Processing and Cleaning Techniques for Modern Accelerator Vacuum Systems

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Aims

• To understand why we need to adopt a rigorous cleaning and processing strategy for vacuum especially modern accelerators
• To understand some of the techniques involved
• To understand some of the aspects of quality control for vacuum
Why Do We Need To Clean For Vacuum?

• We may not need to!
• It depends on what we need vacuum for:
  • Vacuum regime required
  • Ultimate pressure
  • Cleanliness
  • So we need to make a proper assessment of the real requirements of the application
• But in general, modern accelerators require good, “clean” vacuum

Some Reasons for Cleaning

• Irrespective of application - manufacturer desires attractive appearance!
• Characteristics of a surface (surface properties) may be altered by ‘contamination’ at the surface.
• Processes may be poisoned by ‘contaminants’
What strategy should be adopted?

• The least that is proved to be effective for the task in hand
• But understand what is required and the limitations of each process
• Design for cleaning
• Pay enormous attention to detail
• Pay enormous attention to health and safety!
Sources of Residual Gas

- Atmosphere
- Vacuum
- Permeation
- Desorption (Thermal + Stimulated)
- Bulk Diffusion
- Backstreaming
- Leaks Real & Virtual

So to Reduce Residual Gas, we must **Inhibit** or **Reduce** these processes.
Outgassing Rates of Materials in Vacuum

- The outgassing rates may vary in order of magnitudes depending on factors: choice of material, **cleaning procedure**, history of material, pumping time, etc...
- Not all materials are compatible with UHV and XHV system!

<table>
<thead>
<tr>
<th>Material</th>
<th>$\eta_t$ (mbar ⋅ lt/s/cm$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminium (fresh)</td>
<td>$9 \cdot 10^{-9}$</td>
</tr>
<tr>
<td>Aluminium (20h at 150°C)</td>
<td>$5 \cdot 10^{-13}$</td>
</tr>
<tr>
<td>Copper (24h at 150°C)</td>
<td>$6 \cdot 10^{-12}$</td>
</tr>
<tr>
<td>Stainless steel (304)</td>
<td>$2 \cdot 10^{-8}$</td>
</tr>
<tr>
<td>Stainless steel (304, electropolished)</td>
<td>$6 \cdot 10^{-9}$</td>
</tr>
<tr>
<td>Stainless steel (304, mechanically polished)</td>
<td>$2 \cdot 10^{-9}$</td>
</tr>
<tr>
<td>Stainless steel (304, electropolished, 30h at 250°C)</td>
<td>$4 \cdot 10^{-12}$</td>
</tr>
<tr>
<td>Stainless steel (316, vacuum fired, 950°C 2-4 hours)</td>
<td>$5 \cdot 10^{-14}$</td>
</tr>
<tr>
<td>Perbunan</td>
<td>$5 \cdot 10^{-6}$</td>
</tr>
<tr>
<td>Pyrex</td>
<td>$1 \cdot 10^{-8}$</td>
</tr>
<tr>
<td>Teflon</td>
<td>$8 \cdot 10^{-8}$</td>
</tr>
<tr>
<td>Viton A (fresh)</td>
<td>$2 \cdot 10^{-6}$</td>
</tr>
</tbody>
</table>
Pumping speed

Pressure $P$ [mbar] in a vacuum vessel is defined by the total gas load, $Q$ [mbar·l/s], and total pumping speed, $S$ [l/s].

In the case of very simple vacuum chamber it is:

$$P = \frac{Q}{S}$$

*Vacuum Plumbers’ Formula 1*

For $Q = 10^{-6}$ mbar·l/s and $S = 100$ l/s the pressure in vacuum chamber:

$$P = 10^{-8} \text{ mbar}$$
α is dependent only on the ratio of length to diameter dimension, and the shape of the cross section of the duct.

For a cylindrical pipe:

<table>
<thead>
<tr>
<th>$L/D$</th>
<th>$\alpha$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>1</td>
</tr>
<tr>
<td>0.5</td>
<td>0.67</td>
</tr>
<tr>
<td>1</td>
<td>0.51</td>
</tr>
<tr>
<td>10</td>
<td>0.11</td>
</tr>
<tr>
<td>50</td>
<td>0.025</td>
</tr>
</tbody>
</table>

It is common in accelerators for the L/D ratio to be large, hence the restriction in transmission probability.

$$C = \alpha C_A$$

Simplest Equation in Vacuum Science:

$$P = \frac{Q}{S}$$

$Q = $ Outgassing Rate  
$P = $ Pressure  
$S = $ Pumping Speed
In general, in particle accelerators, the effective $S$ varies between 1 to 1000 l.s$^{-1}$) while $Q$ can extend over more than 10 orders of magnitude ($\approx 10^{-5} \rightarrow 10^{-15}$mbar l.s$^{-1}$.cm$^{-2}$).

