the early 1960's when the 3MV Van de Graaff operated in B22. Here, 1", 2" and 4" Dependex was used. When ANSTO purchased the FN Tandem nearly all fittings used were 4" Dependex (100 mm nominal). It is suitable for high vacuum use and often mated with Conflat flanges in systems that operate near ultra high vacuum.

The Dependex seal consists of a supported 'o' ring sealing between two opposing  $5^{\circ}$  metal faces. The 'o' ring is supported around its inner diameter by a ring to ensure the 'o' ring aligns with the centre of the 2 faces. The inner ring also sets the maximum crush on the 'o' ring that is the ring fits into a step on both halves of the Dependex seal keeping them separated at a fixed distance. Another ring which fits on the outer diameter of the 'o' ring does little more than keep the 'o' ring clean. The rings are often called *egg rings* due to their resemblance.

The Dependex flanges are held in place on a tube, or other fitting that is designed to accept Dependex, by a large circlip that fits into an outer groove a few mm from the end of the sealing face.

The flanges are normally drilled such that the holes alternate between being tapped or clearance. 1" and 2" Dependex use <sup>1</sup>/<sub>4</sub>" UNC, cap (socket or Allen) head screws and 4" (100 mm) Dependex use 5/16" UNC, cap (socket or Allen) head screws. Only use stainless steel screws (of one length) and washers. Discard plated steel screws as they are identified during maintenance. Washers are recommended as most flanges are aluminium alloy.

To complete the Dependex assembly, the flanges must be placed over the tube ends and then retained in place with a circlip. Secondly, the 'o' ring is stretched onto an inner ring and then an outer ring is placed around the assembly. The two Dependex ends are brought close together then the seal assembly is placed between them. The inner ring is aligned with the step on one side then the other tube is aligned and than pushed onto the ring. At this point it is important to ensure the 'o' ring assembly stays in place until the bolts are tightened. Screws should be finger-tightened and the flange faces must be parallel. Tighten screws in an opposite pattern. Some old flanges will have worn threads so screws may only be turned with the aid of an Allen key.

#### Problems

There are a number of things that can go wrong with assembling Dependex fittings.

- There have been cases where inner egg rings have been made too wide so that when fitted between the tubes the 'o' ring is prevented from crushing adequately to form a vacuum seal.
- Another problem with the inner rings is where some Dependex seals have been designed with too shallow steps so that a standard inner ring becomes too wide. In this case special inner rings have been made which are narrower. Personnel servicing non-standard joints must take care to reuse the right sized inner ring. A label should be placed on all non-standard joints identifying them.

- Some steps for inner rings have also been found to be out of tolerance preventing standard size inner rings from locating in the step. In this case some operators have cut a small section from the inner rings so that they can be squeezed together. Cutting inner rings like this is only recommended to solve this type of problem.
- Some flanges have been design for special applications with slightly different PCD's. These have unfortunately bee mixed up with standard stocks. As personnel who identify them (sometimes after a frustrating encounter) should put them aside and label them accordingly.
- Old flanges that have been over-tightened may have stretched threads. They are identifiable by the difficulty in starting screws. Either re-tap the threads or discard the flange. There have been case were tight threads have mislead personnel into thinking they have tightened the Dependex joint only to find leaks.
- Circlips can stretch especially those in over or under sized slots. They are easily identified by there distorted shape. Only use circlips that are flat when not under tension. A discerning technician should discard rusty circlips that cannot be successfully cleaned.
- Care must be taken to make sure the two faces of the Dependex joint are parallel before the joint is closed and bolted together. Failing to do this may cause a leak in the joint. It is acceptable if a bellows is used between sections that have misalignments.
- Twisted 'o' rings can cause leaks although small. There have been cases where a twist has caused the 'o' ring to bulge towards the outer ring making a less than adequate seal.

# 8.5 Special Flanges

These consist of flanges that have been designed to fit application where "off-the-shelf" flanges and seals cannot be used. They are typically based on common designs but modified to suit the application.

# Example

The Alphatross ion source uses two stainless steel flanges to hold the heater reservoir in place. Here the vacuum sealing surface is a flat surface about 8mm wide on each flange. The fixed flange is threaded in an imperial thread and the reservoir flange is open hole. The gasket used is aluminium wire which has been fused together to make a ring. The joint is similar in size to the wire. The wire is supported in place using about 3 to 4 strips of aluminium foil looped around the wire and sticky taped to the edges of the flange. The flange is tightened in a circular pattern to ensure maximum crush on the wire.

# Problems

- The wire is essentially unsupported so vacuum must not be applied until the wire is completely crushed.
- Bulbous joints in the aluminium wire dent the sealing faces on the flanges as they are tightened so they must not be used.

# 8.6 ISO or LF (Large Flange)

This is a European range of fittings designed for high vacuums and to suit most tube sizes used in the vacuum industry. There are two types of design which meet most light and heavy duty applications.

ISO-K flanges are a robust joint which are suitable for supporting larger and heavier items such as vacuum pumps and large gate valves. They are characterised by the outer clamping system which holds the two halves of the joint together.

ISO-F flanges are more of a traditional bolted flange type joint like Dependex. ISO-F and ISO-K can be joined together using a half clamp which fits onto a standard ISO-F flange. The half clamps then attach to the ISO-K flange.

Available in sizes from 63 mm nominal bore to 500 mm NB.



Fig 4. ISO family of Flanges

# 8.7 Conflat CF

Conflat is designed as an all metal vacuum seal for ultra high vacuum applications. A copper gasket is sandwiched between two thick stainless steel flanges that have a knife edge machined into the sealing face. As the flanges are tightened together the knife edges "bite" into the copper to produce an all metal seal with very low outgassing.

The term "ConFlat" is a registered trademark of Varian, Inc., so "CF" is commonly used by other flange manufacturers. Conflat sealed systems can reach vacuums as high as  $1 \times 10^{-11}$  Pa.

The copper gasket locates into a partially recessed in a groove in each flange to provide alignment prior to tightening. It also stops the gasket from moving during bake out. Conflat can be baked up to  $450^{\circ}$ C.

Most Conflat flanges have imperial sized bolts. The original style of bolt had a multi-face head that a ring spanner would fit over. This is to allow more purchase on the head from many angles. It is not uncommon now to find hexagon and socket head screws in use. Be aware that the steel must be a high tensile grade to ensure optimum force can be used to crush the gasket. It is essential that the flange faces are parallel during and on final tightening. A torque wrench may be used to facilitate uniform tightening. It is highly recommended that a molybdenum grease or graphite loaded grease is used on the threads.

Conflat flanges are available in sizes from 10 mm nominal bore to 250 mm NB. Sometimes nominal bore is written DN in front of the size, eg DN63.

Square section Viton 'o' rings may be used with Conflat flanges however this compromises the ultra high vacuum design and ultra high vacuums may not be reached.

# Problems

- Copper gasket not aligned in groove and edge is partially clamped on the bolt hole face of the flange. This lead to partial seal and most likely, a leak. Most Conflat flanges have two small grooves a little more than a millimetre wide machined into the bolt face. These are for locating special clips that hold the gasket in place while the flanges are brought into place. Just prior to tightening they clips are removed. They are sometimes mistakenly called pressure relieving grooves.
- Copper gaskets must only be used once. Unless the copper hasn't been fully compressed after the first use there will not be enough material for the knife edge to reform a seal.
- Due to the forces required for a knife edge to indent a copper gasket the flanges are made of stainless steel and quite thick. Large flanges are quite heavy and care must be taken not to drop them.
- Where Conflat flange bolts have been used more than once the threads in the flange and screws may have stretched. In this case, it is recommended that the same screws are reused in the stretched threads (with a little molybdenum grease) as they will have the same profile. Sometimes using new screws in a stretched thread will cause binding. Re-tap the flange if necessary.
- Stretched threads are possible where nuts and bolts are used through open holes so ensure the nuts go onto the bolt the same way they were removed. Alternatively replace the nuts and bolts outright.
- For ultra high vacuum ensure the gaskets are handled with gloves. Copper reacts well with the sweat in a fingerprint leading to a long outgassing process.

# 9. Bellows

Bellows consist of two styles, edge welded for ease of extension or compression and corrugated style which is has minimal movement. Each style is made from a thin stainless steel that has been electron beam welded. The ends are usually finished in a standard flange to suit the application.

# 9.1 Corrugated style

These are typically used for two purposes. Firstly, to allow sections of beam lines or equipment to be compressed to allow easy removal of sections without dragging the sealing surfaces against each other. Secondly, as bellows can distort and still maintain their structural integrity they are ideal to

join sections of beam line or equipment that have a deliberate mis-alignment. They can be purchased with almost any style of flange including specials. See example pictures.



Large bellows. ~300mm diameter. Stainless steel, wall thickness ~ 0.8 mm. This bellows is used to correct alignment offsets in the magnet box

Medium bellows. ~ 100 mm diameter. Stainless steel. wall thickness <0.5 mm. Used to allow sections of the beam line to be compressed to facilitate beam line component removal.



Small bellows. Electron beam edge welded stainless steel. Material thickness <0.25 mm. Used to isolate vibrations in the beam line.

Fig 5. Examples of bellows

#### 9.2 **Edge Welded Bellows**

These bellows are made up from very thin stainless steel discs stacked together and welded along their edges. The advantage of the design is the concertina style bellows that is very flexible allowing designs that have long extensions and short compressions. They have two main applications. Firstly, applications that requires a device to extend or contract into the vacuum space where an elastomeric seal on a sliding shaft is not adequate. Such as a sample manipulator or Faraday cup. Secondly, where a device such as a detector or sample positioner is sensitive to vibration and must be decoupled from the vibration source.

# 10. Valves

Along the axis of the beam lines gate valves are used to enable the isolation of sections for maintenance and also to minimise loss of vacuum in the whole accelerator if one section develops a leak. Most gate valves are electro-pneumatically operated and many are interlocked to local high vacuum gauges. The few that are manually operated are for maintenance isolations.

On ANTARES the electro-pneumatically operated valves are interlocked to close when the pressure, as measured near the gate valve, rises above 5 x  $10^{-5}$  Pa. This figure can vary depending on the location of the valve. See the Accelerator WIKI for values around the ANTARES. Once an interlock has tripped the switch that operates the gate valve is bypassed. To reset the valve:

- The vacuum must be within the high vacuum range as read on the corresponding vacuum gauge controller with a pressure better than the trip point.
- Once this level is reached the bypass button is held down momentarily.
- At this point the pressure will rise then fall as the gas load is pumped away.
- When the system pressure is less than the trip level the bypass switch can be let go.
- An indicator light near the switch will show its status.

#### Notes:

- Operators and technicians must be aware that gate valves must be orientated so that the gate always seals towards the vacuum pump. That is, they seal best in one direction only. Failure to orientate the gate the correct way may cause a vacuum leak when atmospheric pressure forces the gate off its seal.
- In some cases gate valves may remain sealed after they have been switched to open. This can be due to the withholding (sometimes called back pressure) pressure not being adequate enough to lift the gate off the seal.
- Gate valves, while mostly metal can become hot if a gate is closed in front of an ion beam. If left for long periods with high current beams the elastomer seals may warm up and begin to outgas.
- For roughing ports typically 25KF bellows sealed valves are used. On STAR these same valves are used to isolate vacuum gauges for cleaning.
- Where fine flow control is required for either roughing out or venting, metering valves are used. These are basically multi-turn needle valves for fine adjustment.
- Valves do require maintenance to keep the seals clean and in working order. After long periods of time the 'o' ring seals will deform and often will not recover their original shape.

There have been attempts to build fast acting gate valves for the purpose of isolating the accelerator tank's load of  $SF_6$  in the event of a breach in a tube. While it seemed like a good idea the force of the valve closing at high speed could damage the gate seal causing the valve to leak. Designers must use care to ensure that a standard high differential pressure rated gate valve isn't adequate before launching into these expense type valves.

# **11. External Interfaces**

# **11.1 Electrical Feedthroughs**

a) Low Voltage, Low Current

There are few applications where voltages greater than 24Vac are used within the vacuum space. For these voltages at a few amps coaxial feedthroughs such as BNC, SHV, MHV, etc, are often used in place of more expensive dedicated commercial types. In one application 200Vdc is used for faraday Cup suppressors but current is only a few micro-amps at most.

Notes:

• Do not use multi-pin signal feedthroughs to carry voltages unless the pins used for voltage can be a few unused pins away from used signal pins.

- Feedthroughs can be purchased is almost any flange style but are usually restricted to small size flanges.
- Insulation must be used on all wires carrying voltage. It is highly recommended to use single or stranded wires (preferably nickel coated) with loose fitting PTFE (Teflon) spaghetti.

# b) Signal

Signal feedthroughs other than coaxial such as BNC, SMC, Microdot, etc are from one to multiple pins. It is recommended that connections to multi-pin feedthroughs are by the use of slide on pins. Many are either sliver of gold plated for good electrical contact. Avoid soldering unless care is used to remove all traces of flux and no pin-holes in the solder have been created

On coaxial feedthroughs the coaxial connector will be either both sides of the feedthrough and hermetically sealed or with the coaxial connector on the atmospheric side and a plain pin on the vacuum side, also hermetically sealed. For either type, where coaxial cable must continue into the vacuum space it is recommended to remove the outer plastic insulation and replace it with loose fitting PTFE spaghetti. This is also recommended for all single or stranded wires carrying signals.

# c) High Voltage, High Current

There is a wide range of high voltage and high current feedthroughs available in most flange types. High voltage feedthroughs usually have a larger insulator in ceramic and sometimes glass. These must be kept clean on the atmospheric side. Long term breakdown on high voltage feedthroughs can damage the insulator causing vacuum leaks.

For high current applications such as heaters, dedicated high current feedthroughs must be used. These are characterised by larger cross section wires to carry the higher currents. Like HV feedthroughs they must be kept clean. It is recommended to clean the insulators on signal feedthroughs periodically to remove any accumulation of dust.

# d) Power

There are few feedthroughs designed for powering devices at mains voltages. It is highly recommended that experiments are designed not to have mains voltages in vacuum vessels or equipment due to safety concerns.

# **11.2** Mechanical Feedthroughs

# a) Rotary

Rotary feedthroughs allow the transition of rotary movement into the vacuum space to, for example, operate shutters or position samples or detectors. They are widely available in many of the common flange styles. There are three common types both of which are very good in high vacuums.

The first type is a continuous shaft between the atmospheric side and vacuum side using rubber vacuum seals in the form of either supported 'o' rings, square or odd shaped rings (like common shaft seals), and flat rubber sheet seals. These are best for general rotary actuation rather than continually rotating. Okay in high vacuum but care must be taken to keep the seals clean and periodically replaced or whenever wear is noticed.

The second type is a ferro-fluidic type which also uses a continuous shaft between the atmospheric side and vacuum but the seal is made by encapsulating a dense fluid around the shaft. These types usually have bearings to keep the shaft centred and free moving. They are best for high revolutions in high vacuum applications.

The third type is magnetically coupled rotary feedthroughs. These are expensive. They provide a seal by magnetically coupling the shaft on the atmospheric side to the shaft on the vacuum side across a continuous sealing plate. If either shaft is removed the vacuum is not compromised. These are excellent for ultrahigh vacuums and lower. They are for moderate speed revolving applications and general actuations.

# **b**) Sliding

Usually these feedthroughs are a round shaft that can slide in and out of the vacuum as well as rotate. Sliding feedthroughs are used for positioning devices or where a linear actuation is necessary. The length can vary from a few millimetres to hundreds. In either case there is a shaft which penetrates from the atmospheric side to the vacuum side. The type of seals used varies like in the rotary seal. Sometimes dual seals are used to minimise leakage especially in high actuation rate applications. In some applications the void between the two seals is pumped increasing the reliance of the seal. These types are good in high vacuums. Notes:

- Care must be taken to ensure the shaft is lubricated with a suitable vacuum oil or grease. It is imperative that the oil or grease on the shaft is changed regularly to minimise dust build.
- There are bellows sealed sliding seals available. These types do not rotate and can only move in and out a few tens of centimetres at most. Very good in high to ultrahigh vacuums.
- Some sliding seals are able to move radially which will affect alignment. Where alignment is crucial a linear bearing type is recommended.

# c) Wobbler

A wobbler feedthrough allows users to use a shaft as a lever or pick up. They are designed around a flexible rubber or bellows seal they allows a greater range of circular movement.

# **11.3 Manipulators**

These are a class of feedthroughs design for high to extremely high precision positioning of samples, detectors or other instruments. They can be purchased having all or either X, Y, Z and rotary movement. The X and Y axes can have various travel lengths however they are typically less than 100 mm each. The Z axis (vertical) can have greater lengths but is limited as with the X and Y axes by the physical limitations of the vacuum bellows.

# 11.4 Actuators

'Actuators', combine sliding and rotary feedthroughs with pneumatic, electro-pneumatic or mechanical actuators to do the work.

In the accelerator area, actuators can be found on Faraday Cups to insert the cup into the beam line on a bellows sealed sliding feedthrough and inside of the ANTARES pressure vessel to move the stripper tube in and out of the beam axis.

# 11.5 Load locks

Load locks are not common in the accelerator area but are ideal for moving samples into a high vacuum space without significant loss of vacuum pressure. A sample or device is mounted onto the end of the load lock shaft. It is inserted into a tube connected to a chamber but isolated from the chamber vacuum by an in-line gate valve. The tube is sealed and then evacuated to a vacuum pressure similar to the chamber. Once the pressures equalise the gate valve is opened and the shaft pushed into the vacuum space. From this point the sample or device can be removed from the shaft by other remote means.

# **12. Internal Devices**

There are many devices that are commonly used inside of vacuum systems. Not all are represented in this section however similar reasons are use to assess new and unusually devices for use in vacuum.

# 12.1 Lighting

Lighting is necessary in most sample measurement chambers where the operator must be able to see the sample in order to position it in front of the ion beam. In most cases a standard bare 12Vac halogen bulb is used with good results. Care must be taken to ensure the bulb can be dimmed as when running at maximum power the heat load can only be dissipated through the connecting wires. That is, there is little gas inside a vacuum to conduct head away from the bulb. Bright white LED's are also used with mixed success. They have less radial light output as compared with a bare halogen bulb. If the LED is setup correctly the light can be focussed towards the necessary target.

Preferably, wires must be stranded or solid wire with a loose Teflon sleeve but normal plastic coated hook up wire works in high vacuums. In some cases where the wires as not likely to come into contact with other components then they are used uninsulated. All wiring must be restrained so that it cannot move into the path of the ion beam. As a rule-of-thumb wires should be at least 20 mm from the beam.

A standard double BNC feedthrough can be used, that is, only the centre core connections are used. There are many off-the-shelf electrical feedthroughs available that will be more suited for this specific use.

# 12.2 Heating

Providing power to a heating stage on a sample (target) stick requires a high current feedthrough. If a feedthrough isn't provided from the supplier of the heating stage then many types can be found from larger vacuum suppliers. Ensure the feedthrough selected is designed to carry the maximum current that can be supplied and not the maximum current likely to be used. Like in the lighting section above, care must be taken to ensure wires are insulated and away from the beam path.

Heating stages rely on heat dissipation through the support for the stage which is usually made from stainless steel, a relatively poor conductor. When opening devices for handling be sure they are cool to touch first.

# 12.3 Cooling

Cooling stages come in 2 distinctly different types. The first is the Peltier cooled stage which is all electric. Powering the stage can be managed as for lighting and heating. The second type is a liquid nitrogen cooled system. It uses an insulated tube to carry LN2 through a feedthrough into the vacuum space. A typical design is where LN2 is pumped through a coil attached to a heavy copper braid. The braid is then connected to the cooling stage where heat is conducted away from the mounting stage. Thermal isolation of the cooling stage from the holder can be Teflon or sapphire standoffs or similar materials.

These cooling devices must only be switched on when the vacuum is at the working level otherwise condensation and possibly ice can form on their surface becoming a large outgassing problem. Likewise ensure the stage is close to room temperature before opening up the vacuum system to atmospheric pressure. Always vent with a dry gas.

# **13. Pumping**

# 13.1 General

It is not the intention of this document to describe the fundamental operation of particular vacuum pumps. Adequate information is available from manufacturer's catalogues. The following is information on what type of systems are used in the accelerator area and the reasons why.

Vacuum pumps don't suck. They act essentially as one way valves in a system where gas is flowing towards them. This is discussed in Chapter 3 of this document. There effectiveness is there ability to stop recoiling gas molecules returning into the vacuum space such as a beam line or vacuum chamber.

When considering what type of pump to use for a vacuum system the following points must be considered.

- What is the application?
- Will it have a relatively high gas load, that is;
- Will it be pumping a static gas load or changing gas load?
- Will there be a high outgassing rate?
- Are there known hidden/trapped voids to be pumped?
- It is a clean or dirty device? Is it contaminated with grease and oil?
- What pumping speed will be needed to maintain a high vacuum?
- What is the volume of the system? What is the conductance of the system? Will a small pump be just as effective as a large pump?
- What gases will be pumped?
- What type of pumps and how many are being used in the vicinity?
- Will the pumping speed need to be regulated such as for pumping near foils or delicate samples, detectors, etc?
- Cycling time whether it will be required to regularly pump from low vacuums
- Will the pumps operate reliably in their chosen application?
- Are they compatible with other pumps used on the accelerators? Are they cost effective?

Another consideration is vibration. Pump vibration can be significant leading to micrphonic interference in detectors and vibration of samples being measured. Vibration can be reduced by selecting pumps with less inherent vibration like turbomolecular pumps or the more fickle ion pumps. If a pump is a source of vibration it can be decoupled from the detector or measurement chamber by the use of a flexible bellows. An fine edge welded bellows is recommended.

# **13.2 High Vacuum Pumps**

See section 4.5, Pump Down characteristics for cryogenic and turbomolecular vacuum pumps.

# <u>Cryopumps</u>

For ANTARES the main beam line pumps in use are cryopumps. The types of pumps trap (or immobilise) gas molecules. They are used because they have a long lifetime between major

services, can be regenerated without removing them from service, have a high pumping speed for most gases and can maintain high vacuums for long periods in a static vacuum application.

#### Turbomolecular pumps

Where a vacuum system is cycled such as at a measurement chamber, then turbo pumps are used. Turbo pumps are transfer type pumps meaning, gas is transferred from the vacuum space into the atmosphere. A turbo pump can routinely recover a vacuum from low vacuum after a measurement chamber has been roughed out without needing regeneration as for a cryopump.

Turbo pumps are also more effective at pumping lighter molecules such as helium and hydrogen as compared with cryopumps. Using turbo pumps near systems that use these gases will reduce the effect of loading on cryopumps.

Most new turbo pumps can be serviced in-house. Some models purchased require special balancing of the bearings after they had been changed. In this case they are sent back to the manufacturer at great cost. It is important that models chosen can be maintained in-house.

#### Other high vacuum pumps

There have been few applications where titanium sublimation or ion pumps have been used in the accelerator area. Most have been stand alone UHV systems or cryostats for specialised applications. The practicality of cleaning and regenerating these gas trapping type vacuum pumps on systems requiring relatively frequent cycling is prohibitive.

Getter materials once used widely in electronics in tubes are becoming more used within larger static volumes such as beam lines and associated equipment. They can be coated onto surfaces to assist in maintaining uniform high vacuums in spaces that have poor pumping speeds.

# **13.3** Low Vacuum, Roughing or backing pumps

#### Oil free pumps

These pumps are categorised by either being an oil type pump or dry (oil free) pump. Oil free vacuum systems are of choice for accelerator systems now that the technology has been proven to be reliable and cost effective. The majority of roughing and backing pumps now in use are scroll pumps as well. Piston pumps have been trialled with little success due to the short life of the seals. Some applications have used diaphragm pumps and multistage roots pumps but scroll pumps remain the most cost effective and easiest to maintain option. The Edwards type scroll was chosen over other brands as this type of scroll pump is designed with the bearings mounted outside of the vacuum which in other brands often caused the bearings to fail from lubricant loss.

#### Oil vane pumps

Low Vacuum, Roughing or backing pumps are all the same type of pump. Oil vane type pumps were widely used in the accelerator area until 2006 when a systematic change began on the STAR accelerator to replace oil vane pumps with oil free scroll pumps. Some oil vane pumps remain in use where some back streaming oil vapour is not considered a problem. If an oil vane pump is to be used for roughing out purposes, it is high recommended that a liquid nitrogen cold trap is used to trap vapours. It is the ultimate aim to have oil free pumping on all accelerators in the future.

