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Materials for Vacuum and the Processing and Cleaning Techniques for Modern Accelerator Vacuum Systems

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Part 1 – Materials for Vacuum Use

Mechanical Properties for Vacuum Vessels

- Relevant Mechanical properties
  - Strength (over desired range of temperatures)
  - Hardness
  - Expansion coefficients
  - Machining and joining properties
  - Corrosion resistance
- Relevant Physical Properties
  - Electrical conductivity
  - Thermal conductivity
  - Magnetic properties
  - Permeability
  - Residual Activity

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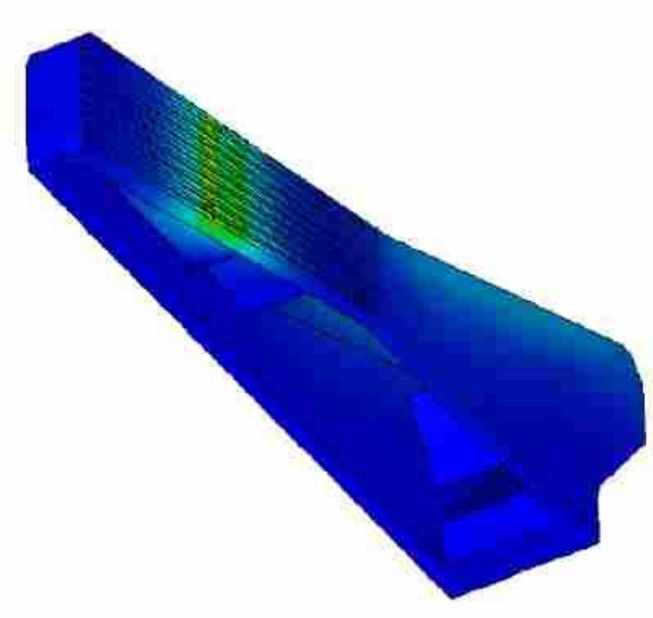
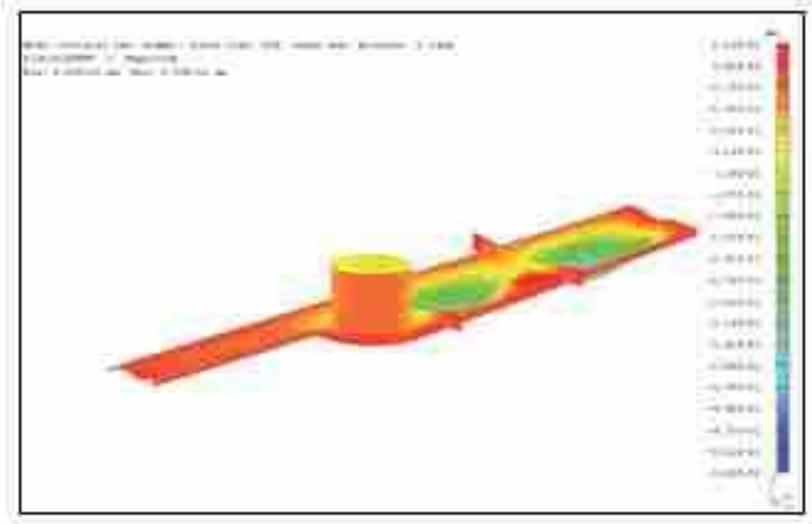
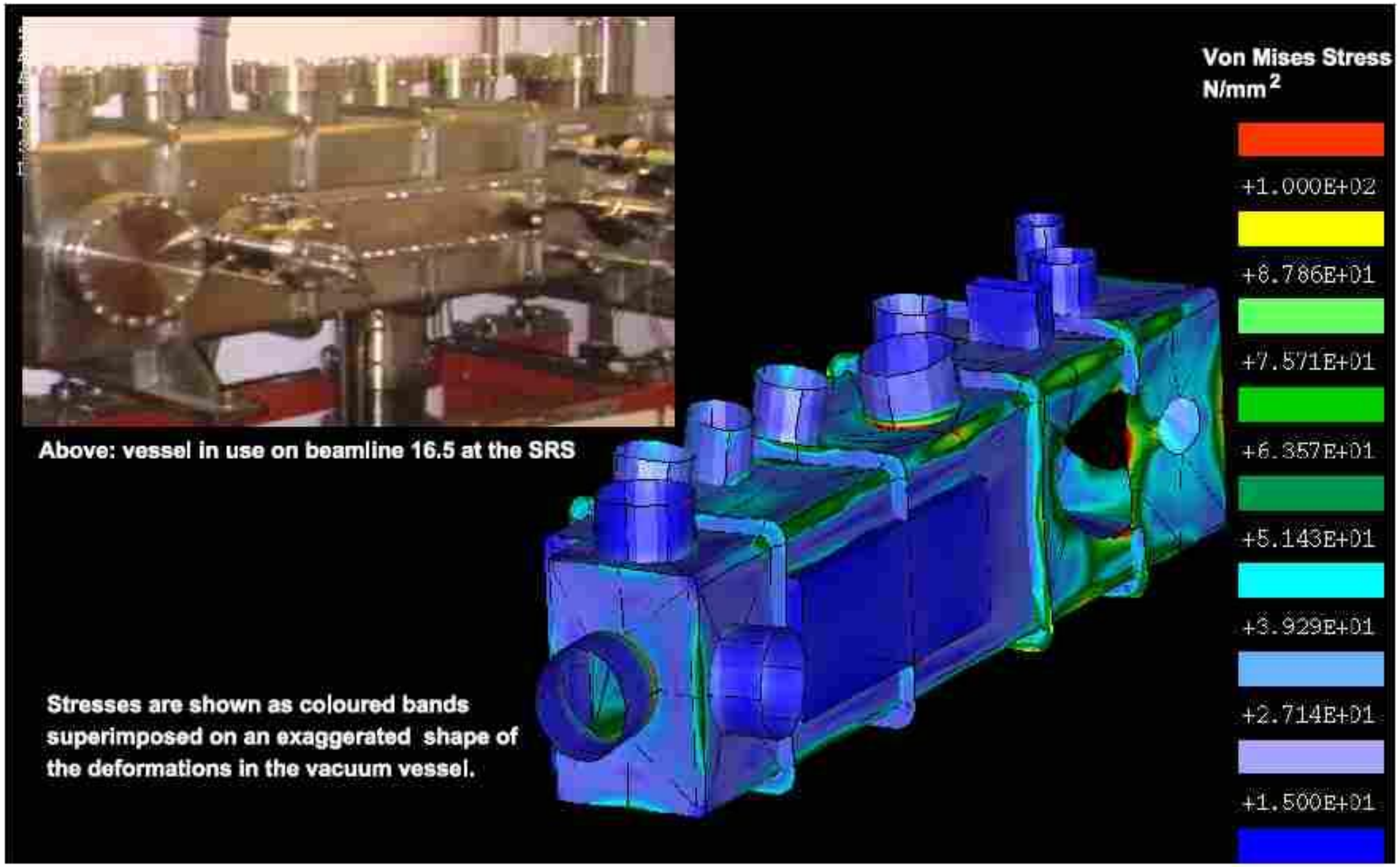
Mechanical Properties

