Development of Cleaning and Processing Techniques for UHV/XHV at Daresbury Laboratory

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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
Sources of Residual Gas

So to Reduce Residual Gas, we must **Inhibit** or **Reduce** these processes.
Conductance Limitations in a vacuum system

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>( \frac{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>0.25</td>
</tr>
</tbody>
</table>

It is common in accelerators for the \( \frac{L}{D} \) ratio to be large, hence the restriction in transmission probability.

\[
C = \alpha C_A
\]

\[
\frac{1}{S_{eff}} = \frac{1}{S_0} + \frac{1}{C}
\]

Simplest Equation in Vacuum Science:

\[
P = \frac{Q}{S}
\]

Q = Outgassing Rate
P = Pressure
S = Pumping Speed
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!

The example of the outgassing rates after one hour pumping:

<table>
<thead>
<tr>
<th>Material</th>
<th>$\eta_t$ (mbar \cdot lt/s/cm$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminium (fresh )</td>
<td>9$\times$10$^{-9}$</td>
</tr>
<tr>
<td>Aluminium (20h at 150°C)</td>
<td>5$\times$10$^{-13}$</td>
</tr>
<tr>
<td>Copper (24h at 150°C)</td>
<td>6$\times$10$^{-12}$</td>
</tr>
<tr>
<td>Stainless steel (304)</td>
<td>2$\times$10$^{-8}$</td>
</tr>
<tr>
<td>Stainless steel (304, electropolished)</td>
<td>6$\times$10$^{-9}$</td>
</tr>
<tr>
<td>Stainless steel (304, mechanically polished)</td>
<td>2$\times$10$^{-9}$</td>
</tr>
<tr>
<td>Stainless steel (304, electropolished, 30h at 250°C)</td>
<td>4$\times$10$^{-14}$</td>
</tr>
<tr>
<td>Perbunan</td>
<td>5$\times$10$^{-6}$</td>
</tr>
<tr>
<td>Pyrex</td>
<td>1$\times$10$^{-8}$</td>
</tr>
<tr>
<td>Teflon</td>
<td>8$\times$10$^{-8}$</td>
</tr>
<tr>
<td>Viton A (fresh)</td>
<td>2$\times$10$^{-6}$</td>
</tr>
<tr>
<td>Neoprene</td>
<td>3$\times$10$^{-4}$</td>
</tr>
</tbody>
</table>
### 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>Wash – Detergent or Solvent</td>
<td>Vacuum Bakeout</td>
<td>Electro-Polish</td>
<td>Vacuum Bakeout</td>
<td>Bead Blasting</td>
</tr>
<tr>
<td>Ultrasonic – Aqueous or Solvent</td>
<td>Vacuum Fire (typical ~950C for STST)</td>
<td>Diamond Paste Machine/Manual</td>
<td>UV Lamps</td>
<td>CO2 Snow</td>
</tr>
<tr>
<td>Vapour Clean – Solvent</td>
<td>Air Bake (up to ~ 400C)</td>
<td>Plasma Etch</td>
<td>Glow Discharge</td>
<td></td>
</tr>
<tr>
<td>ACID Etch – Pickling or Passivation</td>
<td>Vacuum Remelt</td>
<td>Diamond Turning</td>
<td>Chemical</td>
<td></td>
</tr>
<tr>
<td>Power Wash – Water Jet</td>
<td></td>
<td>BCP-Buffered Chemical Polishing</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Cleaning – 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 \( \propto \) Atomic Number\(^2\)**
- **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
How do we determine cleanliness?

<table>
<thead>
<tr>
<th>Pressure Region</th>
<th>General Hydrocarbon Contaminants</th>
<th>Perfluoropolyphenylethers (Fomblin based oils)</th>
<th>Chlorinated Species</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>UHV</td>
<td>0.1%</td>
<td>0.01%</td>
<td>0.01%</td>
<td>After Bakeout</td>
</tr>
</tbody>
</table>
| XHV             | 0.01%                           | 0.001%                                        | 0.001%              | At 10^{-10} mbar
|                 |                                 |                                               |                     | 0.001% is 10^{-15} mbar |

- General hydrocarbon contaminants are the sum of all partial pressures above mass 39, excluding 40, 44 and any noble gases Kr (84, 86, 83) or Xenon (132, 129, 131)
Replacement of Trichloroethylene

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

\[ Q = \frac{P_1 - P_2}{A} \cdot C \]

- **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><strong>0.055</strong></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(^{-2})</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><strong>0.017</strong></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>
‘Typical’ Thermal Outgassing RGA Spectra
ESD RGA data for HFE and Trike

**Trichloroethylene**

**Hydrofluoroether**
ESD RGA data for Aqueous and Solvent (HFE)

Hydrofluoroether

Aqueous Cleaner 1
Cleaning Project ESD Results – N-propyl bromides

N-propyl bromide 1

N-propyl bromide 2
Cleaning Project ESD Results – Aqueous Cleaners

Aqueous cleaner 2

Aqueous cleaner 3
Summary of Findings

- Some Modern **Aqueous Cleaners** clean as well as solvents when considering Room Temperature Outgassing.