# **14. Monitoring**

# 14.1 Vacuum Gauges

The standard type of high vacuum gauge in use is the cold cathode penning type gauge.

- On ANTARES the Pfeiffer brand is in use in two versions; the standard cold cathode type and full range type which has an integrated Pirani gauge.
- On STAR the most widely used brand is Leybold.

Generally, vacuums are measured as an indication only for process controlling and establishing basic quality to ensure pressures are within levels that are accepted for "normal" operation. This varies depending on location and use of the vacuum system for example a measurement chamber is cycled often and affects surrounding vacuum systems. It will more than likely be a much high pressure than surrounding static systems.

In most cases, absolute vacuums are not required to be measured. However on some AMS beam lines where thin windows are used it is important to have accurate measurement of pressures when cycling pressures in the gas detectors. If accuracy is required it must be remembered that different gases ionise at different pressures and so pressures readings from the same volume will be different depending on the dominant background gas. Most gauges have characteristics related to Nitrogen (air).

# **14.2 Gauge Controllers**

On ANTARES the most widely used gauge controller is the Pfeiffer TPG300. This is an aging model and is being replaced in some key areas with the Pfeiffer Maxi-gauge which allows 6 heads to be connected at once.

The TPG300 has inputs for up to 4 heads whether Penning or Pirani. The limitation with the TPG300 is the electrical robustness when operated near the accelerator high voltage generator. Many failures have been attributed to high voltage transient surges. The TPG300 is now technologically aged and will ultimately be replaced outright. Various other models of TPG's are in use on ANTARES especially where single gauges are required. It is the TPG300 controllers on ANTARES that provides the interlocking capabilities.

The MAXI gauge is still being trialled as a replacement to the TPG300. A unit under test for many years at the HE end of the accelerator has already failed during an accelerator sparking event. Its real place may be away from the accelerator where the 6 heads allow consolidation of a whole beam line's vacuum monitoring system.

On STAR the common high vacuum gauge is the Leybold cold cathode type. It has been integrated into the STAR control system via cat 5 cable. They operate using a similar principle of measuring current flow across the plasma of the ionising gas. These gauges have been far more unreliable than the Pfeiffer equivalent. Unfortunately they are not interchangeable.

If venting with gas other than air or nitrogen be aware that the response on the Pirani gauges may show lower pressure than actual pressure.

# 14.3 Bourdon Gauges

Bourdon gauges are normally used for indicating that pressure is below atmospheric. They are not used on accelerator systems as they have poor accuracy in the range of interest i.e., below  $1 \times 10^{-3}$  Pa.

# Chapter 3



# **Overview of Basic Vacuum Technology**

The ANSTO accelerators consist of 140 metres of beam lines under vacuum. Supporting these beam lines are 60 individual high vacuum pumping stations. There are over 70 isolation zones along the beam lines to allow isolation of a zone without interrupting the adjacent zones. This facilitates isolations to repair leaking sections, routine maintenance and cycling of pressure in zones to change samples, etc.

The following information are the general tools used by technicians and engineers to design reliable and high performance vacuum systems for the accelerator facilities.

# 1. Vacuum Overview

# 1.1 What is vacuum?

A vacuum is defined as a diluted gas, or the corresponding state at which its pressure or density is lower than that of the ambient surrounding atmosphere.

Due to the Earth's gravity, atmospheric pressure varies with altitude. As a standard atmospheric pressure at sea level  $p_o = 101.325$  kPa. A Pascal is unit of vacuum relating to a force of newtons per metre squared.

An absolute vacuum of 0 kPa is not practically achievable so in real terms we can only approach zero for example on the ANSTO accelerators we have "operating" vacuums better than  $1 \times 10^{-4}$  Pa. In outer space the vacuum is less than  $10^{-12}$  Pa but not 0 Pa as there are some molecules of gas present which exert a very small partial pressure.

The pressure exerted from a vacuum lies between 0 to 100 kPa (see table below). In an ideal pumped vacuum system the pressure will continue to decease approaching 0 kPa. In accelerator systems the working ranges are medium to ultra high vacuums. In some special cases ion sources may operate in the low vacuum range but generally vacuum systems for the majority of accelerator systems are in the high vacuum range with some vacuum end stations close to ultra high vacuum.

# 1.2 Units used

The SI unit  $Pa = N/m^2$  or kg·m<sup>-1</sup>·s<sup>-2</sup>, will be used where any vacuum quantity is referenced. In the accelerator area the vacuum units used are in Pascals (Pa) and although the vacuum pressures are below atmospheric pressure "negative" pressures are not represented as -kPa but of a magnitude which approaches absolute zero using scientific notation, e.g. 1 x 10<sup>-6</sup> Pa.

#### **1.3** Vacuum categories

Vacuums used in accelerator applications vary in magnitude from low vacuum to ultra high vacuum. These categories describe the pressure ranges which in the vacuum world become references for vacuum quality in a given system. Low vacuum is managed differently from high and ultra high vacuums due to the physical limits of the system designs. Generally, a vacuum system must be designed with the desired ultimate vacuum in mind to ensure the most appropriate materials, seals, pumps and vacuum management are used.

The table below shows each of the ranges based on the European system. This is the closest to that used for the ANSTO accelerators. The shaded section represents the working ranges used throughout the accelerator for example, ion sources may have low to high vacuums, beam lines may have very high vacuums and some endstation vacuum chambers may use high to very high vacuums. This document describes the design factors and particular management controls for achieving vacuums in the various ranges.

Range	Pressure (Pa)
Atmospheric pressure	1.013 x 10 <sup>5</sup>
Low vacuum	$1 \times 10^5$ to $3 \times 10^3$
Medium vacuum	$3 \times 10^3$ to $1 \times 10^{-1}$
High vacuum	$1 \times 10^{-1}$ to 1 x $10^{-4}$
Very high vacuum	$1 \ge 10^{-4}$ to $1 \ge 10^{-7}$
Ultra high vacuum (UHV)	$1 \ge 10^{-7}$ to $1 \ge 10^{-10}$
Extremely high vacuum	< 1 x 10 <sup>-10</sup>
Outer Space	$< 1 \times 10^{-12}$
Perfect vacuum	0 Pa

Table 2. Reference pressure ranges (European scale).

A. Berman, Vacuum Engineering Calculations, Formulas, and Solved Exercises, Academic Press, Inc. (1992).

In the accelerator area:

Low Vacuum = atmospheric pressure to medium vacuum

High Vacuum = high vacuum to very high vacuum

Ultra high vacuum = all those equal to and higher than ultra high vacuum

# 2. Pressure Fundamentals

# 2.1 General

To understand the reason why vacuum systems must be carefully engineered to achieve high vacuums consideration must be given to how gases act in a closed chamber.

Any gas enclosed within a volume (a chamber or pipe work) is uniformly distributed or moving towards uniform distribution as in the case of accelerator tubes connected to stripper gas enclosures. The individual gas particles are constantly moving back and forth at high-speed within the volume; upon striking the vessel wall, they exert a force F on surface A due to pulse transmission. The pressure p that is exerted on the wall is defined as:

$$p = F/A$$

If the gas is made up of different types of gases, each of these gases will exert a pressure that corresponds to its concentration; this is called <u>partial pressure</u>. The sum of all partial pressures equals the total pressure. Air is a good example of this: In addition to its main constituents of nitrogen, oxygen and water vapour, air also contains many trace gases, the sum of which equal  $\sim$ 101.3 kPa, total pressure (atmospheric pressure). See table below.

In an accelerator system it would not be uncommon to have various carbon based compounds, sulphur hexafluoride and specific stripper gases present in trace amounts.

Gas	% by volume	Partial Pressure (Pa)		
Ar	0.92	934		
CO <sub>2</sub>	3.26 x 10 <sup>-2</sup>	33		
H <sub>2</sub>	4.9 x 10 <sup>-5</sup>	4.94 x 10-1		
He	5.16 x 10 <sup>-4</sup>	5.23 x 10-1		
Kr	1.1 x 10 <sup>-4</sup>	1.15 x 10-1		
$N_2$	77.17	78180		
Ne	1.8 x 10 <sup>-3</sup>	1.82		
O <sub>2</sub>	20.7	20970		
Xe	8.59 x 10 <sup>-6</sup>	8.7 x 10-3		
H <sub>2</sub> O	1.18	1200		
Other	remainder	remainder		
Total	100 %	101325 Pa		
Notes: H <sub>2</sub> O may vary depending on environment				
"Other" may include: $CH_4$ , $O_3$ , $N_2O$				

Table 3. Partial Pressures in AirThe Vacuum Technology Book – Pfeiffer Vacuum September 2008

# 2.2 Ideal (General) gas equation

1 mole of any gas at STP occupies 22.414 litres. At a temperature 273.15 K (0 °C) and a pressure of 101,325 Pa (standard pressure) 1 mole of any gas contains 6.02 x  $10^{23}$  particles. This is referred to

as Avogadro's number. The mass of the gas thus enclosed is its molecular weight in grams. The ideal gas equation describes the state of a gas as a function of pressure, temperature and volume.

$$pV = nRT$$

Example:

For a chamber, Ø300 mm x 500 mm long, a volume of 0.035 m<sup>3</sup>, calculate the mass of gas in the chamber at a vacuum of 1 x  $10^{-6}$  Pa.

pV = nRT

n = pV/RT  $n = 1 \times 10^{-6} \times 0.035 / 8.31451 \times 293 = 1.43 \times 10^{-11} \text{ moles}$ Mass of remaining volume = moles x mass = 1.43 x 10<sup>-11</sup> x 29 (air) = 0.415 ng No. of molecules = moles x Avogadro's Number = 1.43 x 10<sup>-11</sup> x 6.02 x 10<sup>23</sup> = 8.6 x 10<sup>12</sup> Per cm<sup>3</sup> = 8.6 x 10<sup>12</sup> / 35000 cm<sup>3</sup> = 246 x 10<sup>6</sup> molecules per cm<sup>3</sup>

Where:

 $p = \text{pressure (Pa)}, V = \text{volume (m}^3), n = moles, R = \text{general gas constant} = 8.314510 \text{ kJ/(kmol K)}, T = \text{thermodynamic temperature (K)}, Avogadro's number = 6.02 x 10^{23}$ 

# **3.** Flow Fundamentals

#### **3.1** Mean free path $(\lambda)$

The mean free path is the mean path length that a molecule traverses between two successive impacts with other molecules. High vacuums provide a longer mean free path than low vacuums. In accelerator systems high vacuums are crucial as ions that collide with gas molecules in beam lines loose energy and are not transported to the point of measurement. In the accelerator tubes high mean free paths are crucial to ensure low energy loss in the ion beam and optimised ion beam transmission.

Gas (at 0°C and 1 atm)	λ (nm)
Air	68
Ar	62.6
Cl <sub>2</sub>	27.4
CO	58.6
CO <sub>2</sub>	39
$H_2$	110.6
Не	173.6
Kr	36
$N_2$	58.8
Ne	124
02	63.3
Xe	26

Table 4. Mean free Paths for various gases

Hirschfelder, Curtiss and Bird (1954) Molecular Theory of Gases and Liquids, Wiley, New York

Pressure (Pa)	Molecules / cm <sup>3</sup>	Molecules / m <sup>3</sup>	Mean free path
101325	$2.7 \times 10^{19}$	$2.7 \times 10^{25}$	68 nm
	<b>—</b> 11 <b>—</b> 31	1	

Table 5. Other characteristics of Air

#### 3.2 Mean Velocity (u) m/s

The residency time of various gases in a system (or the time taken to remove various gases) relates to their individual mean velocity. The following table shows mean velocities for some gases. It can be seen that very light gases travel significant faster than heavy gases. It should be noted that Hydrogen and Helium do not pump efficiently in turbo pumps or cryopumps but heavy gases generally do.

Gas	Molar Mass kg/mol	Mean Velocity m/s	Mach Number
Air	0.028966	463	1.4
Ar	0.039948	394	1.2
Cl <sub>2</sub>	0.0709	296	
СО	0.028011	471	
CO <sub>2</sub>	0.04401	376	1.1
$H_2$	0.002016	1762	5.3
He	0.00402	1246	3.7
Kr	0.0838	272	
$N_2$	0.02801	471	1.4
Ne	0.020179	555	
O <sub>2</sub>	0.03199	441	
Xe	0.1313	217	
H <sub>2</sub> O	0.01802	587	1.8

Table 6. Mean Velocity for gases at 20°C

The Vacuum Technology Book – Pfeiffer Vacuum September 2008

$$u = \sqrt{\frac{8.R.T}{\pi.M}}$$

Where: *M* = Molar mass (kg/mol)

For example: Mean velocity of Argon @  $20^{\circ}$ C u =SQRT (8 x 8.31451 x 293 / 0.039948 x 3.1416) u =394 m/s

#### 3.3 Types of flow

The different flow phases need to be understood in order to select the appropriate pumping system for a particular application. It may be that in most cases a chamber or other piece of equipment is

pumped at the highest flow rate achievable with a given pump but it could be the case for example where a chamber has delicate films inside and gas movement past the foils has the potential to tear them apart. In this case a low steady flow rate is needed.

The flow phases in a vacuum system characterise the gas molecule movement related to pumping speed in a vacuum system. Factors that determine flow include pressure differentials, mean free paths, gas type, geometry, dimensions of the system and temperature. There are many different nomenclatures used to represent flow phases but 3 types in particular are used in the accelerator area, Turbulent, Laminar (both in Viscous phase), and Molecular. Less emphasis is placed on the intermediate or transitional flow as once this state is reached it is assumed that the system is pumping well. At atmospheric pressure up to about 100 Pa, the mean free path of the gas molecules is very small. Therefore, the gas flow is limited by the viscosity of the gas being pumped so the type of flow is called Viscous.

In an *ideal* system, if a roughing pump has a pumping speed of say 22 m<sup>3</sup>/hr and there is negligible flow resistance between the pump and the chamber then a chamber of  $\emptyset$ 300 mm ID x 500 long (a volume of 0.35 m<sup>3</sup>) at atmospheric pressure, will take approximately 1 minute to remove the bulk of the gas. During this time the flow remains in the [Viscous] Laminar and/or Turbulent phase.

Where a system has a leak, depending on the magnitude of the leak it is possible that the pumping will plateau at either the Turbulent, Laminar or Molecular flow phase. With outgassing systems the less referred to transitional state may be reached and with systems designed to reach ultra high vacuums outgassing may be occurring well into the molecular flow state.

Pumping	Flow Phase		Description	Type of vacuum	Flow rate Pa-l/sec
Pumping begins		Turbulent Flow	Initial flow experienced at start of pumping or in a low vacuum environment where the flow rate is high. Chaotic movement of molecules. Eddies and other non-parallel flows occur.	Low Vacuum	>1.01
	Viscous	Laminar Flow	Initial flow experienced at start of pumping or in a low vacuum environment where the flow rate is low. Gas molecules bouncing off other gas molecules (gas-gas) in the vacuum system. Short mean free path. Molecules streaming from system.	Medium Vacuum	10.1 to 1.01 x 10 <sup>-4</sup>
	Transit (Kn	ional Flow udsen)	Intermediate flow between laminar and molecular. Flow is dominated by both gas-gas and gas-wall collisions.	Medium to High Vacuum	1.01 x 10 <sup>-2</sup> to 1.01 x 10 <sup>-5</sup>
Base vacuum reached	Molec	ular Flow	Remaining gas molecules bounce off the walls (gas-wall) of the vacuum system and not each other. Long mean free paths. Flow rate very small.	High to Ultra-High Vacuum	<1.01 x 10 <sup>-5</sup>

Table 7. Vacuum flow phases

By evaluating the Knudsen (Kn) and Reynolds (Re) number, one can predict the various flow phases. These phases can be seen in the picture below which also shows how the gas molecules behave in the different flow phases moving through the opening d.

#### 3.4 Knudsen Number (Kn)

The Knudsen number is a dimensionless number defined as the ratio of the molecular mean free path length to a representative physical length scale. The Knudsen number in a viscous flow phase < 0.01 and molecule-molecule collisions dominate gas behaviour which behaves as a fluid. In molecular flow Kn > 1 and molecule-surface collisions dominate.

$$Kn = \frac{\lambda}{d}$$

 $\lambda$  = Mean free path

d = diameter of the opening that gas will pass through

#### 3.5 Reynolds Number (Re)

Reynolds number indicates whether the flow of a gas is absolutely steady (laminar flow) or on average steady but with small, unsteady changes (turbulent flow). The Reynolds number, Re, has no dimensions and is defined as the size of the flow.

Osborne Reynolds demonstrated in 1883 that the change from laminar to turbulent flow in a pipe occurs when the value of the Reynolds number exceeds 2,100. The exact value of Re for which the flow changes from laminar to turbulent depends on the geometry of the component, its surface roughness and other experimental factors. During evacuation of a vessel, turbulent flow normally occurs only for a short period of time at the beginning. It has been found to be approximately proportional to the root of the pressure gradient.

$$\operatorname{Re} = \left(\frac{\rho}{\eta}\right) d.v$$

 $\rho$  = gas density (kg/m<sup>3</sup>)  $\eta$  = viscosity (Pa.s) (Pascal-second) v = flow velocity (m/s) d = tube diameter (m)

Gas	Formula	Molecular weight	Gas Density [2] kg/m <sup>3</sup>	Viscosity η Pa.s
Air [1]	$N_2 + O_2$	29	1.1839	1.79 x 10 <sup>-5</sup>
Ammonia	NH <sub>3</sub>	17.031	0.7449	1.01 x 10 <sup>-5</sup>
Argon	Ar	39.948	1.723	2.24 x 10 <sup>-5</sup>
Carbon Dioxide	$CO_2$	44.01	1.9105	1.51 x 10 <sup>-5</sup>
Carbon Monoxide	CO	28.01	1.2082	1.74 x 10 <sup>-5</sup>
Chlorine	Cl <sub>2</sub>	70.906	3.1124	1.34 x 10 <sup>-5</sup>
Helium	He	4.02	0.171	1.99 x 10 <sup>-5</sup>
Hydrogen	$H_2$	2.016	0.0868	8.80 x 10 <sup>-5</sup>
Hydrochloric Acid	HCl	36.5	1.5844	1.46 x 10 <sup>-5</sup>
Hydrogen Sulphide	$H_2S$	34.076	1.4876	1.26 x 10 <sup>-5</sup>
iso-Butane	$C_4H_{10}$	58.12	2.3758	7.49 x 10 <sup>-5</sup>
Methane	$CH_4$	16.043	0.6556	1.11 x 10 <sup>-5</sup>
Nitrogen	$N_2$	28.02	1.2088	1.76 x 10 <sup>-5</sup>
Oxygen	$O_2$	32	1.381	2.02 x 10 <sup>-5</sup>
Propane	C <sub>3</sub> H <sub>8</sub>	44.09	1.8024	8.26 x 10 <sup>-5</sup>
Sulphur Hexafluoride	$SF_6$	146.5	6.27	$1.53 \times 10^{-5}$

Table 8. Gas density and viscosities

Unless otherwise indicated gases referenced from McGraw Hill Chemical Properties Handbook 1 atm 25°C. [1] Air STD Atmosphere at Sea Level: 1 atm 15°C [2] Gas Density Values Interpolated From 15°C Data using Charles Law

http://pipeng.com/index.php/gsts/itdmodflup002a/itddaflup00201

# 3.6 Viscous Flow

# Low vacuum, p = 100000 - 100 Pa, where $\lambda \ll d$

What characterises viscous flows, is frequent contact between gas molecules, yet less frequent contact with the walls of the vessel. The mean free path of the gas molecules is therefore very small and the gas flow is limited by the viscosity of the gas being pumped (Viscous Flow). In this case, the mean free path of the gas molecules is significantly shorter than the dimensions d of the vacuum equipment. For both Laminar and Turbulent Flow, Kn < 0.01. In addition, the term viscous flow is used if the product of pressure p and diameter d of the components through which gas is flowing is p.d  $\geq$  60 Pa.cm for air. Whether the flow is in the Laminar or Turbulent flows phases can be calculated using the Reynolds Number formula above.

# 3.7 Knudsen flow

# Medium vacuum, p = 100 - 0.1 Pa with $\lambda \le d$

If the Knudsen number is between 0.01 and 0.5, this is termed Knudsen flow. Because many process pressures are in the medium vacuum range, this type of flow occurs with corresponding frequency. Since this is a transitional flow, this range is transited relatively quickly when pumping down vacuum chambers and hence not usually considered in accelerator systems. This means that the influence of this conductivity on pump-down times is correspondingly low. A simple approximation for the Knudsen range can be obtained by adding the laminar and molecular conductivities. The

table in "Conductivities" below shows the conductivities of round, one meter long tubes of differing diameters in all three flow ranges.

# 3.8 Molecular flow High vacuum, $(p = 0.1 - 10^{-5} \text{ kPa})$ , where $\lambda > d$ and in UHV $(p < 10^{-5} \text{ kPa})$ , with $\lambda >> d$

At Knudsen numbers of Kn > 0.5 molecule-molecule collisions virtually no longer occur. What prevails is molecular flow. In this case, the product of pressure p and component diameter d is p x d  $\leq$  1.3 Pa.cm.



Fig 5. Molecular paths during different flow phases Re reference: http://www.engineersedge.com/fluid\_flow/pressure\_drop/pressure\_drop.htm

# **3.9 Pump throughput qpV**

The concept of pump throughput is of major significance in practice and should not be confused with the pumping speed! The pump throughput is the quantity of gas moved by the pump over a unit of time, expressed in Pa.1.s<sup>-1</sup>. Conversely, the pumping speed is the capacity of the pump to remove a volume of gas within a specific unit of time, measured in  $m^3/h$  or 1/s.

The throughput value is important in determining the size of the backing pump in relationship to the size of a high vacuum pump with which it is connected in series in order to ensure that the backing pump will be able to take away the gas moved by the high vacuum pump.

The pumping capacity (throughput) for a pump is equal either to the mass (m) flow through the pump intake port in a specified time:

$$q_m = \frac{m}{t}$$

Or to the pV (quantity of gas) flow through the pump's intake port:

$$q_{pV} = \frac{pV}{t}$$

It is normally specified in Pa.l.s<sup>-1</sup>. Here p is the pressure on the intake side of the pump. If p and V are constant at the intake side of the pump, the throughput of this pump can be expressed with the simple equation:

 $q_{pV} = p.S$ 

Where:

S = pumping speed of the pump at intake pressure of p.

#### 3.10 Speed of vacuum pump S

The speed of a vacuum pump is defined as

$$S = \frac{q_{pV}}{p}$$

p = Pressure at the pump inlet

However, pumps are usually connected to vacuum chambers via hoses resulting in a lower effective pumping speed  $S_{eff}$  at the chamber. If  $p_1$  is the pressure at the pump and  $p_2$  is the pressure in the vacuum chamber then:

$$q_{pV} = S.p_1 = S_{eff}.p_2$$

for continuous flow. Combining this with  $q_{pV} = C(p_1 - p_2)$  (see Conductance below) results in an expression for S<sub>eff</sub>

$$S_{eff} = \frac{S.C}{S+C}$$

#### **3.11** Conductance C

In a vacuum system, the volume that is pumped by a vacuum pump exhibits a level of flow resistance (Z) due to chamber and pipe sizes and their geometries, and the various sized orifices and other in-stream devices. The terminology used to describe this effect is the reciprocal of flow resistance which is *conductance* (C) or the systems ability to conduct gas.

Conductance is expressed in either l/s or sometimes  $m^3/h$  and is usually for steady, continuous flow through one or more components in a vacuum system. It is affected by the geometry of the piping element and relative to the flow phase and capacity of the pump used. In the high and ultrahigh vacuum ranges (molecular flows), C is a constant which is independent of pressure. In the rough and medium-high vacuums (turbulent and laminar) it is, by contrast, dependent on pressure. As a consequence, the calculation of C for the piping (beam line) elements must be carried out separately for the individual pressure ranges.