- Wall loading is ~10.4 kg m^2
  - Need to consider deflection of thin wall vessels
  - FEA calculations





Mechanical Properties



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Machining and Joining Properties

- Fabrication
  - Sheet metal work
  - Cutting, milling, turning
  - Sintering, hipping
- Joining
  - Welding – conventional (TIG); electron beam, laser, plasma
    - Distortion
  - Brazing
  - Bonding – gluing, diffusion

Physical Properties

- Electrical conductivity
  - Continuity, impedance
  - Insulation
- Thermal conductivity
  - Bakeout
  - Cryogenic
  - Beam/photon stops
- Magnetic properties
  - Weld regions

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Some Suitable Materials (Vessels)

- Metals
  - Stainless Steel – AISI 304, L, LN; 316, L, LN
  - Aluminium – 4043 (5% Si)
    5052 (2.5% Mg, 0.25% Cr)
    6061(0.25% Cu, 0.6% Si; 1% Mg, 0.2% Cr)
    6063 (0.5% Si, 0.1% Cu,Mn,Zn,Ti,Cr, 0.8% Mg)
  - Copper (especially high strength with e.g. 2% Be)
  - Titanium
- Ceramics – Alumina, Beryllia

Some Suitable Materials (Internal)

- All materials shown for vessels
- All refractory metals
- OFHC and OFS Copper
- Copper and aluminium bronzes
- Glidcop®
- Gold, many alloys, silica, glass, etc

- Avoid brass, high sulphur and phosphorus containing alloys.