The right choice of materials and treatments is compulsory in the design of vacuum systems (especially those for accelerators).

In this respect the measurement of outgassing rate is an essential activity for an ultra-high vacuum expert.
Cleaning for Accelerators – Why?

• It’s all about the end product, what do we want to achieve....
  ➢Particles to pass through accelerator WITHOUT scattering
  ➢Maintain Satisfactory Lifetime Stored Electron Beam
• Electron Scatter ∝ Atomic Number²

• Reduce Outgassing Rates - Low Presence of High Mass Species
  ➢Hydrocarbons < 0.1% Pump Lubricants < 0.01%

• Stimulated desorption – Usually the MAJOR Gas Load
  ➢Photon Stimulated Desorption (PSD)
  ➢Electron Stimulated Desorption (ESD)
  ➢Ion Impact Desorption
  ➢Increased Thermal Desorption

• Maintain Clean In-Vacuum Surfaces
  ➢Coating Deposition
  ➢Prevent Particle Target Poisoning
  ➢Maintain Efficient Optical Properties for EM Radiation Transport

Cleanliness is an ‘Essential Step’ in achieving this
Requirements for UHV/XHV

• Minimise desorption
  – Remove ‘contaminants’ (i.e. components with high outgassing/vapour pressure)
  – Deplete reservoirs
    • Bulk gases
    • Surface overlayers (e.g. adventitious graphite)
  – Provide barriers – passivation techniques
Vacuum

- Much ado about nothing!
  - Nature abhors a vacuum
  - We have to work quite hard to get low pressures
    - Understand limitations
      - Outgassing
      - Pumping
- There’s nothing in it!

<table>
<thead>
<tr>
<th></th>
<th>Particles m(^{-3})</th>
</tr>
</thead>
<tbody>
<tr>
<td>Atmosphere</td>
<td>(2.5 \times 10^{25})</td>
</tr>
<tr>
<td>Vacuum Cleaner</td>
<td>(2 \times 10^{25})</td>
</tr>
<tr>
<td>Freeze dryer</td>
<td>(10^{22})</td>
</tr>
<tr>
<td>Light bulb</td>
<td>(10^{20})</td>
</tr>
<tr>
<td>Thermos flask</td>
<td>(10^{19})</td>
</tr>
<tr>
<td>TV Tube</td>
<td>(10^{14})</td>
</tr>
<tr>
<td>Low earth orbit</td>
<td>(10^{14})</td>
</tr>
<tr>
<td>(300km)</td>
<td></td>
</tr>
<tr>
<td>SRS/Diamond</td>
<td>(10^{13})</td>
</tr>
<tr>
<td>ALICE</td>
<td>(10^{11})</td>
</tr>
<tr>
<td>Surface of Moon</td>
<td>(10^{11})</td>
</tr>
<tr>
<td>Interstellar space</td>
<td>(10^{5})</td>
</tr>
</tbody>
</table>
Define your requirements

- For most purposes vacuum is just a tool to have when you need it.
- Define what level of vacuum you need and work appropriately.

STFC Daresbury has 8 specification documents:

- Spc-001 – General Definitions
- Spc-002 – General specification for UHV
- Spc-003 – Cleaning of vacuum items
- Spc-004 – Leak Testing of Vacuum vessels
- Spc-005 – Acceptance tests for vacuum vessels
- Spc-006 – Acceptance tests for clean mechanical pumps
- Spc-007 – Material for Vacuum Flanges
- Spc-008 – Standard Vacuum Notes

Ultra High Vacuum Guide

CLRC Daresbury Laboratory
Synchrotron Radiation Department
Vacuum Support Group.