Flow resistance (sec/litre) 
$$Z = \frac{P_1 - P_2}{q_{_{PV}}}$$

Conductance (litre/sec) therefore

 $C = \frac{1}{Z} = \frac{q_{_{PV}}}{P_1 - P_2} \qquad (continued over)$ 

$$q_{pV} = C(p_1 - p_2)$$

Where:  $q_{pV}$  = Throughput flow rate p = Pressure (Pa)

At the molecular level, the conductivity of a system is reduced due to the friction of both the walls of the system and other molecules. As discussed above in "Types of Flow", these two affects occur at different flow phases. The total effect of conductance is the sum of the various conductance elements. In the case where a valve, a trap and an elbow in series each having a different conductance, the sum is:

$$\frac{1}{C_{total}} = \frac{1}{C_{valve}} + \frac{1}{C_{trap}} + \frac{1}{C_{elbow}}$$

If the components with different conductances are connected in the flow path in parallel, for example, 2 different diameter tubes connecting a chamber to a pump then the equation will be:

$$C_{_{Total}} = C_{_{tube1}} + C_{_{tube2}}$$

For the design of vacuum systems used on the accelerators conductance is mostly calculated for systems in molecular flow.



*Fig 6. Diagram for estimating pipe conductance Pupp/Hartmann, Vakuumtechnik, Grundlagen und Anwendugen, Hanser Verlag* 

# 4. Limitations

The most fundamental problems with vacuum systems are leaks and outgassing. Leaks can be categorised as either real leaks where gas enters the vacuum system from outside of the vessel or leaks form trapped voids which is in effect an internal leak. Trapped voids can contain gas that will continue to outgas for long periods reducing the ultimate vacuum that can be reached in a vessel. Real leaks can be found with the aid of a helium leak detector whereas internal leaks from trapped voids may never be found.

Outgassing can be minimised through careful selection of materials used within the vacuum space. The appendices have helpful tables to estimate outgassing rates. Outgassing may be in the form of desorption, diffusion or vaporisation. Permeation is not outgassing as the source of gas originates from outside of the vacuum space.

Examples commonly used substances in the accelerator systems that outgas,:

- Teflon, PVC, Ertalyte
- Viton, neoprene
- Copper, aluminium, stainless steel, brass, tantalum, rubidium, caesium, lithium, zinc
- Vacuum pump oil, vacuum greases, vacuum epoxies

These are just a few but it demonstrates that all materials will outgas at some point when the temperature and vacuum pressure reach their individual vapour pressure.

Neither of the problems is resolved by increasing the pumping speed (capacity) of the vacuum pump. There will be a point where the rate of gas entering the system will equalise with the pumping speed of the pump. The rate may vary depending on the vapour pressure of the substance outgassing and the temperature of that substance.



Fig 7. Limitation of Pumping From: http://www.mse.ncsu.edu/WideBandgaps/classes/MSE%20751/Handouts/HO02\_Leakage.pdf



Fig 8. Unwanted gas source wheel

# 4.1 Contamination

Vacuum chambers must be clean in order to reach the desired pressure as quickly as possible when they are pumped down. Typical contaminants include oil and grease on screws and seals, process reaction products or condensed vapours, particularly water that is adsorbed on the walls of the vessel. Consequently, it is necessary to ensure that the components are clean when installing vacuum equipment. All components attached in the vacuum chamber must be clean and grease-free. All seals must also be installed dry. If high or ultra high vacuum is to be generated, clean gloves must be worn during the assembly process.

Examples of common contaminants in accelerator vacuum systems

- Rotary pump oil
- Water
- Plasticisers from various plastics
- General airborne dust
- Machining oil
- Residual gases from stripper gases, venting gases, ion source gases
- Sample breakdown

# 4.2 Condensation and vaporisation

All substances can occur in a liquid, solid or gaseous state. Their aggregate status is a function of pressure and temperature. Liquids are transformed into their gaseous state through vaporisation, solids through sublimation. The separation of liquids or solids out of the gaseous phase is termed condensation. Since normal room air contains approximately 10g of water vapour per m<sup>3</sup>, condensed water vapour is always present on all surfaces.

Adsorption on surfaces is especially pronounced due to the strong polarity of the water molecules. Natural fibres, in particular, such as paper, contain large quantities of water that escape during drying processes under vacuum. Cooled condensers are used to separate the water vapour in this connection. Even some metals (Cd, Zn, Mg) can vaporise in noticeable quantity at temperatures of several 100 °C. Consequently, use of these metals is avoided in plant construction.

#### 4.3 Desorption

In addition to water, other substances (oil) can be adsorbed on surfaces. Substances can also diffuse out of the metal walls, which can be evidenced in the residual gas. In the case of particularly rigorous requirements, stainless steel vessels can be baked out under vacuum, thus driving the majority of the volatile components out of the metal walls.

Gas molecules, (primarily water) are bound to the interior surfaces of the vacuum chamber through adsorption and absorption, and gradually desorb again under vacuum. The desorption rate of the metal and glass surfaces in the vacuum system produces a gas yield that is a function of time, however. A good approximation can be obtained by assuming that after a given point in time  $t > t_0$  to the reduction will occur on a linear basis over time.  $t_0$  is typically assumed to be one hour.



Fig 9. Desorption curves



Fig 10. Adsorption Curves From: http://www.mse.ncsu.edu/WideBandgaps/classes/MSE%20751/Handouts/HO02\_Leakage.pdf

#### 4.4 Diffusion with desorption

At operation below  $10^{-4}$  kPa, desorption of plastic surfaces, particularly the seals, assumes greater significance. Plastics mainly give off the gases that are dissolved in these plastics, which first must diffuse on the surface.

Following extended pump downtimes, desorption from plastics can therefore dominate the metal surfaces. Although the surface areas of the seals are relatively small; the decrease in desorption rate over time occurs more slowly in the case of metal surfaces. As an approximation it can be assumed that the reduction over time will occur at the square root of the time.

The gas produced from plastic surfaces can thus be described as:

Desorption from plastic material  $Q_{diff} = q_{diff} \cdot A \sqrt{t_o/t}$ 

Where Ad denotes the surface area of the plastics in the vacuum chamber and  $q_{diff}$  denotes the surface area-specific desorption rate for the respective plastic. At even lower pressures, similar effects also occur with metals, from which hydrogen and carbon escape in the form of CO and CO<sub>2</sub> and can be seen in the residual gas spectrum.

#### 4.5 **Permeation and leaks**

For a gas passing through small holes in a thin wall in the Knudsen Flow regime, the number of molecules that pass through a hole is proportional to the pressure of the gas and inversely proportional to its molecular weight.

To Convert to Leakage	Multiply Helium Leak Rate by:			
Rate of:	Laminar Flow Molecular Fl			
Argon	0.88	0.316		
Air	1.08	0.374		
Nitrogen	1.12	0.374		
Water vapour	2.09	0.469		
Hydrogen	2.23	1.410		

Table 9. Conversation table for leak rates

Seals, and even metal walls, can be penetrated by small gas molecules, such as helium, through diffusion. Since this process is not a function of time, it results in a sustained increase in the desired ultimate pressure. The permeation gas flow is proportional to the pressure gradient  $p_0 / d$  (d = wall thickness,  $p_0 =$  atmospheric pressure = ambient pressure) and to the permeation constants for the various materials  $k_{perm}$ .

Permeation

 $k_{perm}$ .  $Q_{perm} = k_{perm}$ .  $A \cdot p_0/d$ 

Permeation first manifests itself at pressures below  $10^{-6}$  kPa.  $Q_1$ , denotes the leakage rate, i.e. a gas flow that enters the vacuum system through leaks at a volume of V. The leakage rate is defined as the pressure rise  $\Delta p$  over time  $\Delta t$ :

Leakage rate  $Q1 = (\Delta p \cdot V)/\Delta t$ 

If a vessel is continuously pumped out at a volume flow rate S, an equilibrium pressure  $p_{gl}$  will be produced. Throughput is equal to the leakage rate  $Q_l = S \cdot p_{gl}$ . A system is considered to be

adequately tight if the equilibrium pressure  $p_{gl}$  is approximately 10 % of the working pressure. If, for example, a working pressure of  $10^{-4}$  kPa is attained and the vacuum pump that is being used has a pumping speed of 100 I/s, the leakage rate should not be more than  $10^{-3}$  kPa I/s. This corresponds to a leak of approximately 20.20  $\mu$ m<sup>2</sup> in size. Leakage rates  $Q_l$  of less than  $10^{-6}$  kPa I/s can usually be easily attained in clean stainless steel vessels. The ultimate pressure achievable after a given period of time *t* primarily depends upon all of the effects described above and upon the pumping speed of the vacuum pump. The prerequisite is naturally that the ultimate pressure will be high relative to the base pressure of the vacuum pump.



Fig 11. Permeation of Different Gases From: http://www.mse.ncsu.edu/WideBandgaps/classes/MSE%20751/Handouts/HO02\_Leakage.pdf

# 4.6 Leaks

The source of leaks is as wide and varied as can be imagined. There is no such thing as a common leak however there are several types generally seen. The following lists types in rough order of prevalence:

- Dirty seals elastomer type seals with traces of dust, dirt and fibres on the surface
- Jarred seals ill fitted flanges where the seal lays across the sealing surface
- Vacuum pumping tubes where a fitting has been fitted without care and the pump cannot reach its full capability
- Distortion over tightened or misaligned flanges and fittings that "lift" seals off the sealing surface
- Stress cracks usually in places around flanges and fittings that have been incorrectly tightened
- Pressure dependant mostly related to accelerator tubes that develop leaky seals allowing insulation gas to enter when the pressure is greater than atmospheric pressure.

# 4.7 Bake-out

The following prerequisites must be satisfied in order to achieve lower pressures ( $<10^{-6}$  kPa):

- The base pressure of the vacuum pump should be a factor of 10 lower than the required ultimate pressure
- Stainless steel vacuum recipients and components must be used
- Metallic seals (CF flange connections) are required
- Leaks must be avoided and eliminated prior to activating the heater (use helium leak detectors!)
- Clean work is a must, i.e. all parts must be thoroughly cleaned and must be installed with grease-free gloves
- Pump and equipment must be baked out

Bake-out significantly increases desorption and diffusion rates, and this produces significantly shorter pumping times. Bake-out temperatures of up to 300°C are used. The instructions of the pump manufacturers relating to maximum bake-out temperatures and maximum permissible radiation levels in the pump flange must be observed.

Following installation the equipment is switched on, and after reaching a pressure of  $P < 10^{-3}$  kPa the heater is then switched on. During the heating process, all gauge heads must be operated and degassed at intervals of 10 hours. In the case of stainless steel vessels and the use of metallic seals, bake-out temperatures of 120°C and heating times of approximately 48 hours are sufficient for advancing into the pressure range of  $10^{-8}$  kPa. Bake-out should be continued until 100 times the expected ultimate pressure is attained. The heaters for the pump and vacuum chamber are then switched off. After cool-down, the desired ultimate pressure will probably be achieved. In connection with pressures  $P < 5 \times 10^{-8}$  kPa and large interior surface areas, it will be advantageous to use a titanium sublimation pump that pumps the hydrogen escaping from the metals at a high volume flow rate.

# 4.8 Residual gas spectrum

When leaks have been solved in a vacuum system and poor vacuum persists than a residual gas analysis (RGA) measurement should be made to determine the composition of the gas load. This will give vital information on where the vacuum problem may be. Interpreting the information will be trial and error as users will have to think about all possible sources of gases (and outgassing) that may be present. Also relative ratios of gases should be considered to make sense of the measurement. Care should be taken especially if making a measurement near a source such as a cryopump. Some gases that have been trapped on the cryopump may be liberating from the cold surface and will make up a part of the gas spectrum. Other places to take care are near the accelerator tubes and ion sources.

If developing an ultra high vacuum system it is important to know all sources of gas otherwise moving lower than  $1 \times 10^{-6}$  Pa will be an unnecessary challenge. For all other systems it is nice to know but not essential unless residual gases affect ion beam measurements. The dominate gases (and their masses) that may be seen include:

Gas	Mass	Gas	Mass
Water	18	Carbon Monoxide	28
fragment HO	17	Carbon Dioxide	44
Nitrogen	28	Argon	40
Oxygen	32	Sulphur Hexafluoride	146
Hydrogen	2	Helium	4

Table 10. Table of masses for common "in vacuum" gases

# **Appendix 1 – Structural Calculations for Scientific Vacuum Vessel Design**

Calculations for Vacuum Vessel wall and end plate thickness

The following formulae are from the Vacuum Society of Australia (VSA) training course notes which have been in circulation since the early 1980's. VSA continues to support these calculations for training vacuum technologists around Australia. The calculations provide conservative estimates of vacuum vessel wall thicknesses as compared with formulas used in ASME Section VIII – Division 1 UG-28 (Thickness of Shells and Tubes under External Pressure) and associated documents, which have been developed for larger industrial type vacuum vessels.

#### Cylindrical Vessel – Stainless Steel @ 20°C

Thickness of plate in a given diameter (h):

$$\frac{D}{h} \le 105, \ \frac{h}{D} \ge \frac{1}{105}$$

Where: D = diameterh = thickness

Maximum length of vessel for a given diameter (L<sub>c</sub>):

$$\frac{L_c}{D} \le 11.5$$

Where: D = diameter $L_c = \text{length}$ 

# **End Plates – Stainless Steel**

Thickness of plate in a given diameter (h<sub>1</sub>):

$$\frac{D_1}{h_1} \le 89$$
 ,  $\frac{h_1}{D_1} \ge \frac{1}{89}$ 

Where:  $D_1$  = diameter  $h_1$  = thickness

Minimum thickness for given deflection at centre:

 $\frac{h_1}{\delta} \ge 3$ 

Where:  $\delta$  = deflection  $h_1$  = thickness For Hemispherical End (h<sub>2</sub>):

$$\frac{R}{h_2} \le 830 \text{ requires } \frac{h_2}{R} \ge \frac{1}{830}$$

Where: R =radius  $h_2 =$  thickness

**Example 1:** To construct Stainless Steel vacuum chamber at  $20^{\circ}$  C as shown – Length L = 50 cm = 500 mm, D = 40 cm = 400 mm, Maximum allowable deflection at centre  $\delta = 1$  mm. Find  $h, h_1, h_2$  to satisfy vacuum chamber strength requirements.

# For Cylinder

1. 
$$\frac{D}{h} \le 105$$
,  $\frac{h}{D} \ge \frac{1}{105}$   $h \ge 400/105$ ,  $h \ge 3.8 \text{ mm}$   
2.  $\frac{L_c}{D} \le 11.5$ ,  $L_c \le 11.5 \times 400$   $L_c \le 4600 \text{ mm}$  with  $L = 500 \text{ mm} \checkmark$ 

#### **For End Plates**

1. 
$$\frac{D_1}{h_1} \le 89$$
,  $\frac{h_1}{D_1} \ge \frac{1}{89}$   $h_1 \ge 396.2/89$ ,  $h_1 \ge 4.45 \text{ mm}$   
2.  $\frac{h_1}{\delta} \ge 3$ , with  $\delta = 1 \text{ mm}$  then require  $h_1 \ge 3 \text{ mm}$ , however  $h_1 = 4.45 \text{ mm} \checkmark$ 

#### For Hemispherical End

1. 
$$\frac{R}{h_2} \le 830$$
 requires  $\frac{h_2}{R} \ge \frac{1}{830}$   $h_2 \ge 198.1/830 \ge 0.24$ mm

Dimension of cylindrical, planar and hemispherical parts of vacuum enclosures

	At	Cylinders		End Plates		Hemispherical
Material	Temp (°C)	D/h	$L_c/D$	$D_1/h_1$	$h_l/\delta$	<i>R/h</i> <sub>2</sub>
Copper	20	84	10	52	15	600
Copper	500	58	8.5	-	-	-
Nickel	20	100	11	73	8	780
Nickel	500	90	10.5	-	-	-
Aluminium Alloy	20	70	9	37	57	470
Aluminium Alloy	500	62	8.7	-	-	-
Stainless Steel (304)	20	105	11.5	89	3	830
Stainless Steel (304)	500	89	10.5	-	-	-
Glass (hard)	20	70	9	16	117	470
Neoprene	20	2.5	1.7	10	0.2	30
Teflon	20	12	3.8	14	9	-
PVC (Tygon)	-	3.7	2.1	-	-	-
Perspex	-	-	-	30	-	-
Mica	-	-	-	58	15	-

Factor tables for thickness calculations of common materials used in high vacuum systems

 $\delta$  = maximum permissible deflection at centre of plate L<sub>c</sub> = critical length for cylinder = 1.11D(D/h)1/2

**Example 2:** Using the ASME Boiler and Pressure Vessel Code (the Code) Section VIII, Division 1 for the safe design pressures for a vacuum vessel is used to calculate the maximum external pressure acceptable for a given wall thickness. The following example uses the same vessel dimensions as in Example 1, with a wall thickness of 3.8 mm as calculated using the VSA method.

The calculations reference the Vacuum Vessel Cylindrical Shell Thickness using paragraphs UG-28, UG-27 of ASME Boiler and Pressure Vessel Code.

Vessel shell thickness t = 3.8 mm  $D_o = 400$  mm (outer diameter of shell)  $D_o/t = 105.26$  Since this ratio is greater than 10, follow UG-28(c)(1) L = 500 mm (length of stiffened shell)  $L/D_o = 1.25$ E = 28 x 10<sup>6</sup> (modulus of elasticity for 304 stainless steel)

From Table G in Sec II, Subpart 3 to determine Factor A

A = 1 x 10<sup>-3</sup> (Factor A, from Table G of Sec. II, Subpart 3)  $P_{ext} = \frac{2AE}{3(\frac{D_o}{t})}$ 

 $P_ext = 177.33$  psi (max allowable working external pressure for given *t*) = 1222 kPa

A wall thickness of t = 3.8 mm results in a maximum allowable working external pressure of P\_ext = 325 kPa, which is greater than the external pressure that the vacuum vessel will see.

**Example 3:** For comparison, calculate the maximum external pressure as in Example 2 but with a wall thickness of 2.5 mm.

 $D_{o} = 400 \text{ mm}$ t = 2.5  $D_{o}/t = 160$ L = 500 L/D\_{o} = 1.25 E = 28 x 10<sup>6</sup> (modulus of elasticity for 304 stainless steel)

From Table G in Sec II, Subpart 3 to determine Factor A

A =  $5.3 \times 10^{-4}$  (Factor A, from Table G of Sec. II, Subpart 3)

$$P\_ext = \frac{2AE}{3(\frac{D_o}{t})}$$

P\_ext = 61 psi (max allowable working external pressure for given t of 2.5 mm) = 420 kPa

Therefore a wall thickness of 2.5 mm has a P\_ext of 420 kPa.

**Example 4:** This is a worked example for a large vacuum vessel from ASME Boiler and Pressure Vessel Code (the Code) Section VIII, Division 1, paragraph UG-28. It is in imperial units.

Calculate the maximum allowed external working pressure following UG-28. The minimum required thickness takes into account the support of the stiffening rings. Note that the actual vessel shell thickness t = 0.375-inch

 $D_o = 38.0$  inch (965 mm) (outer diameter of shell) t = 0.261 inch (6.63 mm) (minimum required thickness of shell for external pressure of 14.5-psia)  $D_o/t = 145.594$  Since this ratio is greater than 10, follow UG-28(c)(1) L 235.7 inch (6057.5 mm) (length of stiffened shell)  $L/D_o = 6.203$ E =29x10<sup>6</sup> psi (modulus of elasticity for SA-516 Gr 60 carbon steel)

From Table G in Sec II, Subpart 3 to determine Factor A A =  $1.108 \times 10^{-4}$  (Factor A, from Table G of Sec. II, Subpart 3)

$$P\_ext = \frac{2AE}{3(\frac{D_o}{t})}$$

P\_ext = 14.709 psi (maximum allowable working external pressure for given t) = 101.35 kPa

A wall thickness of t = 0.261 inch results in a maximum allowable working external pressure of P\_ext = 14.7 psia, which is greater than the external pressure that the vacuum vessel will see. Since the actual wall thickness is 0.375 inch (9.5 mm), the vessel design is adequate for the working external pressure.



Factor A table for Stainless Steels

# **Appendix 2 – Low Outgassing Specialist Non Metal Materials**

# • Celazole® PBI (PolyBenzImidazole) http://www.boedeker.com/celazo\_p.htm

Celazole® is the highest temperature-capable plastic available. However, it is very brittle (almost ceramic-like) and quite difficult to machine. That said, is it frequently used for bushings, bearings, rollers, and spacers in extreme environments. Its outgassing values are listed as 2.50% TML, 0.00% CVCM, 0.40% WVR.

# • Vespel® (Polyimide)

http://www2.dupont.com/Vespel/en\_US/assets/downloads/vespel\_gen/E61500.pdf

DuPont Vespel® SP-1 is one of the most-used high-temperature plastic materials used in applications where high-purity and electrical properties are needed. Vespel is frequently used in ultra-clean semiconductor and chemical applications. It is also one of the most expensive materials sold, but is flight-approved for NASA, USAF and other aerospace agencies. Its NASA outgassing values are listed as 1.09% TML, 0.00% CVCM, 0.40% WVR.

# • Duratron® XP (Polyimide)

http://www.portplastics.com/download/pdf/plastics/highPerformance/highPerformance26.pdf

Duratron® XP is the first real alternative to Vespel ... it was developed specifically to replace Vespel in extreme applications at a slightly lower price. It contains less than 1% metallic impurities as measured using the ICP-MS test standard. Duratron XP is ideal for use in high-energy gas plasma etch and strip processes. Outgassing values for Duratron XP are 0.75% TML, 0.00% CVCM, 0.49% WVR.

# • Torlon® 4203 unfilled PAI (PolyAmide-Imide) http://www.boedeker.com/torlon-4203.htm

Unfilled Torlon® 4203 has high dielectric properties and low thermal expansion, and is much less expensive than some advanced polymers. Torlon 4203 is typically used for insulators, spacers, and mechanical parts up to 520°F. Its outgassing values are listed as 1.85% TML, 0.00% CVCM and 0.49% WVR.

# • Torlon® 5530 glass-filled PAI (PolyAmide-Imide) http://www.boedeker.com/torlon-5530.htm

Torlon 5530 (30% glass-filled) is typically used for applications where dimensional stability over a wide temperature range is needed, as with temperature test sockets, nests, and fixtures. Its outgassing values are listed as 0.58% TML, 0.00% CVCM (% WVR is not shown). NOTE: Torlon's moisture absorption is a bit high, so critical dimensional stability can be an issue.

# • Semitron® ESd 500HR (filled PTFE)

http://www.portplastics.com/download/pdf/plastics/staticControl/staticControl18.pdf

Semitron® ESd 500HR is antistatic/conductive PTFE. This material is relatively clean, readily machinable, dissipates static electricity reliably ... as a result it is used in test handling equipment, fixtures, and other applications where static generation may cause failures and/or errors in production environments. This material has low outgassing values of 0.04% TML, 0.00% CVCM and 0.01 % WVR. PTFE has good mechanical properties up to approximately 500°F.

# • Neoflon® PCTFE (PolyChloroTetraFluoroEthylene)

http://www.aftonplastics.com/materials/pdfs/neoflan\_pctfe.pdf

PCTFE exhibits high chemical resistance, low and high temperature capability, resistance to most chemicals (including strong acids and bases), low friction, electrical and thermal insulation, and "slipperiness". PCTFE has the lowest outgassing values of any thermoplastic material we sell ... 0.01% TML, 0.00% CVCM, 0.00% WVR.

• **PEEK (PolyEtherEtherKetone)** http://www.dotmar.com.au/ketron-peek-1000/ketron-peek-1000-polyetheretherketone.html

PEEK is pure, easily machinable, chemically resistant, stable, and also has relatively low outgassing values (0.31% TML, 0.00% CVCM, 0.06% WVR). PEEK has good mechanical properties, but will not take temperatures over  $350^{\circ}$ F, so it may not have the mechanical or thermal performance needed.

• **Techtron® PPS (PolyPhenylene Sulfide)** http://www.dotmar.com.au/techtron-hpv-pps/techtron-hpv-pps.html

Techtron® PPS is easily machined to close tolerance, has excellent mechanical, thermal and chemical stability and has one of the lowest outgassing values of any thermoplastic material we offer (0.04% TML, 0.00% CVCM ... % WVR is not shown). Techtron PPS is generally a bit less expensive than PEEK or Torlon, but again, will not take as high temperatures.

• Ultem® PEI (PolyEtherImide) http://www.boedeker.com/ultem\_p.htm

Ultem<sup>®</sup> has good dielectric properties and low thermal expansion, and is considerably less expensive than some other polymers. PEI is also clean and stable, but is not particularly resistant to chemicals or solvents ... it has outgassing values of 0.40% TML, 0.00% CVCM and 0.06 % WVR. PEI has good mechanical properties up to approximately 410°F.