Part 2 – Material Properties in Vacuum

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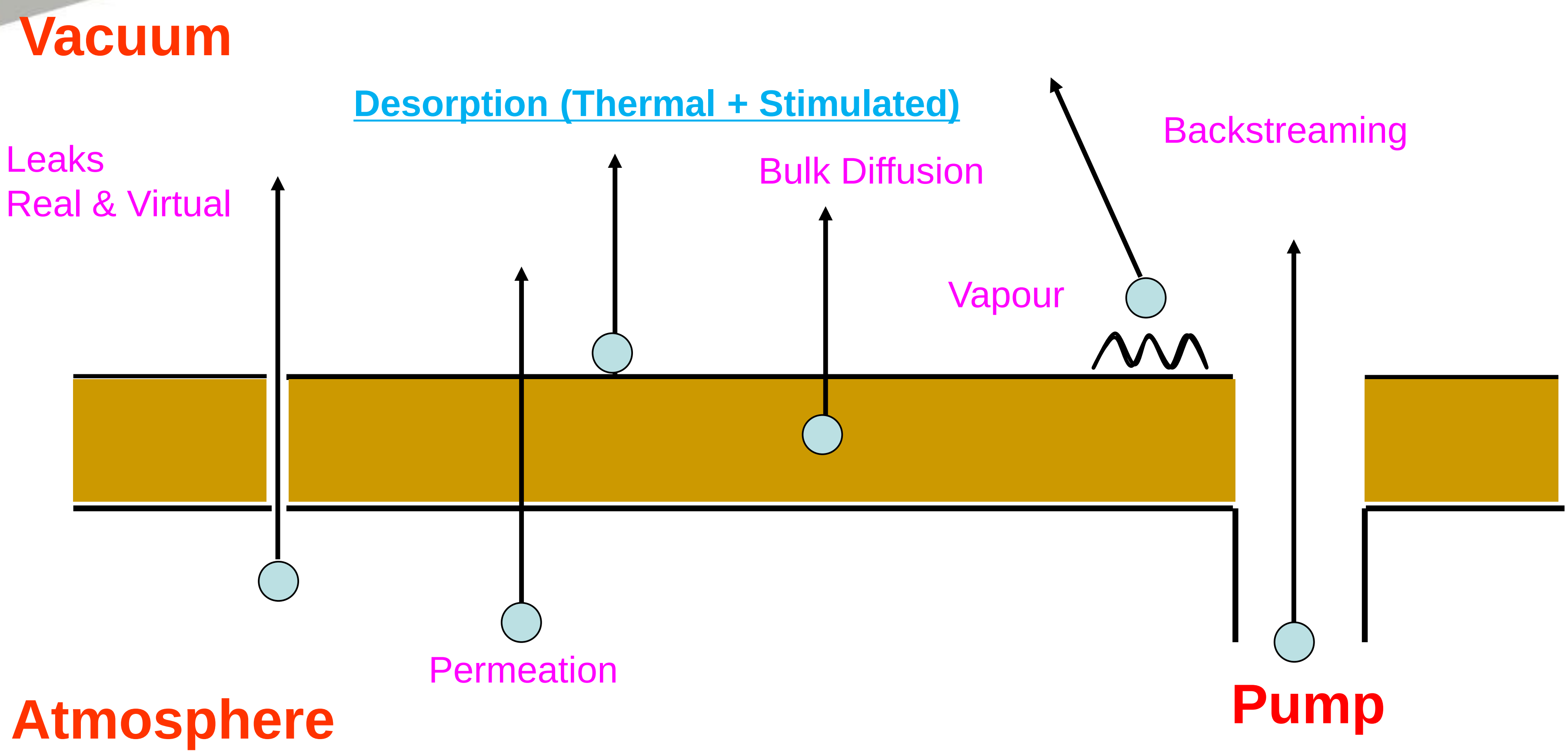
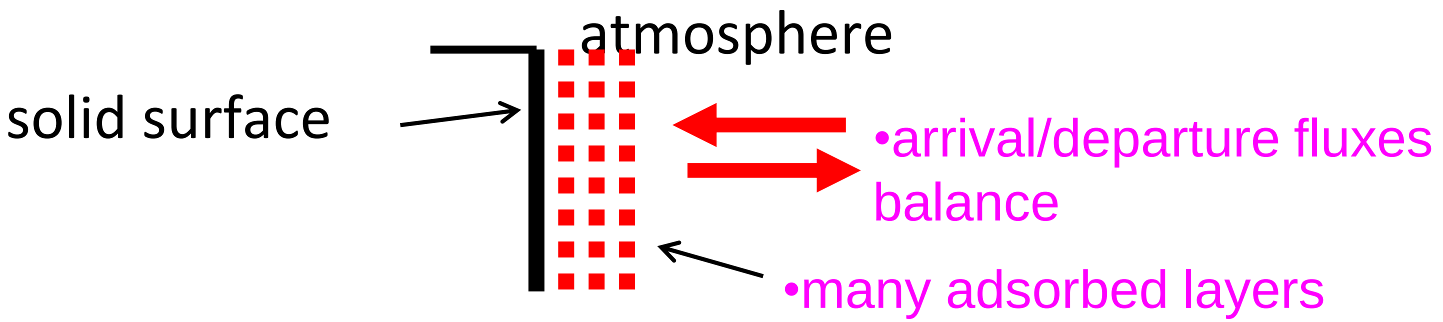
Sources of Residual Gas