A compendium of Procedures and Specifications
©CLRC Daresbury Laboratory, 1996
Accelerators + Vacuum

• Particle accelerators come in many shapes and sizes and require different vacuum pressures:
  – Small LINACs - $10^{-5} – 10^{-6}$ mbar
  – Medical Cyclotrons
  – Electrostatic
  – Synchrotrons - $10^{-7} – 10^{-8}$ mbar
    • Leptons
    • Hadrons
  – Storage Rings- $10^{-9} – 10^{-10}$ mbar
    • Synchrotron Light Sources
  – Colliders + ERL’s - $10^{-11} – 10^{-12}$ mbar
    • LHC
    • ILC
Quality Control

- Accelerator builders are, in general, always building high precision prototypes which must work to a stringent specification.
- To achieve this, good quality control or quality assurance is essential.
- QA systems such as those set up under standards like ISO 9001 are well established and the mechanical aspects of vessel and component manufacture (e.g. materials, dimensions, tolerances) will fall under their aegis.
Vacuum aspects of quality control are much more nebulous.

The system builder needs to specify exactly what is wanted, how it is to be measured and how it is to be assessed.

There are no “standard” standards.

It is also likely that contractors will need to be educated and vacuum equipment may need to be supplied.

Trained vacuum inspectors will also need to be available.
Quality Control

• First define your standards.
Quality Control

• General vacuum specification
  – Materials
  – Techniques
  – Processes
  – Handling
  – Inspection

• (In addition to vessel drawings, mechanical specification, etc.)
•Standard Cleaning Procedure for Stainless Steel Components

Preclean
1. Remove all debris such as swarf by physical means such as blowing out with a high pressure air line, observing normal safety precautions. Remove gross contamination by washing out, swabbing or rinsing with any general purpose solvent. Scrubbing, wire brushing, grinding, filing or other mechanically abrasive methods may not be used (see 5.2 above).

Wash
1. Wash in a high pressure hot water (approx. 80°C) jet, using a simple mild alkaline detergent. Switch off detergent and continue to rinse thoroughly with water until all visible traces of detergent have been eliminated.
2. If necessary, remove any scaling or deposited surface films by stripping with alumina or glass beads in a water jet in a slurry blaster.
3. Wash down with a high pressure hot (approx. 80°C) water jet, with no detergent, ensuring that any residual beads are washed away. Pay particular attention to any trapped areas or crevices.
4. Dry using an air blower with clean dry air, hot if possible.

Chemical Clean
1. Immerse completely in an ultrasonically agitated bath of clean hot stabilised trichloroethylene for at least 15 minutes, or until the item has reached the temperature of the bath, whichever is longer.
2. Vapour wash in trichloroethylene vapour for at least 15 min minutes, or until the item has reached the temperature of the hot vapour, whichever is longer.
3. Ensure that all solvent residues have been drained off, paying particular attention to any trapped areas, blind holes etc.
4. Wash down with a high pressure hot (approx. 80°C) water jet, using clean demineralised water. Detergent must not be used at this stage.
5. Immerse in a bath of hot (60°C) alkaline degreaser (P3 Almecco™ P36 or T5161) with ultrasonic agitation for 5 min. After removal from the bath carry out the next step of the procedure immediately.
6. Wash down with a high pressure hot (approx. 80°C) water jet, using clean demineralised water. Detergent must not be used at this stage. Ensure that any particulate deposits from the alkaline bath are washed away.
7. Dry in an air oven at approx 100°C or with an air blower using clean, dry, hot air.

Finishing
1. Allow to cool in a dry, dust free area. Inspect the item for signs of contamination, faulty cleaning or damage.
2. Pack and protect as in 5.6.3 above.
Quality Control

• Assessment (Tests)
  – Leak test
  – Performance test
    • Base pressure
    • Outgassing rate
    • Cleanliness
Leak tests

• Specify a realistic leak rate
• Specify testing method
Outgassing test

• Rate of Rise (gas accumulation)

In a sealed chamber, \[ Q = \frac{dP}{dt}_{t=0} \cdot \frac{V}{A} \]
How are we going to achieve it?
## Broad Range of Methods Available

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Thermal Treatment</th>
<th>Polishing</th>
<th>In-Situ Treatment</th>
<th>Others...</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Wash</strong> – Detergent or Solvent</td>
<td>Vacuum Bakeout</td>
<td>Electro-Polish</td>
<td>Vacuum Bakeout</td>
<td>Bead Blasting</td>
</tr>
<tr>
<td><strong>Ultrasonic</strong> – Aqueous or Solvent</td>
<td>Vacuum Fire (typical ~950°C for STST)</td>
<td>Diamond Paste Machine/Manual</td>
<td>UV Lamps</td>
<td>CO2 Snow</td>
</tr>
<tr>
<td><strong>Vapour Clean</strong> – Solvent</td>
<td>Air Bake (up to ~ 400°C)</td>
<td>Plasma Etch</td>
<td>Glow Discharge</td>
<td></td>
</tr>
<tr>
<td><strong>ACID Etch</strong> – Pickling or Passivation</td>
<td>Vacuum Remelt</td>
<td>Diamond Turning</td>
<td>Chemical</td>
<td></td>
</tr>
<tr>
<td><strong>Power Wash</strong> – Water Jet</td>
<td>BCP-Buffered Chemical Polishing</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Human Factors

- Wear gloves!
- Use clean tools.