• Semitron® ESd 410C (filled PEI) http://www.boedeker.com/sem410\_p.htm

Semitron® ESd 410C is antistatic/conductive PEI. This material is relatively clean, readily machinable, dissipates static electricity reliably ... as a result it is used in test handling equipment, fixtures, and other applications where static generation may cause failures and/or errors in production environments. This material has outgassing values of 0.46% TML, 0.00% CVCM and 0.17 % WVR. PEI has good mechanical properties up to approximately 340°F.

# • Ertalyte® PET-P (Polyethylene Terephthalate) http://www.boedeker.com/ertaly\_p.htm

Ertalyte® offers the dimensional stability of acetal with the wear resistance of nylon. Ertalyte® PET-Polyester is clean, chemically resistant, stable, and also has relatively low outgassing values (0.13% TML, 0.00% CVCM ... % WVR is not shown). PET-P is considerably less expensive than most of the other materials listed above, but may not have the mechanical or thermal performance needed for all applications.

# • Semitron® ESd 225 (filled acetal) http://www.boedeker.com/sem225\_p.htm

Semitron® ESd 225 is antistatic/conductive acetal. This material is relatively clean, readily machinable, dissipates static electricity reliably ... as a result it is used in test handling equipment, fixtures, and other applications where static generation may cause failures and/or errors in production environments. This material has low outgassing values of 1.00% TML, 0.05% CVCM and 0.60 % WVR. Acetal has good mechanical properties up to approximately 180°F.
### Appendix 3 – Materials for use in Vacuum

The choice of materials is limited for vacuum systems to a range that has little impact on vacuum production and ultimate base vacuums.

#### 1.1 Metals

- <u>Stainless Steel</u> is used for the majority of vacuum system designs due to its:
  - o Durability
  - Surface finishing
  - Ability to withstand moderate temperatures for system bake out
  - Low magnetic susceptibility
  - Low thermal conductivity
  - Resistance to oxidisation
  - Low vapour pressure

Type 304 or 316 stainless steel is ideally suited for vacuum vessel construction because of its machining/welding characteristics, excellent corrosion resistance and overall cost effectiveness.

Not all stainless alloys are acceptable. Free-machining 303 steel contains sulphur, which tends to outgas. Alloys with good weldability using TIG or MIG welding are usually chosen.

- 304 or 316 stainless steel is a common choice of a stainless steel.
- $\circ~$  304L stainless steel, a low-carbon variant of 304 steel, is used for ultra-high vacuum systems.
- 347 stainless steel does not accept high polish.
- $\circ$  321 stainless steel is chosen when low magnetic permeability is needed.
- <u>Mild steel</u> is okay for low to moderate vacuums above 10<sup>-4</sup> Pa. Outgassing can be reduced with suitable plating such as nickel. It has high permeability to hydrogen and tendency to rust. Mild steel must not be used for any components or chambers used near the bending magnets
- <u>Aluminium alloys</u> are easily machined and have a low vapour pressure, unless the alloys contain high proportion of zinc. 6061 grade is good for general use. Components used in high vacuums or better must not be anodized, as the oxide layer can trap water vapour and outgas. Aluminium and its alloys have low strength at high temperatures, distort when being welded, and the copper-containing ones are poorly weldable. Aluminium wire rings can be used as cheap gaskets in demountable seals. Soft alloys must be used to ensure wire gaskets do not damage the seal faces. Aluminium has high thermal conductivity, good corrosion resistance, and low solubility of hydrogen. Loss of strength at high temperatures limits its use in bakeable applications, but aluminium is advantageous for large-size systems due to its lower weight and lower cost than stainless steel. Aluminium is not recommended for vacuum chambers.
- <u>Brass</u> is not acceptable for vacuum chambers however it may be suitable for some small beam line inserts for specific applications. Once used widely but copper can cause problems with neutron production if struck by proton beams of particular energies. Brass is for high thermal conduction applications in cooling baffles or sample mounts that may be heated. Although bare

brass has good corrosion resistance the zinc content may cause outgassing problems. This can be reduced by plating with nickel.

- <u>Nickel</u> is widely used in vacuum technology, e.g. as mechanical parts in vacuum tubes. It is relatively low-cost, can be spot welded, can be easily machined, has high melting point and is resistant to many corrosive fluids and atmospheres. Its potential drawback is its ferromagnetism, which restricts applications that would be influenced by magnetic fields.
- <u>Beryllium</u> is used primarily for x-ray windows.
- <u>Oxygen-free copper</u> is okay for high vacuum but it is difficult to outgas completely. Copper is insensitive to hydrogen and impermeable to hydrogen and helium, has low sensitivity to water vapour, but is attacked by mercury. Although, oxygen-free copper is widely used as it is easily machined and has good corrosion resistance. It is unsuitable for bakeable vacuum envelopes due to its tendency to oxidize and create scales. Conflat flange seals are made from copper. Its strength falls sharply above 200 °C. Its vapour pressure becomes significant at above 500 °C.
- <u>Indium</u> wire is used as a gasket in demountable seals. Not suitable for high temperature above  $\sim 100^{\circ}$ C applications.
- <u>Gold</u> wire is used as a gasket in demountable seals for ultra-high vacuum.
- <u>Tantalum</u> is acceptable in vacuum systems but it is difficult to work with due to its hardness. It is expensive so it is usually purchased in sheet form. Typically mounted as a shield where ion beams contact a surface as it has a low production of secondary electrons. Good for apertures and defining slit faces and anywhere that a metal edge is used to intersect the ion beam.
- <u>Zirconium</u> is corrosion-resistant. Like tantalum, it has low production of secondary electrons, so it is used as a shield of areas where reducing their production is important. It is used for neutron windows. It is expensive and rarekly used. Zirconium and zirconium hydride are used for gettering.

#### **1.2 Plastics**

Plastics or ceramics are not to be used as the primary vacuum chamber structure without engineering advice from ANSTO or a specialist scientific vacuum instrument company. Plastic is very good for electrical insulators, bushes or light weight components are necessary in vacuums.

- <u>Polytetrafluoroethylene</u>, PTFE or Teflon as it is commonly known is suitable for use inside of vacuum systems from low to ultra high vacuums. Being soft PTFE can flow (creep) so for applications such as sample positioning systems or other systems requiring high stability then other plastics may need to be considered. PTFE has be far the highest dielectric strength, for extruded PTFE, 19.7 kV/mm 60 173 kV/mm for an insulating film. This compares with air which is around 1 kV/mm. It is self-lubricating, tolerant to fairly high temperatures, and has low outgassing. It is not suitable for barrier between vacuum and atmosphere, due to its permeability.
- <u>Ertalyte</u> offers the dimensional stability of acetal with the wear resistance of nylon. It is stable with relatively low outgassing values and has very low water absorption. It is very good for small mechanical loads requiring electrical isolation in vacuum systems.

- <u>Polyvinyl Chloride (PVC)</u> is acceptable in vacuum systems but not where heat is above 50°C. It is usually an amorphous thermoplastic material with excellent chemical resistance and dielectric properties, good tensile, flexural and mechanical strength, low moisture absorption, exceptional dimensional stability and good flammability characteristics.
- Other plastics can be used in vacuum systems but must have low vapour pressure. Care must be taken to ensure they are not used in applications that may require temperatures that will cause high outgassing rates.
- <u>Perspex</u> (acrylic plastic) is another plastic which has good dielectric properties but a high vapour pressure especially at raised temperatures making it unsuitable in vacuums however it can be used in small amounts in a well pumped high vacuum system.
- <u>Vespel</u>, a polyimide, is very expensive, but machines well, has good electrical insulator properties and is compatible with ultra-high vacuum. It does however absorb moisture and requires a longer pump down time. It also performs well in extremely low cryogenic temperatures. Good for bushes or where a mechanically stable plastic is required.
- <u>Nylon</u> is self-lubricating but has high outgassing rate and relatively high water absorption.
- <u>Polycarbonates and polystyrene</u> are good electrical insulators with moderate outgassing.
- <u>Mylar</u> is used to make thin windows that allow ion beams to penetrate from vacuum to positive pressures with little energy loss. For example gas detectors mounted onto beam lines for AMS have gas inside of the detector separated from the beam line which is under vacuum. Ion beams pass through the window as they move from the vacuum side to the positive pressure side. Other materials in this category include Kapton which is rated for higher temperatures.

#### **1.3 Elastomers**

Some elastomers have sufficient vacuum properties and are used widely as vacuum seals in the form of 'o' rings.

- <u>Viton</u> is the standard seal used throughout the accelerator area as it is long lasting, low vapour pressure as compared with other elastomers and is bakeable to 200 °C.
- <u>Nitrile</u> rubber is used for vacuum seals. Does tend to break down over time. First signs of breakdown are small splits. If used for vacuum seals then they must be inspected regularly.
- <u>Natural</u> rubber is not typically used in vacuum systems other than inside of vacuum pumps usually on the low vacuum side of the system.
- <u>Silicone</u> rubber is not generally used as a seal in high vacuum environments. Silicone is soft when compared with other elastomers. It has a relatively low vapour pressure.

Further reading: Materials for high vacuum technology: an overview, S. Sgobba, CERN, Geneva, Switzerland. http://cdsweb.cern.ch/record/983744/files/p117.pdf

#### 1.4 Ceramics and glass

• <u>Alumina</u> ceramic based ceramics in vacuum systems perform well provided the ceramic is not porous which can trap gas bubbles. Ceramics are readily available bonded onto metal flanges or mounting plates. Ceramic can also be baked if necessary to speed up the outgassing of surfaces.

Glass is similar to ceramics in this application. It is very low vapour pressure but difficult to work with. Glass viewports can be purchased already mounted in a metal flange.

- <u>Borosilicate glass</u> is often used for smaller assemblies and for viewports.
- <u>Porcelain</u> ceramics, when fully vitrified and therefore non-porous, are excellent insulators usable to 1500 °C. it is generally commercially available bonded to metal in electrical insulators and other feedthroughs.
- <u>Mica</u>, although it is neither ceramic or glass it best fits in this category. Mica has been used in vacuum systems for both electrical and thermal insulation but because Mica is a series of laminations it contains trapped air which is not suitable in high to ultra-high vacuums.
- <u>Macor</u> is a machinable glass made by Corning Incorporated with similar properties to ceramics. It has a low vapour pressure and thermally stable up to 1000°C making it bakeable. It is made from mica and borosilicate glass. It is by far the most flexible of the glass-ceramic options.

 Leakage Testing Handbook, Prepared for Liquid Propulsion Section, Jet Propulsion Laboratory, National Aeronautics and Space Administration, Pasadena, California
 Nondestructive Testing Handbook, Volume One, Leaktesting, American Society for Nondestructive Testing.
 Leakage Testing Handbook, Revised Edition, July 1969, General Electric.
 Fluid Flow in Small Passages, Mars Hablanian, J.W.Marr, Varian

#### 1.5 Greases and oils

There are few acceptable greases and oils for use in vacuum systems. Users must ensure they purchase greases and oils specifically designed for use in vacuums. Typically they will have low vapour pressures, extremely low water absorption and easy to remove for cleaning.

- <u>Apiezon family</u> For further information http://www.apiezon.com/ . These have been used throughout the history of accelerators at ANSTO with excellent results. Wherever greases are used remember to use the minimalist amount.
- <u>Greases</u>
  - Apiezon H High Temperature Vacuum Grease, silicone and halogen free
  - Apiezon L Ultra high vacuum grease, silicone and halogen free
  - Apiezon M High vacuum grease, silicone and halogen free
  - Apiezon N Cryogenic high vacuum grease, silicone and halogen free
  - Apiezon T Medium temperature grease, silicone and halogen free
  - o Apiezon AP100 Ultra High Vacuum Lubricating Grease, silicone free
  - o Apiezon AP101 Anti seize Vacuum Grease, silicone free
- <u>Waxes</u>
  - Apiezon® Waxes and Compounds
  - Apiezon Wax W (Hard Vacuum Sealing)
  - Apiezon Wax W40 (Softer Vacuum Sealing)
  - Apiezon Wax W100 (Softest Vacuum Sealing)

- Apiezon Sealing Compound Q
- <u>Vacuum Oil</u> is used in few applications around the accelerator area. With a change to oil free pumps most oil consuming pumps are redundant. However these types of pumps may still in use in other applications. Oil vane roughing pumps and diffusion pumps were the largest consumers of oil. More recent brands of vane pumps were specifically tuned to oils of particular specifications so generic oils may not be compatible. Diff pumps are more forgiving and high quality generic synthetic oils may be used. The list below is a small sample of what is available and previously used.
  - $\circ\,$  Shell Vitrea 68 oil for roughing pumps. Check specification on pump to ensure compatibility with this mineral oil
  - Fomblin PFPE (Perfluorpolyether) oils designed for rotary pumps. Check specification on pump to ensure compatibility with this mineral oil
  - Apiezon® Diffusion Pump Fluids Apiezon AP 201 The Apiezon diffusion pump fluid is known for its exceptional higher degree of oxidation resistance.

### **Appendix 4 – Surface finishing**

In most cases a clean polished surface will yield the least outgassing as there are minimal surface traps for gas. Also, a polished surface is easier to clean and keep clean. Achieving a polished surface may not be practicable due to accessibility into tubes, chambers and small components but a combination of other methods may provide an adequate surface finish that will allow the desired vacuum level to be reached. The best method for a given surface will depend on the material, geometry and accessibility to its surfaces.

- <u>Polishing</u> A polished surface may be either smooth or uneven but the metal will lustre in either case. Polishing is a method of removing a layer of metal to expose clean metal underneath. This can be done with a mechanical buff or by hand with the aid of a polishing compound. Care must be taken to ensure all traces of the polishing compound are removed during final cleaning. All residues must be removed.
- <u>Grinding, sanding and other coarse to fine abrasive methods</u> These methods tear the surface dragging metal over metal. Some metal breaks away exposing clean metal underneath. The problem with this type of surface preparation is that the metal can fold over and not break away leading to voids of trapped gases. It is important that a fine grit is the last grit used to ensure larger metal drifts on the surface are torn away.
- <u>Grit blasting with grit or beads</u> This method propels grit or beads onto the metal surface causing fragments of metal to be removed with the impact. Like the abrasive techniques metal can be layered on metal (peening) if the grit is too coarse. Best to finish with a fine grit then a mechanical polish. Grit blasting can provide a more uniform surface finish and is very good for removing scale that may be present from rolling or extrusions.
- <u>Pickling</u> by far a very good way to clean welds and other heat induced decolourisations in the metal. It will remove a thin layer of metal, scale, oils, etc. Care must be taken to ensure any chemicals used are neutralised and thoroughly cleaned away. Residues cannot be tolerated. Makes general maintenance of the surface easier.
- <u>Passivating</u> a form of chemical polishing and surface protection. Can be stimulated with an electric current. Passivating is the spontaneous formation of a hard non-reactive surface film that inhibits further corrosion. It isn't a preferred method of surface preparation in vacuum systems as damaged surfaces can form micro cracks trapping water and other gases. For example, damage to anodised aluminium may allow aluminium oxide to form which can readily trap water. Passivation is not typically used for stainless steel as the surfaces are self healing, provided sufficient oxygen is available.

Further reading on surface passivation can be found at: http://www.euro-inox.org/pdf/map/Passivating\_Pickling\_EN.pdfGetter surfaces

• Nickel Plating – has been used on vacuum components to provide stability to the surface of normally oxidising metals such as steel, copper and brass. It is important to ensure that the nickel has uniform bonding to the parent metal. Cases have been observed where the plating has lifted creating voids in the vacuum space. Although nickel is resistant to corrosion it will form a thin layer if exposed to moist atmospheres. A periodic clean is recommended to minimise problems with corrosion.

Element	Melting Point °C	Critical Temp °C	Phase at Critical T
Aluminium	660	1124	Liquid
Americium	1176	896	Solid
Antimony	631	455	Solid
Arsenic	84	270	Liquid
Barium	729	574	Solid
Beryllium	1289	1102	Solid
Cadmium	321	226	Solid
Caesium	29	114	Liquid
Calcium	847	538	Solid
Californium	900	1402	Liquid
Cerium	798	1602	Liquid
Chromium	1863	1220	Solid
Cobalt	1492	1418	Solid
Copper	1084	1146	Liquid
Dysprosium	1412	1025	Solid
Erbium	1529	1139	Solid
Europium	822	547	Solid
Gadolinium	1312	1450	Liquid
Gallium	30	954	Liquid
Germanium	938	1260	Liquid
Gold	1064	1291	Liquid
Hafnium	2231	2255	Liquid
Holmium	1474	1146	Solid
indium	157	836	Liquid
Iridium	2447	1199	Solid
Iron	1538	1253	Solid
Lanthanum	918	1586	Liquid
Lead	328	622	Liquid
Lithium	181	465	Liquid
Lutetium	1663	1517	Solid
Magnesium	651	386	Solid
Manganese	1246	887	Solid
Mercury	-39	22	Liquid
Molybdenum	2623	2319	Solid
Neodymium	1021	1219	Liquid
Neptunium	639	1781	Liquid
Nickel	1455	1409	Solid
Niobium	2468	2501	Liquid
Osmium	3033	2721	Solid
Palladium	1555	1348	Solid
Platinum	1769	1800	Liquid
Plutonium	640	1506	Liquid
Potassium	64	165	Liquid
Praseodymium	931	1381	Liquid
Protactinium	1572	2636	Liquid
Rhenium	3186	2817	Solid
Rhodium	1963	1874	Solid
Rubidium	39	129	Liquid

# **Appendix 5 – Critical Vapour Pressures**

Substance	Vapour Pressure (Pa)	Temperature (°C)
Acetaldehyde	98.7	20
Acetone	24640	20
Butane	22	20
Carbonyl sulphide	1255	25
Ethanol	5.83	20
Ethylene glycol	0.5	20
Formaldehyde	435.7	20
Freon 113	37.9	20
Methanol	12800	20
Methyl isobutyl ketone	26.48	25
Nitrogen (N <sub>2</sub> )	63200	20
Oxygen (O <sub>2</sub> )	54200	20
Propane	2200	55
Propanol	2.4	20
Tungsten	0.1	3203
Water (H <sub>2</sub> O)	2.3	20
Xenon difluoride	0.6	25

# **Appendix 5 – Vapour Pressures continued**

Substance	Vapour Pressure (Pa)	Melting point (°C)
Apiezon Oil J	0.13 at 200°C 1.3 x 10 <sup>-4</sup> at 20°C	-
Apiezon Oil K	0.13 at 300°C 1.3 x 10 <sup>-7</sup> to 10 <sup>-8</sup> at 20°C	-
Apiezon Grease L	0.13 at 100°C 1.3 x 10 <sup>-7</sup> to 10 <sup>-9</sup> at 20°C	47
Apiezon Grease M	0.13 at 200°C 1.3 x 10 <sup>-5</sup> to 10 <sup>-6</sup> at 20°C	44
Apiezon Grease N	0.13 at 200°C 1.3 x 10 <sup>-6</sup> to 10 <sup>-7</sup> at 20°C	43
Apiezon Grease T	About 1.3 x 10 <sup>-6</sup> at 20°C	125
High Vacuum Grease Dow Corning	< 1 x 10 <sup>-4</sup> at 20°C	-
Nylon	~ 1 x 10 <sup>-3</sup> at 20°C	-
Teflon	< 1 x 10 <sup>-4</sup> at 20°C	-
Viton	1 x 10 <sup>-6</sup> at 20°C	-

# Appendix 6 – Outgassing tables for various materials Reference site: http://home.fnal.gov/~mlwong/outgas\_rev.htm

					i sampies.	
Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	(hours)	Test method	Reference	Year
Aluminium	None	1x10 <sup>-6</sup>	1h		Schamus (ref Markley, et al)	1999
Aluminium	Degassed	$1.7 \times 10^{-7}$	1h		Schmaus (ref Markley, et al)	1999
Aluminium	Degassed	2.7x10 <sup>-8</sup>	10h		Schmaus (ref Markley, et al)	1999
Aluminium 6061-T6	Baked 13.5h @ 300°C	1.4x10 <sup>-8</sup>	10h		Schmaus (ref Das)	1999
Aluminium	Cleaned	8x10 <sup>-9</sup>	10h		Schmaus (ref Blears, et al)	1999
Aluminium	Fresh, degreased w/ trichloroethylene & cleaned w/ ethyl alcohol	6.3x10 <sup>-9</sup>	1h	conductance	Elsey (ref Schram)	1975 (1963)
Aluminium	Degassed	$4.6 \times 10^{-9}$	100h		Schmaus (ref Markley, et al)	1999
Aluminium 6061-T6	Baked @ 200°C	4.5x10 <sup>-9</sup>	10h		Schmaus (ref Das)	1999
Aluminium	Degassed 24h, degreased w/ trichloroethylene & cleaned w/ ethyl alcohol	4.14x10 <sup>-9</sup>	1h	conductance	Elsey (ref Schram)	1975 (1963)
Aluminium 6061-T6	None	2.5x10 <sup>-9</sup>	10h		Schmaus (ref Das)	1999
Aluminium 5083-O	Bell jar, as received, room temp.	2.18x10 <sup>-9</sup>	10.3h	Rate-of-rise	Schrank, et al	1964
Aluminium 5083-O	Bell jar, as received, room temp.	1.27x10 <sup>-9</sup>	8h	Conductance	Schrank, et al	1964
Aluminium 5083-O	Bell jar, as received, room temp.	6.6x10 <sup>-10</sup>	24h	Conductance	Schrank, et al	1964
Aluminium 5083-O	Bell jar, as received, room temp. after baking @ 220°C	$4.6 \times 10^{-10}$	50h (baked @27-31h)	Conductance	Schrank, et al	1964
Aluminium 6061-T6	Baked 15h @ 300°C	$1.6 \times 10^{-10}$	10h		Schmaus (ref Das)	1999
Aluminium	Degassed 24h, degreased w/ trichloroethylene & cleaned w/ ethyl alcohol	3.06x10 <sup>-10</sup>	1h	conductance	Elsey (ref Schram)	1975 (1963)
Aluminium 5083-O	Bell jar, no. 4 finish (lapped), room temp.	$2.87 \times 10^{-10}$	6.0h	Rate-of-rise	Schrank, et al	1964
Aluminium	Fresh, degreased w/ trichloroethylene & cleaned w/ ethyl alcohol	6.0x10 <sup>-10</sup>	10h	conductance	Elsey (ref Schram)	1975 (1963)
Aluminium, type 1100	Cleaned w/ detergent, rinsed w/ acetone, pumped 24 hours	~10 <sup>-10</sup>	0	conductance	Young	1968
Aluminium	LEP vacuum chamber, chem clean, baked in situ @ 150°C	$2.3 \times 10^{-11}$	24h		Mathewson, et al	1988
Aluminium 6061-T4	Degreased in acetone w/ methanol rinse; baked 100°C	6x10 <sup>-12</sup>	24h	Rate-of-rise & conductance	Halama, Herrera	1976

Outgassing rates of aluminium Note the different methods of measurement and treatment of samples.