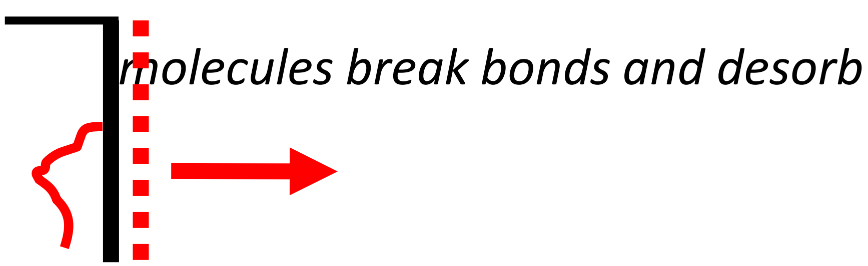
- **So to Reduce Residual Gas, we must <u>Inhibit</u> or <u>Reduce</u> these processes**

Why Outgassing happens …….

- at atmospheric pressure, there is equilibrium….



- with atmosphere removed, adsorption onto surface ceases, flows no longer balanced   …. outgassing

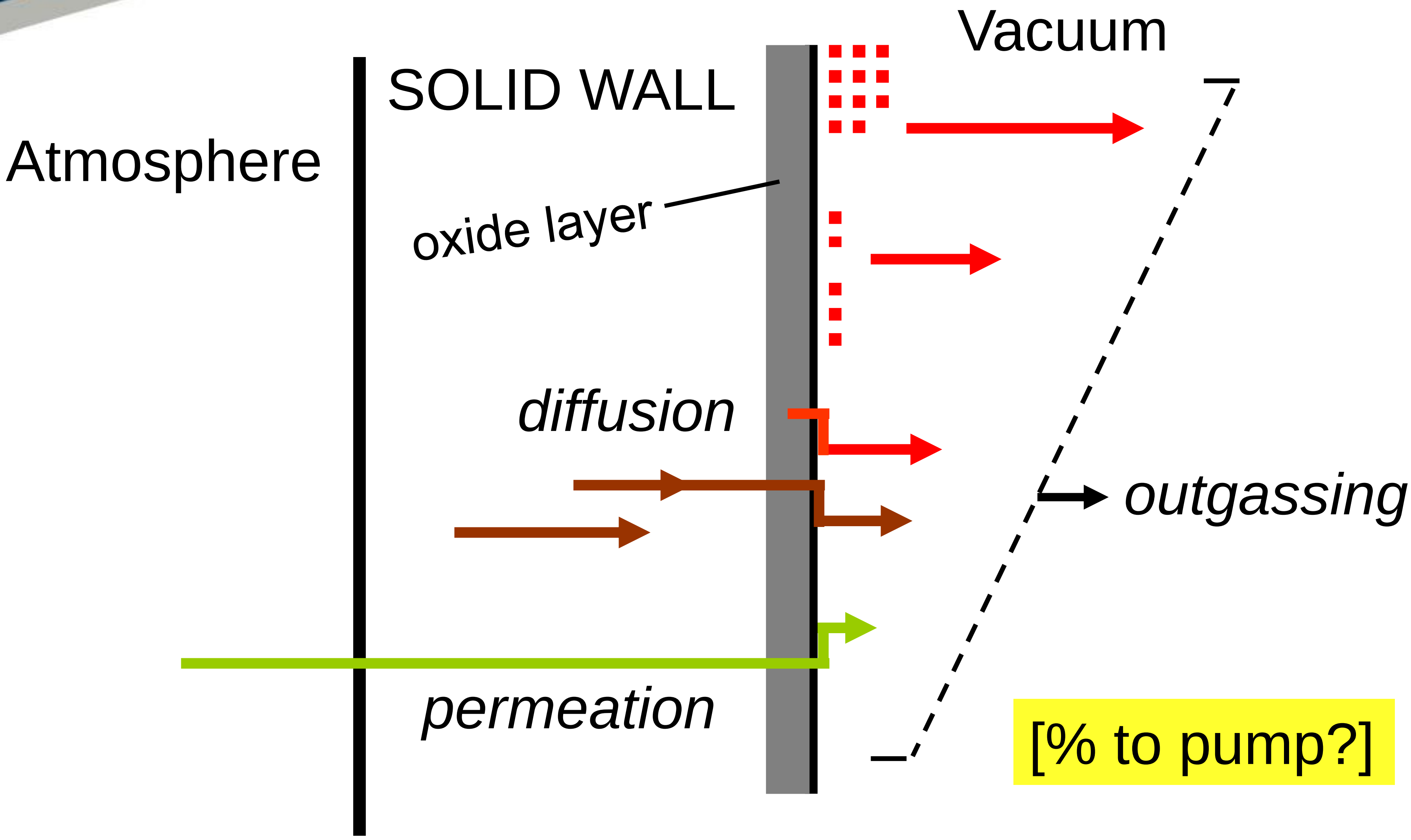


Strength of the adsorptive bond ….

- if weak, soon gone
- if strong, stay bound, (desorbing v slowly at insignificant rate)