Work on clean aluminum foil. Cover any chamber openings with foil and clean plastic covers.

Finger prints outgas at the rate of $1 \times 10^{-5}$ mbar Liters per second! Leaving finger prints on UHV components may prevent the chamber from pumping to a low enough pressure. The same goes for anything else that may leave oil on a UHV component.
# Chemical

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Wash – Detergent or Solvent</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Ultrasonic – Aqueous or Solvent</td>
</tr>
<tr>
<td></td>
<td>Vapour Clean–Solvent</td>
</tr>
</tbody>
</table>
## Typical Cleaning Agents

<table>
<thead>
<tr>
<th>Agent</th>
<th>Examples</th>
<th>Advantages</th>
<th>Disadvantages</th>
<th>Disposal</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>Cheap, readily available</td>
<td>Need de-min for cleanliness. Not a strong solvent</td>
<td>To foul drain</td>
<td></td>
</tr>
<tr>
<td>Alcohols</td>
<td>Relatively cheap and readily available. Quite good solvents</td>
<td>Need control – affect workers; some poisonous; some flammable; stringent safety precautions.</td>
<td>Evaporate or controlled disposal.</td>
<td></td>
</tr>
<tr>
<td>CFC’s</td>
<td>Excellent solvents; evaporate easily with low residue</td>
<td>Banned</td>
<td>Strictly controlled, must not be allowed to evaporate.</td>
<td></td>
</tr>
<tr>
<td>Alkaline degreasers</td>
<td>Aqueous solutions, non- toxic. Moderate solvents</td>
<td>Can leave residues and may throw particulate precipitates</td>
<td>Requires neutralisation, then dilution to foul drain.</td>
<td></td>
</tr>
</tbody>
</table>
Science of Cleaning

- **Solvent** - A solvent is a substance that dissolves another substance or substances to form a solution (a homogeneous mixture). The solvent is the component in the solution that is present in the largest amount or is the one that determines the state of matter (i.e. solid, liquid, gas) of the solution.

[Diagrams of solvent action on soil and soil dispersal]
Aqueous & Solvent Cleaning

- **Special Cleaning Techniques**
  Ultrasonic cleaning - widely used

- Heaters
- **Ultrasonic Waves**
- Cavitation bubbles
- Surface
- Contaminant
Cleaning Process

- Full detailed procedure in ASTeC spc-003 - Cleaning of vacuum items

- Auto washers for small items

- Power wash booth for large items
Solvent wash, HFE72DE

- **2 x Solvent cleaning plants:**
  - Model E1500 – 1500mm x 500mm x 500mm
  - Model S3000 – 3000mm x 600mm x 500mm

- **1 x Automatic solvent cleaning plant, model F100.**

  - 70% Trans-dichloroethylene,
  - 10% Ethyl nonafluorobutyl ether,
  - 10% Ethyl nonafluoroisobutyl ether,
  - 5% Methyl nonafluorobutyl ether,
  - 5% Methyl nonafluoroisobutyl ether.
Solvent Cleaning

- Vapour Stage

- Alkaline degreaser
• Hot drying cabinet.
Daresbury Cleaning History

**Orginally**

- CERN UHV Procedures Sufficient (Ultrasonic and Vapour Cleaning)
  - Trichloroethane
  - CFC113 (Freon)
- Alkaline Degreasing (Almeco/CERN)
- Glow Discharge (added following research at Liverpool University)

**1990’s**

- Research Study to find alternative solution due to Environmental Protection Legislation (e.g. Kyoto Protocol)
  - Restricted use of Ozone depleting chemicals
  - Restriction then Ban of Trichloroethane and CFC113

**Research Summary**

- Trichloroethylene selected (comparable to Trichloroethane)
- Aqueous cleaners NOT SUFFICIENT alone but OK in combination with solvent.
- Glow Discharge – Dropped
Replacement of Trichloroethylene

• **What is important to us?** - Thermal outgassing and Stimulated Desorption

\[ Q = \frac{P1 - P2}{A} \cdot C \]