Aluminium 6061-T4	Degreased in acetone w/ methanol rinse, chem polished, baked 100°C	3x10 <sup>-12</sup>	24h	Rate-of-rise & conductance	Halama, Herrera	1976
Aluminium 6061-T4	Degreased in acetone w/ methanol rinse, chem polished, glow discharge in Ar, baked 100°C	5x10 <sup>-13</sup>	24h	Rate-of-rise & conductance	Halama, Herrera	1976
Aluminium, type 1100	Above plus baked 15h @ 250°C under vacuum	$4x10^{-13}$	24h @ room temp	conductance	Young	1968
Aluminium	PETRA vacuum chamber, glow discharge@145°C	$1 \times 10^{-13}$	Up to 200h	In situ glow discharge, conductance	Mathewson, et al	1977

# Outgassing rates of stainless steels, along with the surface treatment and pumping time.

Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	Time (hours)	Test method	Reference	Year
Stainless steel	None	6.4x10 <sup>-7</sup>	1h		Schamus (ref Markley, et al)	1999
Stainless steel	degreased	4x10 <sup>-7</sup>	1h		Schamus (ref Markley, et al)	1999
Stainless steel	None	2x10 <sup>-7</sup>	1h		Schamus (ref Blears, et al)	1999
Stainless steel	None	2x10 <sup>-8</sup>	10h		Schamus (ref Blears, et al)	1999
Stainless steel NS22S	Fresh	1.4x10 <sup>-8</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel ICN 472	fresh	1.3x10 <sup>-8</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel	None	1.2x10 <sup>-8</sup>	10h		Schamus (ref Das, et al)	1999
Stainless steel ICN 472	sanded	8.3x10 <sup>-9</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel NS22S	electro polished	4.3x10 <sup>-9</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel NS22S	mechanically polished	1.7x10 <sup>-9</sup>	1h	Conductance	Elsey (ref Schram)	1975
Stainless steel	fresh	1.5x10 <sup>-9</sup>	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel ICN 472	Polished & vapor degreased	1.4x10 <sup>-9</sup>	10h		Schamus (ref Dayton, et al)	1999
Stainless steel	None	1.4x10 <sup>-9</sup>	10h		Schamus (ref Das, et al)	1999
Stainless steel NS22S	fresh	1.3x10 <sup>-9</sup>	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel ICN 472	sanded	1.0x10 <sup>-9</sup>	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel	None	7.6x10 <sup>-10</sup>	1h		Schamus (ref Das, et al)	1999
Stainless steel NS22S	mechanically polished	$4.6 \times 10^{-10}$	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel NS22S	electro polished	$4.3 \times 10^{-10}$	10h	Conductance	Elsey (ref Schram)	1975
Stainless steel	Baked 24h @ 200°C	$1.5 \times 10^{-10}$	1h		Schamus (ref Das, et al)	1999

Stainless steel	None	$1.1 \mathrm{x} 10^{-10}$	100h		Schamus (ref Das, et al)	1999
Stainless steel 304	Degrease + water rinse	$4.0 \times 10^{-11}$	40h	Conductance	Strausser	1973
Stainless steel U15C	Baked 25h @ 300°C	$4.5 \times 10^{-12}$			Elsey (ref Calder, et al)	1975
Stainless steel 304	Degrease + water rinse, baked in vacuum 150°C for 12h	4.0x10 <sup>-12</sup>	5h after bakeout	Conductance	Strausser	1967
Stainless steel 304	Electropolished, baked 30h @ 250°C	$3.0 \times 10^{-12}$			Elsey (ref Young)	1975
Stainless steel U15C	Baked 45h @ 360°C	$2.6 \times 10^{-12}$			Elsey (ref Calder, et al)	1975
Stainless steel	Baked 24h @ 200°C	$9.3 \times 10^{-13}$	100h		Schamus (ref Das, et al)	1999
Stainless steel U15C	Baked 3h in vacuum @ 1000°C + 25h in situ @ 360°C	1.6x10 <sup>-14</sup>			Elsey (ref Calder, et al)	1975

Other cleaning methods although outgassing rates are not measured:

Material	Treatment	Results	Reference	Year
Aluminium	Quench a hot aluminium extrusion in an Ar- $O_2$ atmosphere	Dense, thin (~20Å thick) oxide layer	Sasaki, Y.T.	1990
Aluminium 6063	Clean with an alkaline detergent (Almeco 18)	Removes MgO, C, and Al <sub>2</sub> O <sub>3</sub>	Sasaki, Y.T.	1990
Aluminium	Vapour degreasing, pump for 120h before and after bake	Dominant residual gases: H <sub>2</sub> O, H <sub>2</sub> , CO, CO <sub>2</sub> , & CH <sub>4</sub> ; after baking @ 150°C and pumping, H <sub>2</sub> O & CH <sub>4</sub> gone, outgassing rates of other gases reduced 1 order of magnitude except H <sub>2</sub>	Mathewson, A.G., et al	1989
Aluminium	Vapour degreasing, then light alkaline etch (CERN basic procedure), pump for 120h before and after bake	Dominant residual gases: H <sub>2</sub> O, H <sub>2</sub> , CO, & CO <sub>2</sub> ; after baking @ 150°C and pumping, only H <sub>2</sub> exists	Mathewson A.G., et al	1989
Aluminium	Strong alkaline etching in NaOH, pump for 120h before and after bake	Dominant residual gases: H <sub>2</sub> O, H <sub>2</sub> , CO, CO <sub>2</sub> , & CH <sub>4</sub> ; after baking @ 150°C and pumping, only H <sub>2</sub> exists	Mathewson A.G., et al	1989
Aluminium	Vapour degreasing, Ar glow discharge @ 150°C	On initiation of discharge, large pressure increase (normal in Ar glow discharge) was too large for accurate measurement	Mathewson A.G., et al	1989
Aluminium	Vapour degreasing, then light alkaline etch (CERN basic procedure), Ar glow discharge @ 150°C	H <sub>2</sub> , CO dominant gases desorbing from surface during discharge cleaning	Mathewson A.G., et al	1989
Aluminium	Strong alkaline etching in NaOH, Ar glow discharge @ 150°C	H <sub>2</sub> , CO dominant gases desorbing from surface during discharge cleaning at order of magnitude less than light alkaline etch	Mathewson A.G., et al	1989
SS	H <sub>2</sub> glow discharge - 22°C	Dominant residual gases: $CH_4$ , $CO$ , & $C_2H_4$ ; water production insignificant; frequent arcing	Dylla, H.F.	1988

		on all exposed surface noted during initial discharge period		
SS	H <sub>2</sub> glow discharge – 150°C	Dominant residual gases: $CH_4$ , $CO$ , & $C_2H_4$ , and $H_2O$ ; frequent arcing on all exposed surface noted during initial discharge period	Dylla, H.F.	1988
SS	Ar glow discharge – ambient temp	Dominant residual gases that were significantly removed: $CO_2$ , $H_2$	Dylla, H.F.	1988
SS	Ar/O <sub>2</sub> glow discharge	Improved removal of CO and CO2; minor amts of implanted Ar that can be removed by baking @ 350°C	Dylla, H.F.	1988
	Ar/O <sub>2</sub> glow discharge 2h, exposed to air, then Ar/O <sub>2</sub> glow discharge 2h	Some degree of passivation: residual gases were removed with 1 <sup>st</sup> cleaning & did not show up during the 2 <sup>nd</sup> cleaning	Dylla, H.F.	1988
	O <sub>2</sub> glow discharge	Rapid removal of carbon, not of hydrocarbons (which can be baked out initially), increased surf. Oxidation, sputtering of the base metal, and possible enhanced outgassing of O <sub>2</sub> - containing molec.	Dylla, H.F.	1988

### Different outgassing measurements of epoxies

Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	Time (hours)	Test method	Reference	Year
NMA w/ solid alumina filler	Cured 300°F	$1.6 \times 10^{-7}$			Hanson, Patel	1970
Polyimide polymer	Thermosetting; measured @ 40°C	~1.1x10 <sup>-7</sup>	4h+9min	conductance	Kendall, Zabielski	1965
Polyimide polymer	Thermosetting; measured @ 155°C	~5x10 <sup>-8</sup>	4.5h increase temp + 6h	conductance	Kendall, Zabielski	1965
Silicone resin	Needs dicumyl peroxide catalyst; thermosetting, measured @ 35°C	~5x10 <sup>-8</sup>	4h	conductance	Kendall, Zabielski	1965
Araldite epoxies	Cured ~150°C for 15h	~10 <sup>-8</sup>	51h pumping	conductance	Barton, Govier	1965

# Kapton is a newer material and little data exists about its outgassing rate. Below are 3 references

Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	Time (hours)	Test method	Reference	Year
Kapton cable		1.0x10 <sup>-5</sup>	0h	conductance	Postma	1999
Kapton foil		$1.0 \times 10^{-7}$	40h		Ferro-Luzzi	1999
Polyimide	Bake @ 300°C	$4.0 \mathrm{x} 10^{-8}$	12h		Weston	1970
Kapton cable		$2.4 \times 10^{-8}$	168h	conductance	Postma	1999
Kapton cable		2.7x10 <sup>-9</sup>	336h	conductance	Postma	1999
Kapton cable		$6.5 \times 10^{-10}$	504h	conductance	Postma	1999

### Outgassing rates of several types of plastics

Material	Treatment	Outgassing rate (torr-L/sec-cm <sup>2</sup> )	Time (hours)	Test method	Reference	Year
Nylon		$1.2 \times 10^{-5}$	1h		Elsey (ref	1975
					Power, et al)	
Nylon		6.0x10 <sup>-6</sup>	1h		Elsey (ref	1975

					Power, et al)	
PVC	24h @ 95% relative	8.5x10 <sup>-7</sup>	1h	conductance	Elsey (ref	1975
	humidity				Santler)	
PTFE	Fresh	$1.7 \mathrm{x} 10^{-7}$	1h	conductance	Elsey (ref	1975
					Santler)	
Teflon		6.5x10 <sup>-8</sup>	1h	conductance	Elsey (ref	1975
					Santler)	
PTFE	Fresh	3.3x10 <sup>-8</sup>	10h	conductance	Elsey (ref	1975
					Santler)	
Teflon		$2.5 \times 10^{-8}$	10h	conductance	Elsey (ref	1975
					Santler)	
PVC	24h @ 95% relative	$2.0 \times 10^{-8}$	10h	conductance	Elsey (ref	1975
	humidity				Santler)	
G-10		~10 <sup>-8</sup>			Beams	2001
					Division -	
					FNAL	

Reference to the following has been lost but it is believed to have originated in an early Vacuum Catalogue from the 1990's

 $K_1$  is the outgassing rate (air equivalent) after 1 hour pumping  $\alpha_1$  is the slope of the (log K - log t) curve at 1 hour

Material	K <sub>1</sub> Torr litre/sec cm <sup>2</sup> (x 10 <sup>-10</sup> )	α1	K <sub>10</sub> Torr litre/sec cm <sup>2</sup> (x 10 <sup>-10</sup> )	a <sub>10</sub>
Aluminium Alloy (fresh)	63	1	6.0	1
Aluminium Alloy (degassed 24 hours)	41.4	3.2	3.06	0.9
Aluminium Alloy (3 hours in air)	65.5	1.9	4.75	0.9
Aluminium Alloy (anodised -2µ pores)	2760	0.9	322	0.9
Aluminium Alloy (bright rolled)	-	-	75	1
Duralumin	1700	0.75	350	0.75
Brass (wave guide)	4000	2.0	100	1.2
Copper (fresh)	400	1	41.5	1
Copper (mechanical polish)	35	1	3.56	1
OFHC Copper (fresh)	118	1.3	12.6	1.3
OFHC Copper (mechanical polish)	19	1.1	1.63	1.1
Gold (fresh wire)	1580	2.1	5.1	1
Mild steel	5400	1	500	1
Mild steel (slightly rusty)	6000	3.1	130	1
Cr plated steel (fresh)	70.5	1	6.8	1
Cr plated steel (polished)	91	1	8.0	1
Ni plated steel (fresh)	42.4	0.9	4.84	0.9
Ni plated steel	27.6	1.1	2.33	1.1
Chemically Ni plated steel (fresh)	83	1	7.05	1
Chemically Ni plated steel (polished)	52.2	1	4.6	1
• • • • •				

Araldite (moulded)	116	0.8	35.2	0.8
Araldite D	800	0.8	220	0.78
Araldite D	190	0.3	125	0.5
Araldite F	150	0.5	73	0.5
Celluloid	860	0.5	430	0.5
(PTFE) (fresh)	16.6	0.8	3.31	0.9
Kel-F Oak ridge	4	0.57	1.7	0.53
Methyl methacrylate	420	0.9	140	0.57
Mylar V-200 (24 hr at 95% RH)	230	0.75	40	-
Nylon	1200	0.5	600	0.5
Pertinax	620	0.18	290	0.5
Perspex	72	0.44	27	0.44
Perspex	310	0.4	180	0.4
Polyamid	460	0.5	230	0.5
Polyester - glass laminate	250	0.84	80	0.81
Polyethylene	23	0.5	11.5	0.5
Polystyrene	2000	1.6	200	1.6
Polystyrol	56	0.6	12	0.61
Polyvinylcarbazol	160	0.5	80	0.5
PTFE	30	0.45	15	0.56
P.V.C. (24 hr at 95% RH)	85	1.00	2	-
Teflon	6.5	0.6	2.5	0.2
Terephenil (fresh)	62.2	0.5	16.8	0.5
Neoprene	3000	0.4	-	-
Viton	114	0.8	-	-
	1	1		

# **Appendix 7 – Local Plant and Safety Regulations**

Regular maintenance inspections are carried out on ANTARES and STAR accelerator vacuum equipment to ensure operational status, vacuum quality, vacuum pump performance, and the mechanical and electrical integrity of the systems.

Generally, the integrity of the vacuum systems can be measured as a direct function of the known static vacuum pressures in a given vacuum system. That is, a lowering of vacuum from the known average vacuum pressure in a given system will indicate either/and, a leak in a seal, a failing vacuum pumping system or a structural failure.

The following is an overview of the application of the ANSTO OHSE Plant and Safety Standard, AS 2309, to vacuum systems in the accelerator facilities. *Reference: http://docushare.ansto.gov.au/Get/File-32340/Plant\_Safety\_Standard.pdf* 

Risk	Hazard	Risk rating	Controls	Risk rating (with controls in place)
Catastrophic failure vacuum vessels	Implosion – scattering of material fragments and loose fittings becoming missiles	Very low	<ul> <li>Use of vacuum standards to design equipment for safe usage.</li> <li>Protective barriers around equipment and fittings that may be susceptible to knocking</li> </ul>	Very low
Catastrophic failure vacuum beam lines and associated equipment	• Implosion – scattering of material fragments and loose fittings becoming missiles	Very low	<ul> <li>Use of vacuum standards to design equipment for safe usage.</li> <li>Purchased equipment from reputable companies/designers only</li> </ul>	Very low
	• Electric shock from fault in high vacuum gauge	Very low	• Vacuum gauges using high voltages in key areas will automatically switch off at low vacuums/atmospheric pressure	Very low
	• Failure of welded joint	Very low	• System will leak to atmospheric pressure at a rate commensurate with the conductance of the failed joint. No control is deemed necessary.	Very low
Electric shock from a failed vacuum pump or powered fitting	Electric shock from frayed lead or failure of earth inside of equipment	Low	<ul> <li>All equipment is protected from short circuits by circuit breaker protection.</li> <li>Most circuits now have RCD protection.</li> <li>A program is in place to put all circuits onto RCD protection</li> </ul>	Very Low
Failure of support structures	Crush, fall, bump, trip hazards during and after failure	Low	<ul> <li>Use of vacuum standards to design equipment for safe usage</li> <li>Access to equipment at height via approved ladders or dedicated platforms</li> <li>Non acceptance of standing on beam line stands and mounts</li> </ul>	Very Low
breakages to	<ul> <li>Fragments of broken</li> </ul>	LOW	<ul> <li>Barriers around exposed equipment</li> </ul>	LOW

1) Risk assessment of plant – Accelerator Facilities Only

fittings and equipment protruding from vessels and beam lines	<ul> <li>parts become missiles.</li> <li>Cuts to personnel bumping into fittings</li> <li>Secondary injuries including electric shock from exposed wiring</li> </ul>		<ul> <li>that is deemed to be hazardous</li> <li>Access control into accelerator areas restricted to accelerator personnel and visitors who are escorted</li> <li>See electric shock hazard controls above</li> </ul>	
Over pressure of vacuum systems during venting	Positive pressure in beam lines when venting with gases from high pressure storage	Very Low	<ul> <li>Pressure relief valves in use at systems where regular vacuum cycling is necessary</li> <li>Use of portable pressure relief fittings where local venting is required.</li> </ul>	Very Low
Implosion of glass view ports	Shards of glass scattering towards operators	Low	<ul> <li>Use purpose designed commercial vacuum windows</li> <li>Use small diameter (&lt;50mm x 6 mm thick) quartz windows on vacuum systems where ion beams can strike. Quartz is not known to crack through but to develop small surface anomalies</li> <li>Use double rubber seals to support windows to minimise mechanical stresses</li> </ul>	Low
Broken film windows	Sudden venting of vacuum to atmospheric pressure	Very Low	<ul> <li>Damage to equipment – high vacuum gauges must have auto shutdown at low vacuums</li> <li>Vacuum system isolated with gate valve to minimise loss of vacuum in adjoining systems</li> </ul>	Very Low
Breach of accelerator tubes with SF6	Vacuum systems pressurised with SF6 gas	Low	<ul> <li>Large gate valves either end of the accelerator vacuum tubes will shut if pressure rises to maintain SF6 within the vacuum vessels</li> <li>Some gas will enter the vacuum systems outside of the isolated section. The pressure will be relieved through blow off venting ports at each end of the vessel.</li> <li>Oxygen depletion alarms will sound if dangerous levels of SF6 are released. Personnel will be ordered to leave building through PA announcement</li> </ul>	Low

- 2) Risk control measures for eliminating or reducing the assessed risks *See table above*
- 3) Registration or notification of design of plant *Not deemed necessary. See AS 4343-2005*
- 4) Licensing of plant Not deemed necessary. See AS 4343-2005
- 5) Maintenance of plant

*Regular maintenance applied via Accelerator Maintenance system. See ENV-I-076-004 Vacuum Systems Maintenance manual* 

- 6) Plant operations and risk controls *See table above*
- 7) Requirements in relation to specific types of plant *Only vacuum system plant*
- 8) Training and supervision Vacuum technology training is provided in-house to world's best practices. Additional training has been provided by the Vacuum Society of Australia through their training courses.
- 9) Monitoring and evaluation Regular maintenance applied via Accelerator Maintenance system. See ENV-I-076-004 Vacuum Systems Maintenance manual

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# Vacuum Technology

# and

# Vacuum Design Handbook

# for

# **Accelerator Technicians**

Prepared by David Garton November 2011 Revision 0 Vacuum Technology and Vacuum Design Handbook for Accelerator Technicians

This handbook is a compilation of information gathered from over 50 years of direct hands-on experience to applicable information widely available from the vacuum technology industry. It seeks to address common and specific vacuum technology problems whilst clarifying the design standards and philosophies adopted for use in the ANSTO accelerator facilities. The author wishes to thank the reviewers and the many technicians from ANSTO that have contributed directly and indirectly to this booklet.

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### Chapter 1

### Vacuum design standards and good practises for the Accelerator Area

- \* This chapter must be read in conjunction with the detailed vacuum information in this document.
- \* Based on best practises developed at ANSTO and other local Australian accelerator laboratories in conjunction with Australian Standards and industry technical guides, referenced throughout this document.
- \* Where a standard is unavailable for a specific design, best practices are used from industry's best practises.
- \* *Refers to all vacuum chambers, beam lines and other enclosures used on the accelerators or stand alone.*

#### 1. Basic design

- All vacuum vessel designs must be consistent with the design standards in Australian Standard AS 1210-2010 and maintain a Hazard Level of E as per the design conditions tabled in "Hazard Levels of Pressure Equipment" in Section 2, Hazard Levels of Pressure Equipment in Australian Standard AS 4343-2005.
  - The vacuum equipment designed by/for the accelerator area maintains a Hazard level of E. All pressure equipment with a pV  $\leq$  30, pressure (p in MPa) x volume (litres)  $\leq$  30 is classed as Hazard level of E. For vacuum equipment in Hazard Level E the derating value of 0.1 is applied, ie 0.1pV  $\leq$ 30. Note the amount of stored energy at this level is very small. For example a Ø 1000 mm chamber, 1000 mm high will have a pV for vacuum equal to 7.85.
  - AS 4343-2005, Section 2.1.2 Typical hazard levels, part (d), states for Hazard Level E, "This equipment is usually exempt from special regulatory control but is <u>covered</u> by general plant safety regulations". See Appendix 7.
- All designs of chambers excluding general beam lines and fittings should be checked for structural integrity using finite elements analysis. Sound designs with acceptable structural integrity will be released for manufacture. A copy of the design acceptance tests must be filed with the project file. The standard finite element analysis can be performed utilising Solidworks. (Modelling for irregular shaped chambers, calculations Appendix 1 where appropriate for regular shapes)
- Stainless steel (304 or 316) will be used to construct chambers, beam lines and general fittings unless it is a *special* requirement to use other metals. Do not choose substances that have high outgassing rates or can contaminate vacuum systems.
- Measurement chambers and other large volume chambers should be cylindrical unless the chamber is an enclosure between pole faces (eg magnet box) or is an odd shape to accommodate steering plates or devices. When non-cylindrical chambers are required deflection of chamber walls should be modelled and taken into consideration.
- Other chambers including magnet boxes, ESA and deflector coffins to have suitable rib strengthened rectangular or curved sides where it is not practicable to increase the wall thicknesses.
- Minimum wall thickness calculation methods can be found in Appendix 1. As a general rule care must be taken when using formulas for vacuum calculations as historically many units other than SI units have been used to form some equations. For example length in cm, and pressure in torr is common in old notes.

- Top and bottom flanges to have integrated structural support rim which becomes the flange and seal for chamber base and top (user) flanges.
- Ports must be a suitable length to ensure unobstructed insertion for screws into flanges. Suitable weld preparation must allow for adequate weld penetration. See Welds below.
- All internal edges must have a smooth radius. No sharp edges to be left after welding or surface finishing.
- All machined finishes other than vacuum seals to be equal to or better than 1.6 microns
- No sections within the vacuum space will have metal to metal contact where gas can be trapped unless it is at the edge of a seal joint. When this is necessary, pump out grooves must be incorporated in the design.
- Vacuum windows shall be made from toughened glass or quartz. No plastic to be used for windows unless the window is a flange on a chamber that is designed to withstand more than 100 kPa. The large plastic flange/windows should be made from >18 mm thick up to Ø200 and >25 mm thick for up to Ø400 in clear Perspex or polycarbonate.
- For ANTARES beam lines the preferred flanges are Dependex, nominal diameter 100 mm. See section 8 for detail on Dependex flanges.
- For STAR there is no preferred flange type. It must be selected depending on the desired base vacuum and interface to other adjoining flanges.
- As a guide, the MDC catalogue from Vacuum Products Corporation 2003 onwards, Building Blocks for Vacuum Science and Technology, provides vacuum tubing dimensions for most common sizes. The dimensions must be assumed to be minimum dimensions. Other tubing sizes should be calculated using the formulas in Appendix 1.
- Vacuum pumping ports leading to the vacuum pumps must be designed to optimise conductance.
- Pressure relief valves to be incorporated where a vacuum system can be vented to >10 kPa above atmospheric pressure.
- ASME state that the design of an external pressure vessel must consider Material type, diameter of chamber, unstiffened length, temperature and wall thickness

#### 2. Welds

- TIG is preferred for vacuum sealing welds as minimal scale and flux is produced. Electron beam welding is suitable for thin walled materials. TIG produces high quality welds with or without filler rods to produce flat, smooth well penetrated welds. TIG comes into its own where the weld preparation allows for melting together of the parent materials without filler rods. MIG has a higher deposition of filler material so care must be taken to ensure welds are kept smooth and flat.
- No standard exists for vacuum welding of compact scientific vacuum equipment where pressures approaching zero are necessary. Equipment designed in the accelerator area and welded in the ANSTO workshops since 1990 has had penetrations of ≥1mm but ≤2mm on a butt weld and ≥2 mm on a fillet weld. Experience has demonstrated the adequacy of these figures. No failure reported.
- For thin walled materials ie < 1 mm, such as bellows, manufacturers' will use their industry standard. ANSTO does not weld thin wall materials.
- Weld preparation must be provided where possible to ensure even heat penetration.

- Electric arc with rods is not recommended due to flux trapping. This can cause pits which have long lasting outgassing periods.
- All welds that form a seal against atmospheric or other positive pressures must be made to the internal side (vacuum side) of the joint.
- Tack (stitch) welding is recommended on the external side only with less than 50% of the diameter covered in small intervals. This minimises the chance of trapped voids and facilitates He leak testing post welding.
- All welds must be free from pitting and scale. Must be cleaned to bare metal. If chemicals are used to clean welds care must be taken to remove all traces of chemicals.
- Some chambers such as magnet boxes will need to have some joints welded externally due to access problems on thin chambers. In this case, good penetration must be achieved to ensure minimal gas trapping points.

#### 3. Surface finishes

- The inside of the chamber and ports must be 1.6 micron or better for mechanical finishes.
- Can be chemically cleaned (polished) but all traces of chemicals must be neutralised and removed prior to use in vacuum.
- All machining oil to be removed with an alcohol scrub then acetone rinse, minimum. The use of kerosene, turpentine or other oily solvents is not encouraged however where they are used the final clean must be with acetone followed by alcohol. Water based cutting fluids can be used as they are more readily removed therefore having less effect on outgassing.
- Nitrile gloves to be used when assembling cleaned components. Other un-powdered gloves may be used but only those that do not react with the solvents being used.
- Care must be given to cleaning and handling surfaces that will be exposed to vacuum. Gloves should be used to minimise the transfer of dirt and oils from the skin to vacuum surfaces.
- A suitably clean area should be used for the preparation of vacuum systems and all tolls used should also be cleaned (degreased).
- Stainless steel can be cleaned in nitric acid to remove scale that remains from rolling or wire cutting. It is may also be used to prepare stainless steel components for use in ultra-high vacuum systems.