- UNFORTUNATELY, *water* molecules adsorbed in large amounts (from ambient surroundings when surfaces are at atmospheric pressure), and with a bond strength that causes its release at a problematic rate:

  outgassing rate, $q_G \sim 10^{-7}$ mbar·l/(s·cm^2) (after 1 hour pumping)

Supply of Gas



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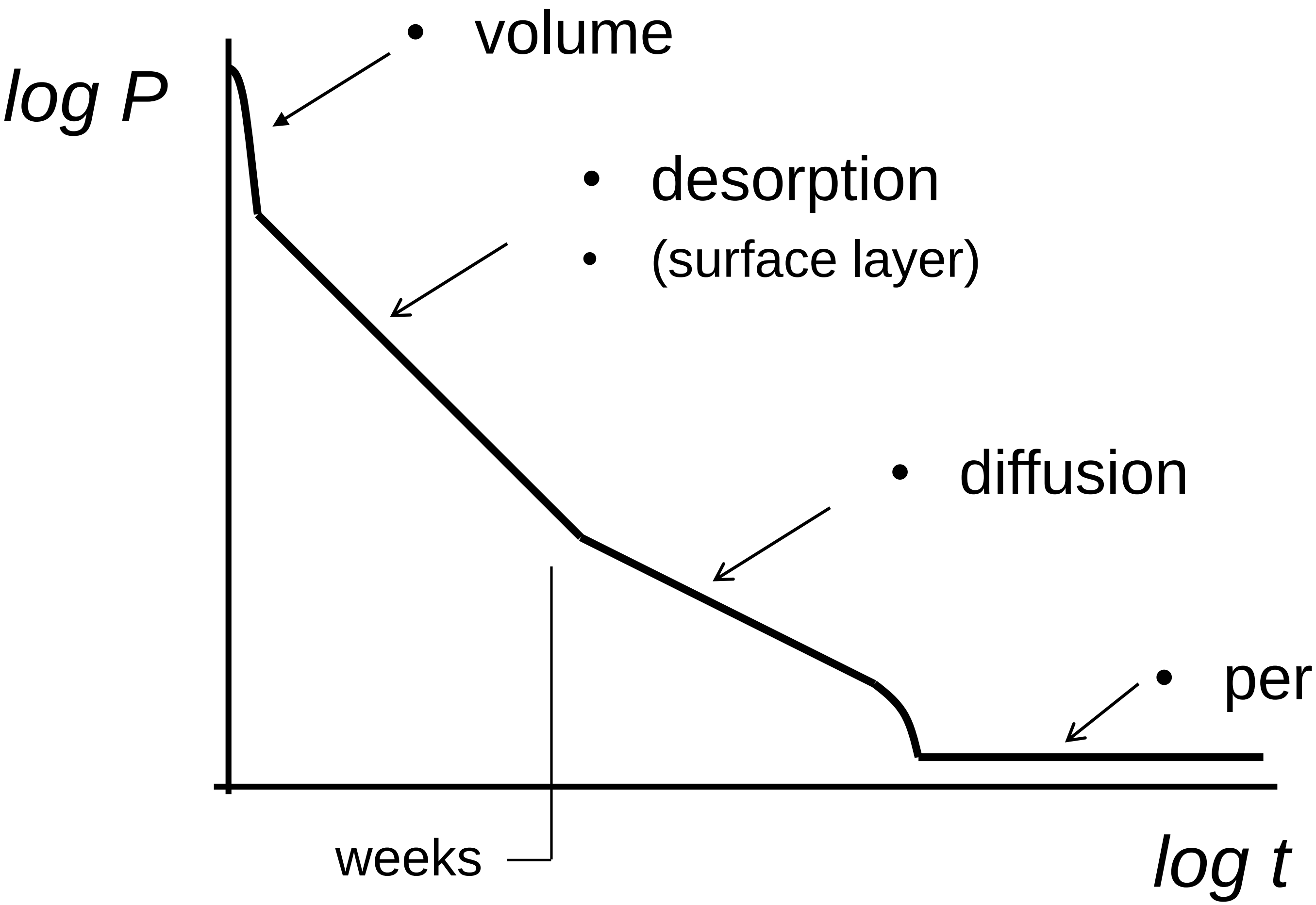
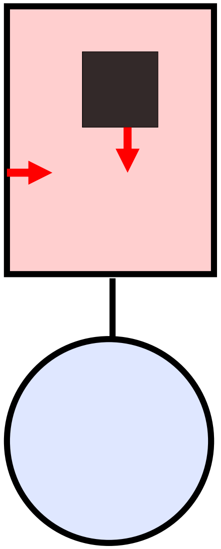
Supply of gas