• **Comparative Tests** - existing procedure proven for 20 years
# Cleaning Project Results

<table>
<thead>
<tr>
<th>Cleaning Agent</th>
<th>Net thermal outgassing rate due to residual contaminants (mbar l s(^{-1}) cm(^{-2}))</th>
<th>Hydrocarbon contamination (%)</th>
<th>Ratio of Mass 69 to Mass 28</th>
<th>Pressure rise from ESD (mbar)</th>
<th>Desorption Yield (molecules/electron)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Blank Run (No sample)</td>
<td>8.2 x 10(^{-13}) ± 5.8 x 10(^{-13})</td>
<td>0.46</td>
<td>1.8 x 10(^{-4})</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Trichloroethylene (No contamination)</td>
<td>&lt;2 x 10(^{-12})</td>
<td>0.58</td>
<td>3.2 x 10(^{-4})</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Trichloroethylene (No contamination)</td>
<td>&lt;2 x 10(^{-12})</td>
<td>0.53</td>
<td>8.3 x 10(^{-4})</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Trichloroethylene (Full contamination)</td>
<td>&lt;2 x 10(^{-12})</td>
<td>0.90</td>
<td>8.5 x 10(^{-4})</td>
<td>6.3 x 10(^{-6})</td>
<td>0.055</td>
</tr>
<tr>
<td>Trichloroethylene (Full contamination)</td>
<td>&lt;2 x 10(^{-12})</td>
<td>0.92</td>
<td>5.8 x 10(^{-4})</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>n-propyl bromide 1 – Manufacturer 1</td>
<td>&lt;2 x 10(^{-12})</td>
<td>1.34</td>
<td>6.1 x 10(^{-4})</td>
<td>3.6 x 10(^{-6})</td>
<td>0.29</td>
</tr>
<tr>
<td>n-propyl bromide 2 – Manufacturer 2</td>
<td>6 x 10(^{-12}) ± 2 x 10(^{-12})</td>
<td>2.52</td>
<td>1.9 x 10(^{-3})</td>
<td>2.7 x 10(^{-5})</td>
<td>2.19</td>
</tr>
<tr>
<td>Hydrofluoroether – Experiment 1</td>
<td>&lt;2 x 10(^{-12})</td>
<td>0.52</td>
<td>4.3 x 10(^{-4})</td>
<td>2.1 x 10(^{-7})</td>
<td>0.017</td>
</tr>
<tr>
<td>Hydrofluoroether – Experiment 2</td>
<td>&lt;2 x 10(^{-12})</td>
<td>0.86</td>
<td>2.7 x 10(^{-4})</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Isopropyl alcohol</td>
<td>&lt;2 x 10(^{-12})</td>
<td>0.93</td>
<td>1.0 x 10(^{-3})</td>
<td>4.3 x 10(^{-6})</td>
<td>0.35</td>
</tr>
<tr>
<td>Aqueous cleaner 1</td>
<td>&lt;2 x 10(^{-12})</td>
<td>2.86</td>
<td>1.6 x 10(^{-3})</td>
<td>5.5 x 10(^{-5})</td>
<td>4.46</td>
</tr>
<tr>
<td>Aqueous cleaner 2</td>
<td>1.2 x 10(^{-11}) ± 2 x 10(^{-12})</td>
<td>2.03</td>
<td>1.93 x 10(^{-3})</td>
<td>3.7 x 10(^{-5})</td>
<td>2.99</td>
</tr>
<tr>
<td>Aqueous cleaner 3</td>
<td>&lt;2 x 10(^{-12})</td>
<td>2.70</td>
<td>2.2 x 10(^{-3})</td>
<td>2.6 x 10(^{-5})</td>
<td>2.12</td>
</tr>
</tbody>
</table>
ESD RGA data for HFE and Trike

Trichloroethylene

Hydrofluoroether
Cleaning Process Scientifically Developed

**Publications:**

- Considered aqueous and solvent based cleaning solutions
- Considered main gas loads in an accelerator – Thermal outgassing and stimulated desorption

**Conclusions**
- Aqueous cleaners suitable only for thermal outgassing and not stimulated desorption
- Solvent based cleaners produced better results
- HFE (Hydrofluoroether) based solvent performed best, even better than our previous solvents
Dry Ice Cleaning

• Dry-ice or involves propelling pellets at extremely high speeds
• The pellets sublimate on impact with little energy transferred to the surface minimising any abrasion.
• The sublimation absorbs heat from the surface due to thermal shock. This removes the top layer of dirt/contamination.
• The rapid change in state from solid to gas causes microscopic shock waves which aid the removal of contamination.