#### 4. Flanges

- Flanges must be standard flanges selected from the group referenced this document for compatibility reasons. Exceptions may be made but all 'o' ring seals must be to Australian Standard AS 2842-1986. External flange rings not in contact with the vacuum space may be aluminium alloy. Stainless flanges are not economical or necessary unless the seal requires forces to crush metal seals such as copper ConFlat, aluminium or indium wire.
- Minimum thicknesses for top and bottom flanges to be calculated using the standard in Appendix 1 Structural Calculations for Scientific Vacuum Vessel Design.
- All other flanges to have threads consistent with their design standard.
- ANTARES Primary flanges will be Dependex unless interface is necessary to accommodate other standard flanging.
- For Dependex, screws to be imperial sizes consistent with UNC series.

• STAR utilises many of the major vacuum flange/seal designs. The most appropriate design must be chosen that meets the specific application.

#### 5. Seals

- 'O' ring sizes and sealing groove dimensions must comply with the Australian Standard AS 2842-1986. Adjustments to groove sizes can be made where specific 'crush' is necessary for special applications.
- Viton rubber 'o' rings to be used unless a special requirement for an alternate elastomer is identified.
- Standard flange and seal sizes to be used unless an application prohibits their use.
- It is recommended that vacuum sealing surfaces are cleaned just before a seal is made to minimise the possibility of dust and hair settling on sealing surfaces.
- Metal seals may be used in specifically designed sealing surfaces including, aluminium and indium wires. They are very good as ultra-high vacuum seals.

#### 6. Screws

- All screws for flanges other than Conflat will be made from stainless steel in either 304 or 316 grades.
- Screws used in Conflat flanges will be made from A2-70 or A4-70.
- All screws to be socket head unless access necessitates hexagon or other special head. Slotted or Philips head must not be used.
- Stainless steel flat washers to be used under all screws where practicable. Do not use lock or star washers on standard flanges.
- In the vacuum space, no screws to be used in holes unless the holes are through holes and can be pumped from both sides. Screws can be used in blind holes if they have a minimum Ø1 mm hole drilled through the centre to allow trapped gas/air to escape.
- Unless a standard flange uses non metric thread, all screw threads must be within the metric series. Unless there is a specific need, avoid using intermediate metric sizes, that is, 0.5 mm steps such as M1.5 mm, M2.5 mm etc.
- Anti-seizing compounds can be used on threads but extreme care must be taken to ensure they do not enter the vacuum space. Graphite powder can be used as a lubricant inside the vacuum space. It can be applied using ethanol to wet the surface to be lubricated. Allow solvent to evaporate prior to pumping.

#### 7. Feedthroughs

- Sliding seals should have dual 'o' ring seals with a gland formed seal at least at one end. The shaft finish must be 0.8 micron or better.
- All sliding shafts must have retainers to stop them being pulled through into the vacuum space.
- All coax feedthroughs to be glass or ceramic hermetically sealed. No plastic.

#### 8. Lubricants, vacuum greases and epoxies

- Only vacuum specific grease and oils to be used in vacuum systems.
- Silicone based or particular Apiezon family lubricants design for vacuum use, are recommended on sliding seals. Thin smears only.
- No vacuum greases should be used unless there is a small leak through a damaged sealing surface and all other options are not feasible. If grease must be used it must be designed for high vacuum use and only a very thin smear to be used.
- Do not use grease to fill suspected leaks in welds or fittings.
- Where a vacuum epoxy must be used the surface must be well prepared to ensure appropriate bonding. Care must be taken to ensure the epoxy seal doesn't create a trapped void of air which may become an outgassing problem. 'Torr-Seal' is commonly used due to its relatively low vapour pressure, and thermally stable to 120°C. (this is a Varian product)
- Vacuum oils are not recommended for general lubrication as they can creep on surfaces spreading contamination and increasing the outgassing surface.
- See Appendix 3 section 1.5.

#### 9. Mounting

- The load of the chamber assembly must rest on a standard accelerator beam line table ANSTO, HVEC, HVEE or NEC or a purpose built stand that minimises vibration.
- Vibration decoupling will be achieved by supporting the chamber separately from the vacuum pump and using an edge welded bellows between the chamber and pump.
- For ANTARES beam line vacuum equipment the beam axis alignment is nominally 1750 mm from floor height.
- For STAR beam line vacuum equipment the beam axis alignment is nominally 1193 mm from floor height.

#### 10. Access

- The working face of the chamber must face an area that is easily and comfortably accessible. The floor space immediately in front of the working face should be raised using steps to facilitate access.
- Heavy or awkward shaped vacuum equipment must be designed to be lifted with the aid of over cranes for example lifting lugs.

#### 11. Testing

- Vacuum equipment must be tested prior to routine use to minimise time spent chasing leaks in completed systems.
- Vacuum vessels must be helium leak tested to ensure all seals are better than  $1 \ge 10^{-10}$  Pa.m<sup>3</sup>/s ( $1 \ge 10^{-9}$  mbar.l/s). As a standard, during a leak test there should be no deviation from this baseline level for a leak-tight system. A record of the test is to be noted on the engineering drawing showing, date, maximum leak rate, leak location/s, leak detector used and operator name.

- For all other vacuum equipment in-service testing may be necessary to allow complete operation of various feedthroughs and positioners.
- Caution must be taken before positive pressure testing vacuum equipment as the equipment may be irreversibly damaged. Warning Do not use water testing on any vacuum equipment.
- A Residual Gas Analyser (RGA) can be used to give more detailed information about the gas composition inside a vacuum volume. An RGA can be used as a substitute for a helium leak detector with the benefit of providing more information about outgassing, or virtual leaks inside the vacuum volume. The pressure of the vacuum system must be  $< 1 \times 10^{-2}$  Pa to prevent damage to the filament inside the instrument. Instruction manuals must be checked before using the RGA head to ensure safe and effective use.

#### 12. Operation and Continued Monitoring

- All chambers should have a high vacuum gauge sensing within the chamber space. This includes all adjoining compartments that may be isolated by a valve.
- The chamber must have valves attached that allow complete vacuum isolation from the beam lines and other equipment.
- To protect high vacuum gauges against the effects of operating in low vacuum, high vacuum gauges should be automatically isolated if the vacuum pressure rises above  $1 \times 10^{-2}$  Pa.
- High vacuum gauges must be cleaned at minimum every 3 years or whenever a gauge develops instability as compared with adjacent gauge readings.
- Systems indicating higher than normally observed pressure must be isolated and repaired. A helium leak test is recommended if the leak location is not clear.
- To avoid undesired contamination, vacuum chambers should be vented with dry nitrogen or argon instead of air. This minimises water vapour and oil from depositing on the vessel walls.

### Chapter 2

### Design Criteria, Information and Philosophies for Vacuum Systems used in the Accelerator Area

#### Overview

In an accelerator system ion beams are transported from ion sources through to end stations (or detectors) through vacuum space maintained inside of beam tubes (lines). The relatively low number of gas molecules in the vacuum ensures a higher mean free path for the accelerated ions and hence less loss of ions through collision which is crucial especially for AMS measurements when counting a very small number of ions. A high vacuum also minimises the formation of plasmas in an electric field at high voltages which can avalanche into spark breakdown damaging sensitive equipment.

The vacuum systems used in the accelerator area are designed to be fail-safe to protect people and equipment. A very high proportion of problems with vacuum systems relate to, seals leaking on first use, being incorrectly mounted or failing due to aging 'o' rings. Other problems, however less frequent, include mechanical damage to sealing surfaces or fatigued parts such as bellows. The problems are all characterised by a poor vacuum or a previously steady vacuum that begins degrading slowly as observed during the operation of the vacuum system over its life time. The remaining problems are due to poor welds or vacuum fittings breaking. Poor welds are usually found during the initial helium leak test. They are remedied by either removing and re-welding the section or using a commercial vacuum epoxy. The choice is dependent on whether the weld is on a structural component or just a seal. Broken vacuum fittings are replaced or repaired.

Within the accelerator area. physicists work directly with technicians who apply broad knowledge and experience in vacuum technology to develop their concepts for use in the accelerator environment. This mix has been successful for 50 years. However not all designs are completed in the accelerator area. There are cases where commercial projects call for a more production based design or designs of chambers with complex noncylindrical shapes. These may go to the ANSTO drawing office for completion or a specialist vacuum design company.



Fig 1. ANSTO designed and developed beam lines on the STAR accelerator in building 22

It is not possible and often not necessary to standardise the design requirements for all components used in the accelerator area so the following information is intended to help designers of vacuum equipment to comply with accelerator specific and Australian standards. As of 2011, there are no known commercial workshops in Australia who specialise in the production of vacuum vessels. Local representatives from vacuum suppliers can provide access to overseas specialist workshops.

### 1. Terminology

**ANTARES** – Australian National Tandem for Applied RESearch. The HVEC FN Tandem accelerator in B53.

Backing – To pump on the exhaust (back) of a high vacuum pump

Beam line – The evacuated tubes that carry the ion beams from the ion sources to the end stations

**Cryopump** – Cryogenic vacuum pump. Uses a helium cooling interface to the vacuum space to freeze gases.

**Dependex** – A type of flange which is the standard flange used on ANTARES and ANSTO built beam lines.

**Fore line** – The vacuum line, tube, pipe, whatever between the high vacuum pump and the backing pump

High vacuum – See details in Chapter 3, para 1.3

ISO - International Standards Organisation

KF – Klein (small) Flange

LF – Large Flange

Low Vacuum – See details in Chapter 3, para 1.3

Outgassing - the liberation of gaseous vapours from the surfaces within a vacuum space

Pa – Unit of pressure, Pascal. 1 Pa = 1 N/m<sup>2</sup> = 0.01 mBar = 7.5 mTorr = 1.45 x 10<sup>-4</sup> PSI

**Pump down = Pump out** – evacuating gas from a vacuum space

**Roughing or Roughing-out** – Usually used to describe the initial transfer of gas from atmospheric pressure to low vacuum.

STAR – Small Tandem for Accelerator Research. The HVEE Tandetron accelerator in B22.

Turbo – an abbreviation for turbomolecular vacuum pump

**Ultra-high vacuum –** See details in Chapter 3, para 1.3

Venting – the equalisation of negative pressure to atmospheric pressure in vacuum equipment

Vessel – generic for a vacuum chamber where an instrument is mounted or interfaced into the vacuum space.

#### 2. Standard Units

#### 2.1 General

Although metric standard is adopted across all ANSTO accelerator facilities it does not mean that all equipment is metric as the US built FN Tandem is mostly imperial, conversely, the Dutch built HVEE Tandetron is all metric. There is a combination of metric and imperial devices used throughout the facilities but designers must ensure that where practicable all new designs are metric.

#### 2.2 Threads

New equipment for use in the accelerator area, where practicable will be designed with metric standard threads.

• Contrary to the standard, on ANTARES there is a combination of threads in use. The accelerator high voltage generator and parts that have originated from the United States are

mostly imperial. Typically, UNC and some UNF. Most beam lines and endstations are metric but all Dependex and most Conflat flanges are imperial.

• ON STAR all threads are metric. ANSTO designed and built beam lines are metric except for the Dependex flanges which are UNC.

Care must be taken to identify threads and use the correct screws. Where practicable, metric threads are to be used other than on Dependex flanging.

#### 2.3 Pressure

All pressure indicating devices shall be in the SI unit, Pascal. Historically, Torr was used but this was replaced by Pa in the late 1980's. Most international accelerator labs use mbar. The conversion is as simple as multiplying mbar by 100 to get Pa. Pascal refers to a physical force of newtons per metre squared.

### 3. General Design Philosophies

There are many varieties of vacuum chambers and equipment in use in the accelerator area. Most have been designed and made at ANSTO. Examples include vacuum equipment measurement chambers where samples are measured, beam line transport components, ion source components or sensitive detectors, to name a few. Each has its own unique characteristics and vacuum design considerations.

In order to maintain a suitable mean free path for ion beams whether being transmitted from a source or scattered from a sample into a detection system, high to ultra-high vacuums are necessary. In a high vacuum the density of gas is low enough to minimise electron flow from surfaces at high voltages to surfaces at lesser potentials. It is therefore prudent to ensure designs of vacuum equipment that directly couple to the accelerator are designed for a minimum of high vacuum operation.

In every case the designers must ask the following questions:

- What shape? How big? Where will it fit? How will it be mounted? What level of vacuum?
- Will it be ridged enough and have low enough distortion to mount ion optical components?
- Is the design flexible enough to enable small scope changes in the future? Is it designed with consideration for reuse in another application when the project finishes?
- Does the design use vacuum technology best practises?
- Does it meet Australian and local standards where applicable?
- Is it a safe design?
- Is it achievable with resources available?
- Is it cost effective. Can we modify existing equipment to achieve a similar result?
- Does it have low environmental impact?

#### 3.1 The Shape

The first consideration is to define the actual use of the vacuum equipment relative to the experiment. Where possible cylindrical shapes should be considered first as they are stronger under vacuum and consequently being round, ports can be welded into the chamber aligned radially to the centre which ideally aligns instruments including detectors and cameras with the intersection point of the ion beam and the sample being measured.

Non-cylindrical chambers must be carefully designed to included adequate strengthen ribs to minimise distortion and hence misalignment of instruments.



Fig 2. A few examples of vacuum vessels used in the accelerator area

#### 3.2 The size

The size is dictated by the space needed inside of the vessel or equipment in order to meet the operational demands. In the case of measurement chambers too big may mean problems with the distance between samples and detection systems and too small may mean manual handling of samples may be inhibited. Large chambers may have to be certified as per AS 4343-2005, Hazard Levels of Pressure Equipment in Australian Standard.

#### 3.3 Location

It will be an agreement between the accelerator user groups as to where equipment and beam lines will be located. This agreement will endeavour to minimise conflicts in beam line space, usage, accessibility and resourcing.

#### 3.4 Mounting

In general equipment must be secured onto the floor unless it is designed as mobile. An accelerator compliant beam line table should be used with standard beam line supports. Vibration must be identified during the design stage and decoupling stages provided.

Insertion bellows must be provided in long sections of beam lines to enable compression of the beam lines to facilitate dismantling and to ensure minimal strain on vacuum joints.

Designs must consider trip, bump and fall hazards. Structures that support platforms or heavy equipment that may otherwise fall must not be reliant on the structure supporting the vacuum chambers and beam lines. For example, ladders, walkways, etc.

- For ANTARES beam line vacuum equipment the beam axis alignment is nominally 1750 mm from floor height.
- For STAR beam line vacuum equipment the beam axis alignment is nominally 1193 mm from floor height.

#### 3.5 Flexible design

It is prudent to ensure where practicable designs ensure some redundancy for feedthroughs, access ports and internal space. A major problem with early chambers was the lack of space allowed for inserting samples by hand or manually manipulating internal equipment and wiring. It is easier to design flexibility into the chamber or equipment at the design stage rather than having to dismantle it and have it reworked in the workshops later.

#### 3.6 Can it be made?

When designing any equipment seek advice from a qualified machinist. Often designs include machining that may be very expensive or not possible. Early in the design phase ensure that the resources are available to service the vacuum equipment. It is recommended to consider the following early in the design stage:

- Power
- Cooling water
- Air cooling
- Signal lines
- Control lines
- Add-on electronic and mechanical instrumentation

#### 3.7 Safe design

There are no cases that the author has been able to find of scientific vacuum chambers or equipment, like those used in accelerator facilities, failing and causing injury. Vacuum pressure is relatively low, -100 kPa, or about half the equivalent magnitude in a car tyre.

For safe designs the structure must meet the minimum dimensions as calculated in Appendix 1 - Structural Calculations for Scientific Vacuum Vessel Design.

Care must be taken with the design of the following:

- Glass view ports (most glass breakages are in the form of a crack which leak to atmospheric pressure quickly without the spread of shards)
- No internal sharp edges; in the case where a person must insert their hands blindly to access components.
- Venting limits pressure relief for positive pressure?
- Evacuation rate regulation; especially in the case where very thin material windows separate different pressure systems within a single vacuum space for example beryllium windows on x-ray detectors.
- Equipment mounts/stands; where an operator must climb up high to operate devices on the system
- If oil type vacuum pumps are used then mist filters must be fitted to the exhausts.

#### **3.8** Cost effective design

All *bells and whistles* may not be necessary even after considering some design redundancy for future changes. Where possible, designs should consider integrating components that are available off the shelf. Designing what has already been designed by someone else is wasting time and effort.

It may be cheaper to modify an existing chamber for use rather than start from the beginning. Many chambers used around the accelerator area have successfully had one or more lives.

#### **3.9** Design by numbers

This is a matter of ensuring structural integrity and being able to prove it. Likewise, designing the vacuum pumping system requires calculating the most appropriate size pump rather than going for the "biggest"!

There are formulas in Chapter 3 that allow must crucial calculations to be made from wall thicknesses to conductance. Good enough is not acceptable. Replication of previously designed equipment is acceptable.

#### **3.10** Australian Standards

Compliance is mandatory however there are few standards that are relevant to scientific vacuum vessels and equipment. The industry has developed its own standards for flange and seal designs which can be seen in the catalogues of most leading manufacturers of vacuum equipment. It is the responsibility of the designer to ensure compliance with all relevant Australian Standards. Chapter 1 discusses what standards are included.

#### 3.11 Environmental impact

Most vacuum systems used in the accelerator area today are designed to be oil free. That is, they do not use oils for lubrication of vacuum seals. There is however some oil vane and diffusion pumps still in use but the oils are responsibly disposed of through waste management at ANSTO. Some old diffusion pumps had been used with mercury as the pumping agent. Personnel must be cautious of using old diffusion pumps and their associated backing pumps as there may be residual mercury on surfaces and in the backing pump oil.

Solvents used for cleaning are used in small amounts and usually for wiping rather than immersing. Where baths are needed the solvents are reused when possible. The solvent bath used for degreasing, self recycles the solvent by design however there is a small loss of solvent over time into the atmosphere.

Where possible solvents used are selected with the environmental impact in mind.

#### 3.12 Manufacture

If ANSTO cannot provide the level of manufacture quality in-house then scientific vacuum equipment is best manufactured by companies that specialise in high tolerance machining and welding. A general metal fabricator will not suffice.

Attention must be paid to considering standard sized tooling during manufacture. Some companies will charge for the purchase of tooling that they consider to be non-standard.

It is imperative that the drawings for manufacture state all tolerances and qualities for the finished product.

On all design drawings, where the angle of the welded ports is crucial, ensure angular tolerances are clear and achievable. If necessary, specify the use of mandrels to hold ports in place during welding.

Some success has been made outsourcing the manufacture through vacuum companies to overseas manufacturers who specialise in high quality small runs.

#### 3.13 Testing

Testing the vacuum equipment or chamber is essential to establish compliance with the design and the all important ultimate base vacuum. Helium leak testing is the standard method used for chambers and most equipment however on some equipment in-service testing may be the only option. This is where add on vacuum equipment doesn't change the original base vacuum of the system it is connected to.

#### 3.14 Inspection

All parts should be cleaned prior to inspection. This doesn't need to be the final clean but enough to ensure all welds and surfaces can be clearly inspected. Welds must be checked for pits and alignment along the weld lines and seals must be checked for surface finishes. All crucial dimensional must be checked. It is recommended that any non-complying findings are photographed and documented in the project file. All non-complying parts should be reworked rather than *patched up*.

#### 3.15 Certification

As most vacuum vessels and equipment in the accelerator area fall within Hazard Level E of AS 4343-2005, certification is a local process. For large measurement vacuum vessels, results from the inspection and leak testing will be recorded on the engineering drawing and registered in the local accelerator register and/or ANSTO drawing register as appropriate.

#### 3.16 Documentation

For all large measurement chambers requiring any level of design calculations, design documents must be kept with project files.

#### 3.17 Maintaining

Vacuum chambers and beam lines do not require high levels of maintenance other than a general periodic clean and occasional seal replacement. Vacuum fittings and devices will have their own level of maintenance so access to these components must not be difficult.

The design must be easy to maintain especially moving parts, sliding or rotating seal and other parts in the vacuum space that may wear. Also vacuum pumping systems that may be mounted under or near the vacuum chambers. Generally any part that will require any level of routine maintenance must be accessible without the need for a major disassembly.

Vacuum pumping systems other than oil free systems, connected onto beam lines and chambers must include suitable oil traps between the backing and high vacuum pumps and where mist filters on all exhausts.

Some high vacuum gauges will require periodic cleaning. On ANTARES high vacuum cold cathode gauges are scheduled for cleaning every 12-18 months or max 3 years for difficult to service or less crucial instrument locations. Otherwise gauges will be cleaned on demand within the periods.

#### 4. Performance and Operations

#### 4.1 Ultimate Vacuum

The ultimate vacuum is the maximum vacuum or lowest pressure the vacuum equipment typically reached after 3-5 days of uninterrupted pumping. This is a benchmark value which is used to assess the long term quality of the vacuum system. It is sometimes referred to as the *base vacuum or static vacuum*.

Generally, vacuums in the beam lines and chambers are well within the high vacuum range. Vacuums lower than this range will indicate a higher than normal gas load, a failing seal or a pump malfunction. On the accelerators the two main types of high vacuum pumps are turbomolecular and cryogenic pumps. For a system designed for high vacuum or better with no leaks and low outgassing then vacuums better than  $4 \times 10^{-6}$  Pa should be readily achieved.
The ultimate vacuum is improved by the correct choice of materials, seal types, high vacuum pumps used cleanliness and correct operation. All fittings must be designed or selected deliberately for high to ultrahigh vacuum use. Surface preparation and cleanliness will contribute to improved vacuums. A finger print can outgas for long periods of time. A bigger (higher speed, higher ultimate vacuum) vacuum pump does not mean a better vacuum!

## 4.2 Working Vacuum

The working vacuum may be lower than the ultimate vacuum due to sample cycling (changing), introduced gas loads from adjoining systems or outgassing samples. A system must be designed to ensure the high vacuum pumping system has the optimum pumping speed and ultimate vacuum. Once a vacuum system reaches the "working vacuum" little operator intervention is necessary to maintain it but there are some important points regarding ongoing monitoring to ensure a failure is not immanent.

For sections of beam lines that are holding a static vacuum a cursory glance at the local vacuum gauge will indicate the stability of the vacuum. Generally, the (vacuum) pressure will change slightly throughout a day for example due to diurnal changes in ambient temperature and perceived changes due to ionisation gauges "wandering" as they become dirty.

In sections of beam lines that are in the vicinity of operating beam lines or near end stations the vacuum will be continually changing. Factors that influence these changes include increased gas loads from samples outgassing or surfaces that have just be exposed to high humidity, outgassing due to temperature changes within the vacuum space (including bake outs), outgassing from ion beams striking plastics, etc. Gas molecules don't have a preference to whether they travel towards or away from a pump during molecular flow so the source of gas can come from just about anywhere in the open system. These factors must be considered when monitoring vacuums throughout an entire system.

If a vacuum system's pressure rises slowly over many days or weeks if it is not related to the cryopump loading then it may be a seal failing. In this case a helium leak test is recommended.

A fundamental flaw in many vacuum system designs is the neglect for monitoring the backing pressure between the backing pumps and turbo pumps. Pirani gauges which are best suited for this role are relatively cheap and are the best diagnostic tool for determining overall vacuum system performance. The backing pressure should be consistent with changes in the high vacuum pressure. By monitoring the backing pressure over time, the base vacuum will be realised relative to the high vacuum so a pending failure of the backing pump can be determined, hopefully, well before it happens.

Care must be taken to maintain systems that have the potential to degrade over time for example an oil vane roughing pump will back stream oil mist that may accumulate in the high vacuum side of the system over time. This will contribute to lower vacuums due to higher gas loads from the partial pressures of contaminants.