- **ESD Data** – Solvents Much Better

- **HFE** (Hydrofluoroether – 3M™ Novec™) – Preferred Trike replacement

- Reference:- Phase 1 – *Vacuum 81 (2007) 793-798*  
  Phase 2 – In Print

*
Current and Future Challenges

• Currently Developing XHV and Low Particle Processing Techniques

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

  ➢ Requirements for High Average Current Photoinjectors ($P_T < 10^{-12}$ mbar, $P_{O_2} < 10^{-15}$ mbar, low levels of particles and surface contaminants)

  ➢ Reduce gas density in region of photo-injector
  ➢ E.g. To reduce ion back bombardment on photocathode material and to prevent cathode poisoning. May lead to reduced QE.
ALICE Energy Recovery Linac

LINAC: Superconducting RF Cavity to 35MeV

- Nominal gun energy: 350 keV
- Injector energy: 8.35 MeV
- Circulating beam energy: 35 MeV
- Linac RF frequency: 1.3 GHz
- Bunch repetition rate: 81.25 MHz
- Max bunch charge: 80 pC
- Bunch train: 100 µs
- Maximum average current: 13 µA

Gun: High brightness photocathode electron source
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 (HFE)
ALICE Gun and Power Supply

- Anode Plate
- Gun Power Supply
- Cathode Ball
- Ceramic
- GaAs Cathode
- ALICE Gun and Power Supply

Diagram showing various components of the ALICE Gun and Power Supply system.
The Insulating Ceramic & Cathode Ball
Activation of GaAs cathode

- **Gun Activation process:**
  - Heat cycle of cathode to 580°C
  - Deposit alternate layers of Cs and Oxygen onto ‘clean’ GaAs surface until maximum QE is achieved.
  - All done within the gun vacuum
Vacuum – A critical parameter

- Once activated GaAs is our true measure of vacuum performance
- Once activated the QE has a finite lifetime (1/e decay)
- Lifetime affected by 2 key parameters:
  - Vacuum environment (H₂, CH₄, N₂, CO, O₂, CO₂)
  - Ion back bombardment
- Ultimate goal is to have extremely low base pressure and control of the residual gas species within the vacuum chamber.
- How do we achieve this:
  - Improve vacuum processing (H₂, CH₄, N₂, CO, O₂, CO₂)
  - Improve the design
Vacuum Bake Specification

• Detailed procedures were put in place for cleaning and baking of all components.

• Initially an in-situ bake of the photoinjector was carried out at 200°C for a period until a < 10% pressure drop over 24 hours was observed.

• During the 1st phase of photoinjector commissioning problems were encountered with cathode lifetimes and cathode QE.

• Evidence suggests that partial pressures of any oxygen containing species (CO, CO$_2$ and H$_2$O) should be < 10$^{-14}$ mbar.

• A new in-situ bakeout procedure was developed which monitored the ratio of gas species in the vacuum system during the bake.

• The criteria was to ensure a minimum of 3 orders of magnitude in pressure between H$_2$ and any oxygen containing species at 250°C.
Vacuum Bake Specification

- During the early bakeouts on the photoinjector, the criteria was to achieve a < 10% pressure drop over 24 hours. RGA scan shown left.

\[ y = 2.6105e^{-0.0098x} \]

1/e lifetime = 102 hours
Vacuum Bake Specification

Monitoring of RGA data during the bakeout process in order to reduce oxygen containing species.

RGA scan at 250°C

1/e lifetime = 891 hours

RGA scan at RT
Base Pressure = 6 x 10^{-12} mbar
In-situ bakeout monitoring for XHV now standard

- The scan opposite shows an in-situ bakeout scan observed on the ALICE gun with significant contamination. This led to a change in our procedures as the gun had to be completely stripped and re-cleaned causing a delay of 3 months in the program.

- Turned out the NEG heaters had been processed with components from another job and became contaminated. The contamination was only visible at bakeout temperature.

- The trend scan opposite shows an ALICE component being baked offline and the trends are monitoring for the main ‘hydrocarbon’ species throughout the complete bake cycle to ensure no contamination is observed.
Summary

- General factors affecting Vacuum
- Conductance limitations in Vacuum
- Cannot increase pumping speed massively but can reduce outgassing rates considerably
- Demonstrated why cleaning is so important for UHV/XHV in reducing outgassing rates
- Results of recent studies show solvents are a much better solution than aqueous cleaners for our application
- Introduced particle control procedures
- XHV requirements for a GaAs photocathode
- In-situ monitoring of RGA data now a requirement for XHV.