• Main Uses:
  – Food industry
  – Semiconductor
  – Aerospace
  – RF structures for accelerators
### Thermal Treatments

<table>
<thead>
<tr>
<th>Thermal Treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vacuum Bakeout</td>
</tr>
<tr>
<td>Vacuum Fire</td>
</tr>
<tr>
<td>(typical ~950°C for STST)</td>
</tr>
<tr>
<td>Air Bake</td>
</tr>
<tr>
<td>(up to ~400°C)</td>
</tr>
</tbody>
</table>
Vacuum Firing

- The manufacturing process for steel means large quantities of H₂ are left in the bulk of the material.
- This H₂ is the limiting factor in achieving the best possible outgassing rates for UHV/XHV systems.
- Vacuum firing (or annealing) is the process by which the material is heated up to high temperature (~950°C) whilst in a vacuum furnace.
- This high temperature heating allows rapid diffusion of the H₂ from the bulk to the surface layers and allows it to escape from the material.
- This process can improve the outgassing rate of stainless steel by up to 2-3 orders of magnitude.
- The high temperature treatment also reduces the magnetic permeability of a material, something which is very useful for accelerators.

Main Uses:
- Used in many industrial sectors as a way of performing processes in a controlled atmosphere (vacuum), the same process in air would lead to oxidation and the addition of contaminants.
Bakeout to moderate temperatures (250°C) is an efficient way of reducing outgassing, especially of water.

In an accelerator, bakeout has to be undertaken with care to ensure temperature gradients are minimised and damage does not occur.

Vessel supports must be designed to accommodate the movements due to thermal expansion and contraction (and so that vessels get back to where they started!)

Bellows are used to accommodate these movements.
• With stainless steel, in machines the most common way to bake is to use wrapped heater tapes and bands with ceramic blanket insulation.

• For simpler vessels, close wrapped Kapton insulated printed heaters with superinsulation can be used to reduce the overall thickness to less than 1mm.

• For aluminium where temperatures used are less than 180°C, superheated water or wrapped film are used.
Bakeout is best performed into external pumps, e.g. turbo pump sets.

Towards the end, during cooldown, hot filament gauges should be degassed, *in situ* TSPs and NEG pumps carefully degassed and conditioned and ion pumps “flashed” for conditioning.

Bakeout should be *monitored* rather than for fixed times – terminating when water in the rga spectrum falls to a predefined level.

Heating and cooldown rates must be carefully controlled.
In situ bakeout is the most commonly used method of achieving UHV in a laboratory environment. Heating to 150-250°C for 12-48 hours is a well-established method for removing water vapor from within your vacuum system.
Bakeout Ex-Situ

Ex situ bakeout is when the possibility of bake out in-situ is not possible. It is quite common on accelerators to NOT have the capability to perform in-situ bakeout. This can be for a variety of reasons – cost, risk, time, some components cannot be baked.

When such a situation arises, how should we solve the problem?

Ex-situ bakeout

This is a method by which vacuum chambers can be prepared for UHV by removing the water vapour and any other unwanted contaminants in a conventional bakeout oven.

We can verify with the use of residual gas analysis that the vacuum chambers are UHV compatible – how? By working to an agreed standard.

CLRC Daresbury Laboratory
Synchrotron Radiation Department
Vacuum Support Group.

Ultra High Vacuum Guide
A compendium of Procedures and Specifications
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<table>
<thead>
<tr>
<th>Line Number</th>
<th>Pressure Region</th>
<th>General Contaminants (%)</th>
<th>Perfluoropolyphenylethers Sum of (peak at 69 and 77 amu) (%)</th>
<th>Chlorinated species Sum of peaks at 35 and 37 amu (%)</th>
<th>Comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>UHV</td>
<td>0.1</td>
<td>0.01</td>
<td>0.01</td>
<td>Assuming system baked.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Calculation to be done at $10^{-9}$ mbar or below</td>
</tr>
<tr>
<td>2</td>
<td>HV - UHV</td>
<td>0.75</td>
<td>0.075</td>
<td>0.075</td>
<td>Assuming system unbaked.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Calculation to be done at $10^{-7}$ mbar or below</td>
</tr>
</tbody>
</table>

What is an acceptable RGA Scan?

The residual gas spectrum MUST have been recorded over 1 – 200 amu.

The limits shown in Table 1 below are expressed in terms of percentages of the total pressure in the system.