## 4.3 Sustainable Operation

Now that the vacuum system is designed, manufactured and commissioned for routine service, operating procedures have to be defined and adhered to, to maintain reliable trouble-free service. There are 3 processes that summarise the continuous cycle of operating a vacuum system:

- Pumping down from atmospheric pressure
- Using the working vacuum
- Venting to atmospheric pressure

The accelerator beam lines are divided into stand alone vacuum systems but in essence function as one. Each system can impact on a neighbouring system if things go wrong. In the case of end stations which cycle up and down in pressure more than any other devices an operator must be 100% sure of the pump down sequence in order to avoid damaging, pumps, samples, high voltage devices, detectors, etc. It is possible to shut down the accelerators as well. Most sections of the accelerator vacuum systems are interlocked to isolate a pressure surge to small area. Beam line vacuum systems are design so that both the vacuum pump and the beam line are isolated via gate valves. This is a typical arrangement for endstations as well.

The following must be read remembering that all 3 processes work in a cyclic fashion in a continuous loop.

## 4.4 **Operational Protocols**

Key vacuum systems operate continuously, 24 hours per day, seven days per week. There are several vacuum systems that are cycled on and off as usage demands. All vacuum systems have one or more high vacuum gauges, some of which are interlocked to make the systems fail-safe. Any slow leaks or major catastrophic failures will close valves and isolate the system. In the few cases where no interlocks are used, a catastrophic failure will result in the high vacuum pump shutting down.

- For a <u>turbo pump</u>, as the pressure rises the gas load on the pump will cause the pump to draw more current to maintain it's speed. It will eventually fail when the current trip level is reached in the controller.
- For a <u>cryopump</u>, as the pressure rises the inflowing gas will condense or freeze onto the in the pump until it begins to warm up. Eventually there will be a thermal trip activated o the compressor which will shut is down. In both cases it is fail-safe.

As part of the accelerator's maintenance system, daily monitoring of all vacuum pressures is carried out. Where a pressure is seen to rise by a few percent without a particular reason, further monitoring will occur and corrective action started. Instantaneous failures are dealt with immediately. All observations and whether any maintenance carried out is noted on the maintenance log.

Where a vacuum system is isolated for service or where it has failed the appropriate tags are placed on the power leads.

Unattended out of hour's operation notification is not normally used as vacuum systems run continuously.

#### 4.5 Pump down

#### General

Pump down time is when most mistakes are made and significant time lost. Before a pump down begins the operator must ensure that all seals that were disturbed had been cleaned and replaced if damaged. The system must then be sealed ready for pump down and the roughing-out valve on the rough-out port, closed.

If a dry pump is to be used, such as a scroll pump, for rough-out then the tube is connected to the pumping port. If an oil vane rotary pump is used then ensure the cold trap has been filled beforehand.

#### Rough-out

Start the roughing pump and allow it to reach base vacuum. This is your first indicator of whether you have a leak in the system being pumped and when you have reached the pumping limit of the

pump. Slowly open the pump out valve on the system. This is where mistakes are made and damaged caused. Relatively speaking the volume in most sections of beam lines and endstations is small and the bulk of the gas can be removed very quickly. The problem is that a reduction in pressure too quickly can cause mechanical shock to fragile parts that must adjust slowly in order to minimise stress such as thin windows on detectors and detectors or devices that contain carbon foils.

Operators must always open roughing out valves slowly then gradually increasing as the gas load reduces. The change in the sound of the loaded pump is a very good indicator. A good design for more sensitive pump outs is to use a metering valve in line with the rough-out port to act as a regulating orifice.

#### Cryogenic Vacuum Pumps

For systems using cryopumps as the primary high vacuum pump it is essential to bring the base pressure down to the limit of the roughing pump before changing over pumping to the high vacuum pump. This reduces the gas load to be trapped (captured) on the cryo pump and extends the service life of the cryopump. Cryopumps are widely used on ANTARES as the primary high vacuum pumps but their limitation is that the warm up as they trap more gas causing a rise in base pressure. That is, when a cryopump is reaching saturation the temperature on the surface increases. More energy is required to hold the molecules on the surface and since it is almost fixed by design the temperature increases. A cryopump's effective pumping speed depends on the freezing and boiling points of the gases being pump relative to the cryopump's temperature. As the temperature increases more molecules leave the surface (boil away) raising the pressure. Some molecules are re-trapped as they hit the surface of the arrays and loose kinetic energy but they have a short residency time and soon add to the rising pressure in the system. Eventually the base pressure in the system becomes unusable so the pump must be regenerated.

#### Turbomolecular Vacuum Pumps

The other type of high vacuum pump widely used in the accelerator area is a turbo pump. Unlike a cryo pump that traps or captures gas molecules a turbo pump transfers the gas by conducting it almost one-way out of the vacuum space. The lowest roughing pressure before changing to a high vacuum pump is not as crucial when a turbo pump is being used. In saying this, the bulk of the gas must be removed before changing from roughing out to high vacuum pumping otherwise the vanes on the turbo pump may be overly stressed due to the pressure surge. A typical changeover pressure range is 5 - 50 Pa.

The optimum changeover time to minimise pump down times can be estimated with the assistance of the formulas found within the document. Letting the roughing pressure reduce to the limit of the roughing pump before changing over to the turbo pump is an inefficient method of system pumping. A turbo pump is more efficient at removing gas molecules as the flow moves towards molecular flow. Changing pumping to a turbo pump just before molecular flow is reached will increase the pumping speed and base vacuum will be reached sooner.

## Pumping Speed

Each type of gas will pump away at a different speed depending on the mean velocity of the molecule which is a function of its mass. A hydrogen molecule of 0.02 kg/mol and mean velocity of 1762 m/s will reach a pump much quicker than say a Xenon atom of 0.13 kg/mol with a mean velocity of 217 m/s.

## **Limitations**

In rare circumstances roughing pumps may be left pumping high vacuum systems by mistake. It is unlikely to damage either pump but the ultimate vacuum will not be reached as the roughing pumps are ineffective at conducting away molecules in molecular flow. Water vapour and residual solvents are difficult to remove from a vacuum space with *normal* vacuum pumping. Most low vacuum backing pumps provide gas ballast which can speed up the removal of water vapour and solvents that may otherwise condense inside of the pump during the compression stage. By introducing a small amount of air near the outlet of the compression stage the small increase in pressure helps carry water vapour and solvents to the exhaust before they can condense.

Some vacuum systems have been designed to allow chambers and beam lines to be heated. Heating causes contaminants to be removed from surfaces at lower than normal vacuums. The higher the temperature the faster the removal will be. Care must be taken to ensure heating doesn't inadvertently cause plastics and elastomers used within the vacuum space to outgas. Most bakeable systems use ceramics and metals only.

It is important that where vacuum systems are permanently connected to high voltage devices for roughing out, the insulated (plastic) pump out lines between the roughing pump and high voltage device must be brought up to atmospheric pressure before high voltages are reapplied. Otherwise the low molecular density of gas in the tube may ionise causing burning of the tube or even spark damage that can puncture the tube causing a leak.

## 4.6 Venting

## General

Venting can cause damage to vacuum components just like pumping out a system from atmospheric pressure too fast.

#### Venting Speed

As discussed earlier the volume of vacuum systems on the accelerators is measured in litres to a several tens of litres. A catastrophic loss of vacuum will be more of a brief audible event rather than a dangerous mechanical failure. Little energy is required to change these systems by 100 kPa. There are a few devices in the vacuum space that may be damaged due to pumping out too fast but none that are considered a danger to personnel.

Other than venting too fast the second point to consider is the introduction of water vapour if venting with air. Key vacuum systems on the accelerators are vented with either dry nitrogen or argon both of which are delivered through a closed reticulation system from gas bottles. The choice of gas is dependent on molecular contamination within the system for example nitrogen is not preferred to vent the 846 ion source as nitrogen is an element that is known to bond with other elements causing molecular ion beams.

#### Regulation of Flow

There are many low flow regulators and rotameters (tubular flow meters) available that provide adequate control over venting. A simple in-house gas regulating system which works well is a tee piece connected between the venting port and the venting gas supply line. The centre port of the tee piece faces upwards and a ping pong ball sits over the hole with a cage over it so that it cant be blown away. Under the ball is an 'o' ring seal. Before the venting valve is opened the venting gas is allowed to flow. The pressure of the gas lifts the ping pong ball of its seal. The gas flow is adjusted depending on the height of the ball above the seal. The ball can only travel a centimetre or so. Once the flow is set the venting valve is opened and the now regulated gas flows into the vacuum space and the balls drops onto the seal stopping air from entering the vacuum space. As the pressure reaches equilibrium the ball begins to rise off the seal until eventually it rises to the original set height indicating the vacuum space is now at atmospheric pressure.

#### Precautions

- Before venting a vacuum system it is wise to isolate all ports and beam lines that must remain under vacuum. To save stressing a beryllium window on an x-ray detector due to the changes in pressure the system should be designed so that the x-ray detector can be isolated with a gate valve. The same logic can be applied to all equipment that may be effected. It is also important to ensure all power supplies and high voltages that may be hazardous to personnel or equipment are isolated prior to venting. This can be done either by administrative protocols or using a vacuum switch such as programmable relays in a vacuum gauge controller.
- Some vacuum gauge controllers use the signal from a Pirani gauge measuring at the same location as a high vacuum gauge to switch off the high vacuum gauge when the pressure becomes too high. This saves the gauge from ionising air when the system is at atmospheric pressure which will significantly reduce its life time.
- Another problem with venting too quickly is the disturbance of dust. Accelerator tubes must be vented slowly to minimise equalisation stresses and also the disturbance of dust. In this case a metering valve is used to limit the vent flow rate.
- Venting can cause loud high frequency noise so care must be taken to dampen the noise to a comfortable level by controlling the air admittance rate, or use hearing protection.
- Most beam line vacuum pumping systems are interlocked to isolate either the beam line or pump or both. In the event where an accidental venting occurs the gate valves will close minimising the effect on surrounding systems.

# 5. Safety

## 5.1 General

Catastrophic failure causing injuries are not common and no reference is known from overseas accelerator facilities. This is almost certainly due to the low pressures involved in vacuums and the size and nature of the scientific apparatus. There are of course many failures reported for industrial sized tanks and equipment.

The vacuum systems are essentially fail-safe from absolute vacuum to atmospheric pressure. The worst case has been a number of beam lines and measurement chambers developing leaks and thin windows that have ruptured. In each case there has been a gradual rise in pressure to atmospheric pressure.

However, a few rules do apply.

- All vacuum systems must have one or more vacuum gauges attached with the exception to some static chambers used for storing moisture sensitive items or radiation detectors.
- High vacuum gauges must be switched off before vacuum systems are vented to atmospheric pressure.
- Only nitrogen, argon or other inert gas to be used for venting. No flammable gases to be used such as hydrogen.
- Turbomolecular pumps must not be vented from full speed. See manufacturer's recommendations.
- All designs must be consistent or better than the best practises outlined in this document. Inferior equipment must be isolated and reported to the Leader of Accelerator Operations.

- Cryopumps must not be operated without a serviceable pop off valve.
- Cold traps must be warmed and blown dry at least every 6 months to minimise  $O_2$  concentrating (and for de-icing as necessary).

## 5.2 Interlocks

Nearly all vacuum systems on the accelerators are interlocked via a set of integrated relays in a vacuum gauge controller, to protect equipment. Generally, if a rise in pressure is detected above a particular set level then the interlock relays will activate to close corresponding gate and line valves. This is designed to minimise the number of vacuums that must be regenerated in a fault. The interlock system also protects accelerator tubes from exposure to moisture and in the event of a catastrophic failure of the accelerator tubes; the vacuum system will close the main accelerator gate valves to trap SF6 gas.

If an interlocked failed causing a gate valve to stay open in the event of a pressure rise then the next interlock should activate the next gate valve. If the next interlock fails it is possible that the two interlocks share the same vacuum gauge controller which is at fault. In that case the next interlock should activate. If no interlocks activated there would be no increased risk of injury to personnel but all vacuum systems will require regeneration or a restart after the interlocks were repaired and tested.

## 5.3 Fumes from exhausts

There are a number of oil vane pumps in use on the accelerators. In the past, the exhaust on the pumps has released oil mist into the atmosphere inside the buildings. Now, on nearly all oil vane pumps in continual service there are mist filters attached or the exhaust is plumbed into an extraction system that vents the exhaust fumes outside of the building.

## 5.4 Earthing

Some vacuum gauge controllers are prone to high voltage transient surge damage. These sometimes cause unnecessary isolation of systems requiring resetting. It is very important that all vacuum gauge controllers are well earthed.

## 6. Serviceability

## 6.1 Access

Most beam lines with the exception to some measurement chambers are static volumes of vacuum requiring little operator intervention other than during preventative maintenance. Vacuum pumping systems do require a higher level of intervention during maintenance and operation. With these points in mind the designer must ensure that all controls and monitoring devices are localised at the working face of the beam line or chamber.

Due to beam lines being so high above floor level, working on them usually requires some form of ladder or steps. When designing beam lines, be sure to have pump out ports facing the passageway beside the beam line. The same applies to vacuum chambers and other large volume vessels.

Measurement chamber will need to have the access port accessible from a location near the control side of the chamber. A platform is recommended to allow easy reach and eay viewing into the ports.

## 6.2 Maintenance

A well designed vacuum system will require routine maintenance including:

- Changing faulty seals cleaning seals that are regularly opened
- Cleaning vacuum gauges especially cold cathode type high vacuum gauges
- General cleaning inside of measurement chambers and equipment that is; regularly opened and touched, whenever it looks dirty, when the base vacuum degrades or when the surfaces are exposed to the atmosphere for long periods at a time (weeks).
- Servicing of vacuum pumps the group of the most routine maintenance tasks includes; cryopump regeneration, turbo pump bearings, scroll pump tip seals and oil vane pump oil changes to name a few
- Actuator service sliding shafts that penetrate into the vacuum space

## 6.3 Cleaning

Cleaning is by far the most essential part of general maintenance of a vacuum system. Poor cleanliness will lead to poor vacuums and possibly sample contamination. It is a general philosophy that cleaning is done with solvents, wiped over and/or scrubbed and thoroughly dried.

## **Solvents**

Typically ethanol, methanol and acetone are used. These solvents remove most greases and oils that are encountered. What ever solvent is chosen the most crucial point to remember is that it must not leave any residue.

For surface finishing see section on 'Surface Finishes'.

Chemical polishing is not commonly used in the accelerator area due to the size of the baths that are needed for large parts, the problem of cleaning or neutralising residual chemicals and not having a suitable facility for handling the types of chemicals used. Although once used electro-polishing is no longer encourages for the same reasons. If suitable facilities were available both chemical and electro-polishing are real options for very good cleans.

A general clean will consist of a number of different levels of cleaning. A single clean with the same solvent will not remove some oils and greases therefore a progressive approached is used using a variety of solvents and wiping materials. The following is a guide only of some such techniques.

Using a dry gas or filtered compressed air, most loose dust can be blown away. This is sometimes the best starting point for cleaning. A final blow down is also useful to ensure complete removal of lint.

The first clean will take away coarse particulates or chemicals. Ethanol dampened tissues if good for this level of cleaning. The ethanol helps remove some oils and greases and mixes with water to reduce the evaporation time. It is also good for mopping up dirt and dust. Scour pads (or 1200 wet or dry paper) moistened with ethanol is good for removing stubborn dirt. This must be continued until better than 95% of dirt, dust, oil and grease is removed. For stubborn dirt it may be necessary to use a petroleum based solvent followed by an ethanol rinse and wipe. Wiping is key to cleaning. The light mechanical brushing dislodges most dirt.

The second clean starts with ethanol but finishes with acetone. Only lint free wipes are used such as Kimwipes. At the end of this level of cleaning the equipment must look ready for use in the vacuum. That doesn't mean it is. It will more than likely have residues remaining on the surface.

Final clean. It is recommended that methanol is used for the final clean or another low residue solvent. The final clean must use lint free wipes dampened with methanol and lightly rubbed over the entire surface. An alternative final clean is to use the solvent bath and suspend the equipment in the hot vapour for a few minutes. Care must be taken with the bath as the hot vapour will degrease skin very quickly.

If a shiny (healthy looking) clean is desired then a metal polish such as Brasso can be used. Whenever Brasso is used the residue must be cleaned off with ethanol. Care must be taken not to get metal polish pastes into gaps that cannot be thoroughly cleaned out. Brasso is an enemy of vacuum, it traps water.

## 6.4 leak detection

In the accelerator area, a leak is characterised by a flow of gas molecules into a space that is below atmospheric pressure. The pressure of the gas entering the space will restrict the vacuum system from reaching its design pressure which is typically in the high vacuum range. As a benchmark, if a vacuum is  $1 \times 10^{-5}$  Pa or more then disregarding the effects from outgassing, there may be a leak. In system designed for ultrahigh vacuum the benchmark will be in the order of  $1 \times 10^{-7}$  Pa.

Leak detection is usually only necessary for new chambers or equipment. It can be done offline or after the part has been mounted into a beam line. The most common method of leak detection is a helium leak test. Helium atoms being so small will find their way through openings that are too small for most other molecules. Generally, the smaller the leak, the less helium that can make its way along a leak path. The leak detector is usually a mass spectrometer tuned to have highest sensitivity for helium. The detector is connected directly to the device being tested. Any helium that makes its way through the leak will eventually be measured in the mass spectrometer. Residual Gas Analysers (RGA), although not specifically leak detectors, can be used for just that. Working on the same principle, mass spectrometry, the RGA can detect helium as well as other gases.

In some cases leaks can be too large for a helium leak test, that is, the amount of gas in the device being tested is too high diluting the helium to an ineffective percentage. Also, if the pressure is too high the leak detector cannot be opened to the device as the pressure gradient will be too high. Leak detectors work best from the higher end of the low vacuum range.

Often there are multiple leaks in systems. It may mean repairing large leaks in order to find small leaks. Where a leak is too large for the helium leak detector a few other methods can be used to locate leaks.

- Solvents have a very low viscosity and vaporise very quickly in air. Wetting surfaces with small amounts of acetone or ethanol can show small changes on Pirani and Penning gauges.
- Isolation of seals can, by process of elimination, restrict the testing area allowing large leaks to be isolated. For example, if a chamber with many ports and devices attached has a leak. It may be prudent to close all valves leading away from the chamber except for the leak detector valve then sequentially blank off the ports one at a time (using a sound flange and new seal) and test for a leak.

## 7. Soldering wires vs mechanical joints

It is preferable that wires in a vacuum space are joined together with a mechanical clamping type joint. The clamp must allow for gas to escape. Where a direct joint is necessary then soldering is permitted. Standard lead/zinc with resin core is okay provided that ALL flux is removed with ethanol. This may require some soaking in solvent with gentle agitation. Solder without resin core

can be used on stainless steel with a zinc chloride based eutectic flux, used sparingly. Ensure all flux is removed especially between the wire strands.

Be aware that in Faraday cups the continual flexing of wires can work harden the wire causing it to break. These are typically soldered joints. If soldering, be sure not to melt the insulation. It is highly recommended to replace normal plastic insulation with loose fitting PTFE (Teflon) spaghetti.

# 8. Flanges and seals

## 8.1 Common vacuum seals used in the accelerator area

A variety of standard vacuum seals are used on all vessels depending on the degree of vacuum to be achieved and the type of fitting that will be used. The types are widely used in the vacuum industry. ANTARES uses the Dependex style seals and flanges. This was developed for use with HVEC accelerators over 50 years ago. Other types in use include Conflat, ISO, and KF are used as well.

Туре	Vacuum level	Flange type	Seal type
KF	Low to High	Clamp (typically)	Viton 'o' ring, Al, PTFE
Edwards	Low	Threaded retaining nut	Viton 'o' ring
Dependex	High	Ring retained by spring clip	Viton 'o' ring supported by a ring
LF or ISO	High	Ring or clamps	Viton 'o' ring supported by a ring or in a groove
Conflat	Ultra high	Ring fixed or rotating	Copper ring, Square section 'o' ring
Specials	Various	Various	Aluminium or indium wire

Table 1. Common types of vacuum flanges and seals. Each type is available in multiple sizes

## 8.2 KF – Klein Flange

A common flange and seal used for general low vacuum applications due its ease of assembly and vacuum performance. It is common to have KF flanges and seals within the high vacuum side of a system. Vacuum performance can be enhanced by the use of aluminium or PTFE seals which directly replace the Viton 'o' ring.

Many general and specialised vacuum equipment is fitted with KF fittings as the range of adaptors from KF to other major types is readily available.





Fig 3. Assembly drawing for a typical KF flanges seal and a photo of the flange, seal and clamp

## 8.3 Edwards

This is no longer a preferred type that is used in the accelerator area however some equipment still uses this type.

One end of the fitting is shaped to retain an 'o' ring while the other end has an angled sealing surface that mates and crushes the 'o' ring to make a seal. They are connected by a threaded nut which when tightened squeezes the two halves together. It is normally for low vacuum applications.

## 8.4 Dependex

This was designed for use on HVEC Van der Graaff accelerators and beam lines. It was introduced to ANSTO in the early 1960's when the 3MV Van de Graaff operated in B22. Here, 1", 2" and 4" Dependex was used. When ANSTO purchased the FN Tandem nearly all fittings used were 4" Dependex (100 mm nominal). It is suitable for high vacuum use and often mated with Conflat flanges in systems that operate near ultra high vacuum.

The Dependex seal consists of a supported 'o' ring sealing between two opposing  $5^{\circ}$  metal faces. The 'o' ring is supported around its inner diameter by a ring to ensure the 'o' ring aligns with the centre of the 2 faces. The inner ring also sets the maximum crush on the 'o' ring that is the ring fits into a step on both halves of the Dependex seal keeping them separated at a fixed distance. Another ring which fits on the outer diameter of the 'o' ring does little more than keep the 'o' ring clean. The rings are often called *egg rings* due to their resemblance.

The Dependex flanges are held in place on a tube, or other fitting that is designed to accept Dependex, by a large circlip that fits into an outer groove a few mm from the end of the sealing face.

The flanges are normally drilled such that the holes alternate between being tapped or clearance. 1" and 2" Dependex use <sup>1</sup>/<sub>4</sub>" UNC, cap (socket or Allen) head screws and 4" (100 mm) Dependex use 5/16" UNC, cap (socket or Allen) head screws. Only use stainless steel screws (of one length) and washers. Discard plated steel screws as they are identified during maintenance. Washers are recommended as most flanges are aluminium alloy.

To complete the Dependex assembly, the flanges must be placed over the tube ends and then retained in place with a circlip. Secondly, the 'o' ring is stretched onto an inner ring and then an outer ring is placed around the assembly. The two Dependex ends are brought close together then the seal assembly is placed between them. The inner ring is aligned with the step on one side then the other tube is aligned and than pushed onto the ring. At this point it is important to ensure the 'o' ring assembly stays in place until the bolts are tightened. Screws should be finger-tightened and the flange faces must be parallel. Tighten screws in an opposite pattern. Some old flanges will have worn threads so screws may only be turned with the aid of an Allen key.

#### Problems

There are a number of things that can go wrong with assembling Dependex fittings.

- There have been cases where inner egg rings have been made too wide so that when fitted between the tubes the 'o' ring is prevented from crushing adequately to form a vacuum seal.
- Another problem with the inner rings is where some Dependex seals have been designed with too shallow steps so that a standard inner ring becomes too wide. In this case special inner rings have been made which are narrower. Personnel servicing non-standard joints must take care to reuse the right sized inner ring. A label should be placed on all non-standard joints identifying them.

- Some steps for inner rings have also been found to be out of tolerance preventing standard size inner rings from locating in the step. In this case some operators have cut a small section from the inner rings so that they can be squeezed together. Cutting inner rings like this is only recommended to solve this type of problem.
- Some flanges have been design for special applications with slightly different PCD's. These have unfortunately bee mixed up with standard stocks. As personnel who identify them (sometimes after a frustrating encounter) should put them aside and label them accordingly.
- Old flanges that have been over-tightened may have stretched threads. They are identifiable by the difficulty in starting screws. Either re-tap the threads or discard the flange. There have been case were tight threads have mislead personnel into thinking they have tightened the Dependex joint only to find leaks.
- Circlips can stretch especially those in over or under sized slots. They are easily identified by there distorted shape. Only use circlips that are flat when not under tension. A discerning technician should discard rusty circlips that cannot be successfully cleaned.
- Care must be taken to make sure the two faces of the Dependex joint are parallel before the joint is closed and bolted together. Failing to do this may cause a leak in the joint. It is acceptable if a bellows is used between sections that have misalignments.
- Twisted 'o' rings can cause leaks although small. There have been cases where a twist has caused the 'o' ring to bulge towards the outer ring making a less than adequate seal.