The definition of “general contaminants” is the sum of the partial pressures of all peaks present in the residual gas spectrum of mass to charge ratio (amu) equal to 39, 41-43 and 45 and above (excluding any above-45 specifically listed in the table below). Also to be excluded from this summation are any peaks related to the rare gases xenon (i.e. 132, 129, 131) and krypton (i.e. 84, 86, 83).

The level of “general contaminants” in the system shall be calculated. It shall sum all general contaminant peaks as defined in point 3 above and divide this number by the total pressure (excluding peaks at any water peaks at Masses 17 & 18 amu) then multiply by 100 to give the answer as a percentage.

The total pressure MUST be $< 10^{-7}$ mbar or below before the calculation is performed.

There are 2 acceptance criteria as shown in table 1 below:

1) Line 1 assumes the component to be tested has been baked ‘in-situ’ and therefore the vacuum pressure should be below $10^{-9}$ mbar.
2) Line 2 assumes the component to be tested has NOT been baked ‘in-situ’ and therefore the pressure achieved will not reach $10^{-9}$ mbar, however, it must be $< 10^{-7}$ mbar.

Table 1: Acceptable levels of general contaminants for the ESS BTM Project.
Bakeout Ex-Situ

- Following ex situ bakeout and when acceptable standards have been achieved it is critical that the vessel be handled and treated the right way.

- How?

  - Ensure system is vented with a ‘dry’ inert gas to prevent any re-adsorption, typically $N_2$ or Ar are used.
  - Define what is ‘dry’?
  - For accelerators we want to minimise the re-adsorption of water, therefore before venting we measure the dew point of the inert gas down to -70°C.
  - Store the vessel appropriately, sealed off until ready for use.
  - We have experience to show that vessels that have been handled and stored correctly remain suitable for use months later.
  - When ready to use or install the vacuum chamber ensure any exposure to air is minimised to the shortest time practically possible. Also use a ‘dry’ $N_2$ purge to ensure no water ingress from the surrounding air.
Passivation Techniques

• Using barriers to inhibit outgassing
  – Air Baking
  – Electropolishing
  – NEG or TiN coatings

• But note that all of these have some cleaning effect!
• The use of a barriers to inhibit outgassing
  – Coatings
    • NEG
    • TiN
  – Surface modifications
    • Electropolishing
    • Acid Etching
    • Laser modified surfaces
Air Baking

• The simple process of heating a vacuum chamber to a particular temperature in air.

• Typically baked to around 400°C
  - Helps remove H₂ from the bulk but at a lower temperature the rate of diffusion is much lower, therefore not as effective at depleting H₂ reservoirs as vacuum firing
  - Cheaper than vacuum firing
  - Visually the vacuum components have a dull colour

• Forms an oxide layer on the vacuum chamber, this helps minimise the desorption of contaminants from the vacuum surface into the vacuum.
M. Bernardini et al. [7] Hydrogen is most responsible gas of outgassing rates also in SS vacuum chamber. Heating the raw material at 400 °C in air was suggested as a money saving alternative to the classical vacuum heating at 950 °C. In this paper concluded that air bake-out drives out most of the hydrogen absorbed in the bulk stainless steel. Results show that bake-out in air is effective in reducing the hydrogen outgassing rate of a very large stainless steel vacuum chamber. The hydrogen content and the diffusion parameters for a 304 L type stainless steel have been measured by desorption tests on small samples. It is concluded that the effect of the heating treatment in air is mainly to reduce the hydrogen content. Outgassing rate can be decreased with baking of materials as shown in figure 4.

**Figure 4. Hydrogen outgassing rate with and without air baking**[7]

1) Reduces gas desorption:
   - A pure metal (Ti, Zr, V, Hf, etc.) film ~1-μm thick without contaminants.
   - A barrier for molecules from the bulk of vacuum chamber.

2) Increases distributed pumping speed, $S$:
   - A sorbing surface on whole vacuum chamber surface
   
   \[ S = \alpha \cdot A \cdot v / 4; \]

   where $\alpha$ – sticking probability,
   $A$ – surface area,
   $v$ – mean molecular velocity
SEM images of films (film morphology)

- columnar
- dense

- **Columnar layer:**
  - Activated at lower temperature
  - Provides higher sticking probability and pumping capacity

- **Dense layer:**
  - Provides lower ESD

- **Dual Layer:**
  - Combines benefit of both
  - For more details: see A. Hannah’s poster EM286 on Thursday
Dual layer
High SEY materials are a problem in positively charged accelerators.