## 8.5 Special Flanges

These consist of flanges that have been designed to fit application where "off-the-shelf" flanges and seals cannot be used. They are typically based on common designs but modified to suit the application.

## Example

The Alphatross ion source uses two stainless steel flanges to hold the heater reservoir in place. Here the vacuum sealing surface is a flat surface about 8mm wide on each flange. The fixed flange is threaded in an imperial thread and the reservoir flange is open hole. The gasket used is aluminium wire which has been fused together to make a ring. The joint is similar in size to the wire. The wire is supported in place using about 3 to 4 strips of aluminium foil looped around the wire and sticky taped to the edges of the flange. The flange is tightened in a circular pattern to ensure maximum crush on the wire.

## Problems

- The wire is essentially unsupported so vacuum must not be applied until the wire is completely crushed.
- Bulbous joints in the aluminium wire dent the sealing faces on the flanges as they are tightened so they must not be used.

## 8.6 ISO or LF (Large Flange)

This is a European range of fittings designed for high vacuums and to suit most tube sizes used in the vacuum industry. There are two types of design which meet most light and heavy duty applications.

ISO-K flanges are a robust joint which are suitable for supporting larger and heavier items such as vacuum pumps and large gate valves. They are characterised by the outer clamping system which holds the two halves of the joint together.

ISO-F flanges are more of a traditional bolted flange type joint like Dependex. ISO-F and ISO-K can be joined together using a half clamp which fits onto a standard ISO-F flange. The half clamps then attach to the ISO-K flange.

Available in sizes from 63 mm nominal bore to 500 mm NB.



Fig 4. ISO family of Flanges

## 8.7 Conflat CF

Conflat is designed as an all metal vacuum seal for ultra high vacuum applications. A copper gasket is sandwiched between two thick stainless steel flanges that have a knife edge machined into the sealing face. As the flanges are tightened together the knife edges "bite" into the copper to produce an all metal seal with very low outgassing.

The term "ConFlat" is a registered trademark of Varian, Inc., so "CF" is commonly used by other flange manufacturers. Conflat sealed systems can reach vacuums as high as  $1 \times 10^{-11}$  Pa.

The copper gasket locates into a partially recessed in a groove in each flange to provide alignment prior to tightening. It also stops the gasket from moving during bake out. Conflat can be baked up to  $450^{\circ}$ C.

Most Conflat flanges have imperial sized bolts. The original style of bolt had a multi-face head that a ring spanner would fit over. This is to allow more purchase on the head from many angles. It is not uncommon now to find hexagon and socket head screws in use. Be aware that the steel must be a high tensile grade to ensure optimum force can be used to crush the gasket. It is essential that the flange faces are parallel during and on final tightening. A torque wrench may be used to facilitate uniform tightening. It is highly recommended that a molybdenum grease or graphite loaded grease is used on the threads.

Conflat flanges are available in sizes from 10 mm nominal bore to 250 mm NB. Sometimes nominal bore is written DN in front of the size, eg DN63.

Square section Viton 'o' rings may be used with Conflat flanges however this compromises the ultra high vacuum design and ultra high vacuums may not be reached.

## Problems

- Copper gasket not aligned in groove and edge is partially clamped on the bolt hole face of the flange. This lead to partial seal and most likely, a leak. Most Conflat flanges have two small grooves a little more than a millimetre wide machined into the bolt face. These are for locating special clips that hold the gasket in place while the flanges are brought into place. Just prior to tightening they clips are removed. They are sometimes mistakenly called pressure relieving grooves.
- Copper gaskets must only be used once. Unless the copper hasn't been fully compressed after the first use there will not be enough material for the knife edge to reform a seal.
- Due to the forces required for a knife edge to indent a copper gasket the flanges are made of stainless steel and quite thick. Large flanges are quite heavy and care must be taken not to drop them.
- Where Conflat flange bolts have been used more than once the threads in the flange and screws may have stretched. In this case, it is recommended that the same screws are reused in the stretched threads (with a little molybdenum grease) as they will have the same profile. Sometimes using new screws in a stretched thread will cause binding. Re-tap the flange if necessary.
- Stretched threads are possible where nuts and bolts are used through open holes so ensure the nuts go onto the bolt the same way they were removed. Alternatively replace the nuts and bolts outright.
- For ultra high vacuum ensure the gaskets are handled with gloves. Copper reacts well with the sweat in a fingerprint leading to a long outgassing process.

# 9. Bellows

Bellows consist of two styles, edge welded for ease of extension or compression and corrugated style which is has minimal movement. Each style is made from a thin stainless steel that has been electron beam welded. The ends are usually finished in a standard flange to suit the application.

## 9.1 Corrugated style

These are typically used for two purposes. Firstly, to allow sections of beam lines or equipment to be compressed to allow easy removal of sections without dragging the sealing surfaces against each other. Secondly, as bellows can distort and still maintain their structural integrity they are ideal to

join sections of beam line or equipment that have a deliberate mis-alignment. They can be purchased with almost any style of flange including specials. See example pictures.



Large bellows. ~300mm diameter. Stainless steel, wall thickness ~ 0.8 mm. This bellows is used to correct alignment offsets in the magnet box

Medium bellows. ~ 100 mm diameter. Stainless steel. wall thickness <0.5 mm. Used to allow sections of the beam line to be compressed to facilitate beam line component removal.



Small bellows. Electron beam edge welded stainless steel. Material thickness <0.25 mm. Used to isolate vibrations in the beam line.

Fig 5. Examples of bellows

#### 9.2 **Edge Welded Bellows**

These bellows are made up from very thin stainless steel discs stacked together and welded along their edges. The advantage of the design is the concertina style bellows that is very flexible allowing designs that have long extensions and short compressions. They have two main applications. Firstly, applications that requires a device to extend or contract into the vacuum space where an elastomeric seal on a sliding shaft is not adequate. Such as a sample manipulator or Faraday cup. Secondly, where a device such as a detector or sample positioner is sensitive to vibration and must be decoupled from the vibration source.

## 10. Valves

Along the axis of the beam lines gate valves are used to enable the isolation of sections for maintenance and also to minimise loss of vacuum in the whole accelerator if one section develops a leak. Most gate valves are electro-pneumatically operated and many are interlocked to local high vacuum gauges. The few that are manually operated are for maintenance isolations.

On ANTARES the electro-pneumatically operated valves are interlocked to close when the pressure, as measured near the gate valve, rises above 5 x  $10^{-5}$  Pa. This figure can vary depending on the location of the valve. See the Accelerator WIKI for values around the ANTARES. Once an interlock has tripped the switch that operates the gate valve is bypassed. To reset the valve:

- The vacuum must be within the high vacuum range as read on the corresponding vacuum gauge controller with a pressure better than the trip point.
- Once this level is reached the bypass button is held down momentarily.
- At this point the pressure will rise then fall as the gas load is pumped away.
- When the system pressure is less than the trip level the bypass switch can be let go.
- An indicator light near the switch will show its status.

#### Notes:

- Operators and technicians must be aware that gate valves must be orientated so that the gate always seals towards the vacuum pump. That is, they seal best in one direction only. Failure to orientate the gate the correct way may cause a vacuum leak when atmospheric pressure forces the gate off its seal.
- In some cases gate valves may remain sealed after they have been switched to open. This can be due to the withholding (sometimes called back pressure) pressure not being adequate enough to lift the gate off the seal.
- Gate valves, while mostly metal can become hot if a gate is closed in front of an ion beam. If left for long periods with high current beams the elastomer seals may warm up and begin to outgas.
- For roughing ports typically 25KF bellows sealed valves are used. On STAR these same valves are used to isolate vacuum gauges for cleaning.
- Where fine flow control is required for either roughing out or venting, metering valves are used. These are basically multi-turn needle valves for fine adjustment.
- Valves do require maintenance to keep the seals clean and in working order. After long periods of time the 'o' ring seals will deform and often will not recover their original shape.

There have been attempts to build fast acting gate valves for the purpose of isolating the accelerator tank's load of  $SF_6$  in the event of a breach in a tube. While it seemed like a good idea the force of the valve closing at high speed could damage the gate seal causing the valve to leak. Designers must use care to ensure that a standard high differential pressure rated gate valve isn't adequate before launching into these expense type valves.

# **11. External Interfaces**

## **11.1 Electrical Feedthroughs**

a) Low Voltage, Low Current

There are few applications where voltages greater than 24Vac are used within the vacuum space. For these voltages at a few amps coaxial feedthroughs such as BNC, SHV, MHV, etc, are often used in place of more expensive dedicated commercial types. In one application 200Vdc is used for faraday Cup suppressors but current is only a few micro-amps at most.

Notes:

• Do not use multi-pin signal feedthroughs to carry voltages unless the pins used for voltage can be a few unused pins away from used signal pins.

- Feedthroughs can be purchased is almost any flange style but are usually restricted to small size flanges.
- Insulation must be used on all wires carrying voltage. It is highly recommended to use single or stranded wires (preferably nickel coated) with loose fitting PTFE (Teflon) spaghetti.

## b) Signal

Signal feedthroughs other than coaxial such as BNC, SMC, Microdot, etc are from one to multiple pins. It is recommended that connections to multi-pin feedthroughs are by the use of slide on pins. Many are either sliver of gold plated for good electrical contact. Avoid soldering unless care is used to remove all traces of flux and no pin-holes in the solder have been created

On coaxial feedthroughs the coaxial connector will be either both sides of the feedthrough and hermetically sealed or with the coaxial connector on the atmospheric side and a plain pin on the vacuum side, also hermetically sealed. For either type, where coaxial cable must continue into the vacuum space it is recommended to remove the outer plastic insulation and replace it with loose fitting PTFE spaghetti. This is also recommended for all single or stranded wires carrying signals.

## c) High Voltage, High Current

There is a wide range of high voltage and high current feedthroughs available in most flange types. High voltage feedthroughs usually have a larger insulator in ceramic and sometimes glass. These must be kept clean on the atmospheric side. Long term breakdown on high voltage feedthroughs can damage the insulator causing vacuum leaks.

For high current applications such as heaters, dedicated high current feedthroughs must be used. These are characterised by larger cross section wires to carry the higher currents. Like HV feedthroughs they must be kept clean. It is recommended to clean the insulators on signal feedthroughs periodically to remove any accumulation of dust.

## d) Power

There are few feedthroughs designed for powering devices at mains voltages. It is highly recommended that experiments are designed not to have mains voltages in vacuum vessels or equipment due to safety concerns.

## **11.2** Mechanical Feedthroughs

## a) Rotary

Rotary feedthroughs allow the transition of rotary movement into the vacuum space to, for example, operate shutters or position samples or detectors. They are widely available in many of the common flange styles. There are three common types both of which are very good in high vacuums.

The first type is a continuous shaft between the atmospheric side and vacuum side using rubber vacuum seals in the form of either supported 'o' rings, square or odd shaped rings (like common shaft seals), and flat rubber sheet seals. These are best for general rotary actuation rather than continually rotating. Okay in high vacuum but care must be taken to keep the seals clean and periodically replaced or whenever wear is noticed.

The second type is a ferro-fluidic type which also uses a continuous shaft between the atmospheric side and vacuum but the seal is made by encapsulating a dense fluid around the shaft. These types usually have bearings to keep the shaft centred and free moving. They are best for high revolutions in high vacuum applications.

The third type is magnetically coupled rotary feedthroughs. These are expensive. They provide a seal by magnetically coupling the shaft on the atmospheric side to the shaft on the vacuum side across a continuous sealing plate. If either shaft is removed the vacuum is not compromised. These are excellent for ultrahigh vacuums and lower. They are for moderate speed revolving applications and general actuations.

## **b**) Sliding

Usually these feedthroughs are a round shaft that can slide in and out of the vacuum as well as rotate. Sliding feedthroughs are used for positioning devices or where a linear actuation is necessary. The length can vary from a few millimetres to hundreds. In either case there is a shaft which penetrates from the atmospheric side to the vacuum side. The type of seals used varies like in the rotary seal. Sometimes dual seals are used to minimise leakage especially in high actuation rate applications. In some applications the void between the two seals is pumped increasing the reliance of the seal. These types are good in high vacuums. Notes:

- Care must be taken to ensure the shaft is lubricated with a suitable vacuum oil or grease. It is imperative that the oil or grease on the shaft is changed regularly to minimise dust build.
- There are bellows sealed sliding seals available. These types do not rotate and can only move in and out a few tens of centimetres at most. Very good in high to ultrahigh vacuums.
- Some sliding seals are able to move radially which will affect alignment. Where alignment is crucial a linear bearing type is recommended.

## c) Wobbler

A wobbler feedthrough allows users to use a shaft as a lever or pick up. They are designed around a flexible rubber or bellows seal they allows a greater range of circular movement.

## **11.3 Manipulators**

These are a class of feedthroughs design for high to extremely high precision positioning of samples, detectors or other instruments. They can be purchased having all or either X, Y, Z and rotary movement. The X and Y axes can have various travel lengths however they are typically less than 100 mm each. The Z axis (vertical) can have greater lengths but is limited as with the X and Y axes by the physical limitations of the vacuum bellows.

## 11.4 Actuators

'Actuators', combine sliding and rotary feedthroughs with pneumatic, electro-pneumatic or mechanical actuators to do the work.

In the accelerator area, actuators can be found on Faraday Cups to insert the cup into the beam line on a bellows sealed sliding feedthrough and inside of the ANTARES pressure vessel to move the stripper tube in and out of the beam axis.

## 11.5 Load locks

Load locks are not common in the accelerator area but are ideal for moving samples into a high vacuum space without significant loss of vacuum pressure. A sample or device is mounted onto the end of the load lock shaft. It is inserted into a tube connected to a chamber but isolated from the chamber vacuum by an in-line gate valve. The tube is sealed and then evacuated to a vacuum pressure similar to the chamber. Once the pressures equalise the gate valve is opened and the shaft pushed into the vacuum space. From this point the sample or device can be removed from the shaft by other remote means.

# **12. Internal Devices**

There are many devices that are commonly used inside of vacuum systems. Not all are represented in this section however similar reasons are use to assess new and unusually devices for use in vacuum.

## 12.1 Lighting

Lighting is necessary in most sample measurement chambers where the operator must be able to see the sample in order to position it in front of the ion beam. In most cases a standard bare 12Vac halogen bulb is used with good results. Care must be taken to ensure the bulb can be dimmed as when running at maximum power the heat load can only be dissipated through the connecting wires. That is, there is little gas inside a vacuum to conduct head away from the bulb. Bright white LED's are also used with mixed success. They have less radial light output as compared with a bare halogen bulb. If the LED is setup correctly the light can be focussed towards the necessary target.

Preferably, wires must be stranded or solid wire with a loose Teflon sleeve but normal plastic coated hook up wire works in high vacuums. In some cases where the wires as not likely to come into contact with other components then they are used uninsulated. All wiring must be restrained so that it cannot move into the path of the ion beam. As a rule-of-thumb wires should be at least 20 mm from the beam.

A standard double BNC feedthrough can be used, that is, only the centre core connections are used. There are many off-the-shelf electrical feedthroughs available that will be more suited for this specific use.

## 12.2 Heating

Providing power to a heating stage on a sample (target) stick requires a high current feedthrough. If a feedthrough isn't provided from the supplier of the heating stage then many types can be found from larger vacuum suppliers. Ensure the feedthrough selected is designed to carry the maximum current that can be supplied and not the maximum current likely to be used. Like in the lighting section above, care must be taken to ensure wires are insulated and away from the beam path.

Heating stages rely on heat dissipation through the support for the stage which is usually made from stainless steel, a relatively poor conductor. When opening devices for handling be sure they are cool to touch first.

## 12.3 Cooling

Cooling stages come in 2 distinctly different types. The first is the Peltier cooled stage which is all electric. Powering the stage can be managed as for lighting and heating. The second type is a liquid nitrogen cooled system. It uses an insulated tube to carry LN2 through a feedthrough into the vacuum space. A typical design is where LN2 is pumped through a coil attached to a heavy copper braid. The braid is then connected to the cooling stage where heat is conducted away from the mounting stage. Thermal isolation of the cooling stage from the holder can be Teflon or sapphire standoffs or similar materials.

These cooling devices must only be switched on when the vacuum is at the working level otherwise condensation and possibly ice can form on their surface becoming a large outgassing problem. Likewise ensure the stage is close to room temperature before opening up the vacuum system to atmospheric pressure. Always vent with a dry gas.

# **13. Pumping**

## 13.1 General

It is not the intention of this document to describe the fundamental operation of particular vacuum pumps. Adequate information is available from manufacturer's catalogues. The following is information on what type of systems are used in the accelerator area and the reasons why.

Vacuum pumps don't suck. They act essentially as one way valves in a system where gas is flowing towards them. This is discussed in Chapter 3 of this document. There effectiveness is there ability to stop recoiling gas molecules returning into the vacuum space such as a beam line or vacuum chamber.

When considering what type of pump to use for a vacuum system the following points must be considered.

- What is the application?
- Will it have a relatively high gas load, that is;
- Will it be pumping a static gas load or changing gas load?
- Will there be a high outgassing rate?
- Are there known hidden/trapped voids to be pumped?
- It is a clean or dirty device? Is it contaminated with grease and oil?
- What pumping speed will be needed to maintain a high vacuum?
- What is the volume of the system? What is the conductance of the system? Will a small pump be just as effective as a large pump?
- What gases will be pumped?
- What type of pumps and how many are being used in the vicinity?
- Will the pumping speed need to be regulated such as for pumping near foils or delicate samples, detectors, etc?
- Cycling time whether it will be required to regularly pump from low vacuums
- Will the pumps operate reliably in their chosen application?
- Are they compatible with other pumps used on the accelerators? Are they cost effective?

Another consideration is vibration. Pump vibration can be significant leading to micrphonic interference in detectors and vibration of samples being measured. Vibration can be reduced by selecting pumps with less inherent vibration like turbomolecular pumps or the more fickle ion pumps. If a pump is a source of vibration it can be decoupled from the detector or measurement chamber by the use of a flexible bellows. An fine edge welded bellows is recommended.

## **13.2 High Vacuum Pumps**

See section 4.5, Pump Down characteristics for cryogenic and turbomolecular vacuum pumps.

## <u>Cryopumps</u>

For ANTARES the main beam line pumps in use are cryopumps. The types of pumps trap (or immobilise) gas molecules. They are used because they have a long lifetime between major

services, can be regenerated without removing them from service, have a high pumping speed for most gases and can maintain high vacuums for long periods in a static vacuum application.

#### Turbomolecular pumps

Where a vacuum system is cycled such as at a measurement chamber, then turbo pumps are used. Turbo pumps are transfer type pumps meaning, gas is transferred from the vacuum space into the atmosphere. A turbo pump can routinely recover a vacuum from low vacuum after a measurement chamber has been roughed out without needing regeneration as for a cryopump.

Turbo pumps are also more effective at pumping lighter molecules such as helium and hydrogen as compared with cryopumps. Using turbo pumps near systems that use these gases will reduce the effect of loading on cryopumps.

Most new turbo pumps can be serviced in-house. Some models purchased require special balancing of the bearings after they had been changed. In this case they are sent back to the manufacturer at great cost. It is important that models chosen can be maintained in-house.

#### Other high vacuum pumps

There have been few applications where titanium sublimation or ion pumps have been used in the accelerator area. Most have been stand alone UHV systems or cryostats for specialised applications. The practicality of cleaning and regenerating these gas trapping type vacuum pumps on systems requiring relatively frequent cycling is prohibitive.

Getter materials once used widely in electronics in tubes are becoming more used within larger static volumes such as beam lines and associated equipment. They can be coated onto surfaces to assist in maintaining uniform high vacuums in spaces that have poor pumping speeds.

## **13.3** Low Vacuum, Roughing or backing pumps

#### Oil free pumps

These pumps are categorised by either being an oil type pump or dry (oil free) pump. Oil free vacuum systems are of choice for accelerator systems now that the technology has been proven to be reliable and cost effective. The majority of roughing and backing pumps now in use are scroll pumps as well. Piston pumps have been trialled with little success due to the short life of the seals. Some applications have used diaphragm pumps and multistage roots pumps but scroll pumps remain the most cost effective and easiest to maintain option. The Edwards type scroll was chosen over other brands as this type of scroll pump is designed with the bearings mounted outside of the vacuum which in other brands often caused the bearings to fail from lubricant loss.

#### Oil vane pumps

Low Vacuum, Roughing or backing pumps are all the same type of pump. Oil vane type pumps were widely used in the accelerator area until 2006 when a systematic change began on the STAR accelerator to replace oil vane pumps with oil free scroll pumps. Some oil vane pumps remain in use where some back streaming oil vapour is not considered a problem. If an oil vane pump is to be used for roughing out purposes, it is high recommended that a liquid nitrogen cold trap is used to trap vapours. It is the ultimate aim to have oil free pumping on all accelerators in the future.

# 14. Monitoring

## 14.1 Vacuum Gauges

The standard type of high vacuum gauge in use is the cold cathode penning type gauge.

- On ANTARES the Pfeiffer brand is in use in two versions; the standard cold cathode type and full range type which has an integrated Pirani gauge.
- On STAR the most widely used brand is Leybold.

Generally, vacuums are measured as an indication only for process controlling and establishing basic quality to ensure pressures are within levels that are accepted for "normal" operation. This varies depending on location and use of the vacuum system for example a measurement chamber is cycled often and affects surrounding vacuum systems. It will more than likely be a much high pressure than surrounding static systems.

In most cases, absolute vacuums are not required to be measured. However on some AMS beam lines where thin windows are used it is important to have accurate measurement of pressures when cycling pressures in the gas detectors. If accuracy is required it must be remembered that different gases ionise at different pressures and so pressures readings from the same volume will be different depending on the dominant background gas. Most gauges have characteristics related to Nitrogen (air).

## **14.2 Gauge Controllers**

On ANTARES the most widely used gauge controller is the Pfeiffer TPG300. This is an aging model and is being replaced in some key areas with the Pfeiffer Maxi-gauge which allows 6 heads to be connected at once.

The TPG300 has inputs for up to 4 heads whether Penning or Pirani. The limitation with the TPG300 is the electrical robustness when operated near the accelerator high voltage generator. Many failures have been attributed to high voltage transient surges. The TPG300 is now technologically aged and will ultimately be replaced outright. Various other models of TPG's are in use on ANTARES especially where single gauges are required. It is the TPG300 controllers on ANTARES that provides the interlocking capabilities.

The MAXI gauge is still being trialled as a replacement to the TPG300. A unit under test for many years at the HE end of the accelerator has already failed during an accelerator sparking event. Its real place may be away from the accelerator where the 6 heads allow consolidation of a whole beam line's vacuum monitoring system.

On STAR the common high vacuum gauge is the Leybold cold cathode type. It has been integrated into the STAR control system via cat 5 cable. They operate using a similar principle of measuring current flow across the plasma of the ionising gas. These gauges have been far more unreliable than the Pfeiffer equivalent. Unfortunately they are not interchangeable.

If venting with gas other than air or nitrogen be aware that the response on the Pirani gauges may show lower pressure than actual pressure.

## 14.3 Bourdon Gauges

Bourdon gauges are normally used for indicating that pressure is below atmospheric. They are not used on accelerator systems as they have poor accuracy in the range of interest i.e., below  $1 \times 10^{-3}$  Pa.

# Chapter 3



# **Overview of Basic Vacuum Technology**

The ANSTO accelerators consist of 140 metres of beam lines under vacuum. Supporting these beam lines are 60 individual high vacuum pumping stations. There are over 70 isolation zones along the beam lines to allow isolation of a zone without interrupting the adjacent zones. This facilitates isolations to repair leaking sections, routine maintenance and cycling of pressure in zones to change samples, etc.

The following information are the general tools used by technicians and engineers to design reliable and high performance vacuum systems for the accelerator facilities.

# 1. Vacuum Overview

## 1.1 What is vacuum?

A vacuum is defined as a diluted gas, or the corresponding state at which its pressure or density is lower than that of the ambient surrounding atmosphere.

Due to the Earth's gravity, atmospheric pressure varies with altitude. As a standard atmospheric pressure at sea level  $p_o = 101.325$  kPa. A Pascal is unit of vacuum relating to a force of newtons per metre squared.

An absolute vacuum of 0 kPa is not practically achievable so in real terms we can only approach zero for example on the ANSTO accelerators we have "operating" vacuums better than  $1 \times 10^{-4}$  Pa. In outer space the vacuum is less than  $10^{-12}$  Pa but not 0 Pa as there are some molecules of gas present which exert a very small partial pressure.

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