When a charged particle is accelerated it emits Photons.

These photons release electrons in the wall via the photoelectric effect and also ionise residual gas in the chamber.

For future accelerators the optimum situation is to have a SEY < 1.
To calculate the total SEY we use
\[ \delta = \frac{I_{IF}}{I_{IP}} = \frac{I_{IF}}{I_{IF}} + I_{IS} \]

Where:
- \( \delta \) is the total SEY
- \( I_{IF} \) is the current on the cup
- \( I_{IS} \) is the secondary electron current
- \( I_{IP} \) is the beam current

SEY measurement facility
• Surfaces have a micro and nanostructure
• Optically black
• Various laser parameters can be varied to change the topology
• Such modified surfaces increase surface area and therefore outgassing is likely to be higher
• Surface resistance is also increased – a potential issue for accelerators
Polishing techniques

• For use in vacuum polishing techniques are often employed.
• Polishing effectively reduces the surface area, if we reduce the surface area then we potentially reduce the outgassing rate.
• This may NOT always be the case, polishing can actually grind contaminants into the surface or leave particular species in the subsurface layers – mainly $\text{H}_2$
• For polishing techniques to be completely effective they are often finished with some additional technique – mainly heating
• Electropolishing followed by vacuum firing can produce outgassing rates in $10^{-14}$ mbar l/s/cm² range and give a nice surface finish but the electropolishing doesn’t improve significantly the outgassing rate compared to just vacuum firing.

• Polishing techniques are often used for additional purposes and not necessarily for vacuum performance
Buffered Chemical Polish

- In order to remove any defects or damage to the surface, an acid etch is applied to the cavities

  → Buffer Chemical Polish (BCP) removes 100-150µm

- Acid mixture
  - Hydrofluoric acid; HF (49%)
  - Nitric Acid; HNO₃ (65%)
  - Phosphoric Acid; H₃PO₄ (85%)
  - In a 1:1:1 mixture

- Risk of hydrogen contamination
  - Correct mixture should be used
  - Temperature of acid should be kept below <18 °C, to control the exothermic reaction
  - Vacuum processing required

- Cavity is the high pressure rinsed (HPR) with ultrapure water
• Work piece acts as the anode
• A current passes from the anode & the surface is oxidised and dissolved into the electrolyte.
• At the cathode Hydrogen is produced as a by product
Electropolishing achieves a smoother finish than BCP and typically higher gradients.

The cavity is an anode and an aluminium cathode is immersed in an electrolyte.

Again hydrogen is produced so vacuum processing and HPR are required.
Current and Future Challenges

- Currently Developing XHV and Low Particle Processing Techniques

  - Use of SRF ($P_T < 10^{-10} \text{ mbar}$, low levels of particles and surface contaminants)

  - Requirements for High Average Current Photoinjectors ($P_T < 10^{-11} \text{ mbar}$, $P_{O_2} < 10^{-14} \text{ mbar}$, low levels of particles and surface contaminants)

  - Reduce gas density in region of photoinjector

  - E.g. To reduce ion back bombardment on photocathode material and to prevent cathode poisoning. May lead to reduced QE.
Dust particles in a vacuum chamber

- The dust micro-particle in the beam vacuum chamber might be ionised by photons or photoelectrons and then be trapped by the beam electric field.
- This may cause the significant loss of the beam.

Potential sources of the dust micro-particles:
- Dust from the atmosphere during storage, installation or venting
- Dust from moving parts: manipulators, bellows, valves, etc
- Micro-particles from getters, cryosorbers
- Micro-particles from working IP.

How to avoid:
- Proper cleaning and storing
- Positioning of potential dust sources in regard to the beam
- Clean environment when vacuum chamber is open
- Clean gas for venting (for example, boil-off nitrogen)
Particle Control

• Systems of flushing and counting particles

• Use of Clean Hoods and Clean Rooms

• Careful Design to Minimize Particle Sources or Position Them Safely away from Beam.

• Careful Selection of in-vacuum components

• Use of gas filters during let up

• Controlled gas flow (pump down/letup speed)

• Good Cleaning Procedures
Summary

- General factors affecting Vacuum
- Considerations for cleaning – why we need it, define your specification
- Cannot increase pumping speed massively but can reduce outgassing rates considerably
- Demonstrated why cleaning is so important for UHV/XHV in reducing outgassing rates
- Discussed the importance of quality control
- Reviewed various processes which are known to affect vacuum performance
- Introduced particle control procedures