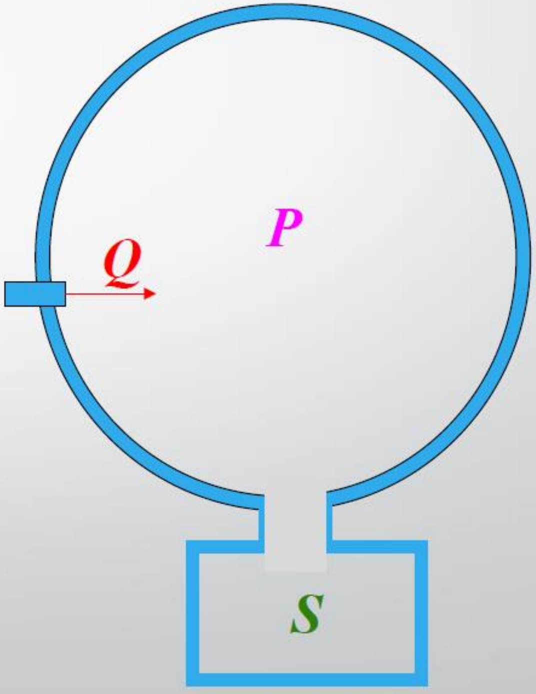
Pumping speed

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



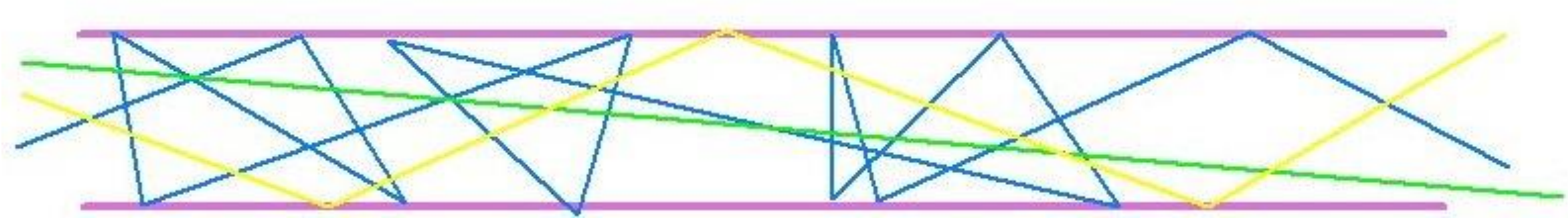
For ***Q*** = 10^{-6} mbar·l/s and ***S*** = 100 l/s the pressure in vacuum chamber:

P = $\mathbf{10^{-8}}$ mbar

How do we remove Gas?

- Pumping
- Rough vacuum
  - "Sucking"
- Medium to High vacuum - < 10^{-3} mbar
  - Pumps do not suck!
  - Gas molecules are acting independently
  - As vacuum specialists we 'design' the system so the pump is in the right location and the molecules find their way to the pump!

Conductance Limitations



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:

$^L/_D$	α
0	1
0.5	0.67
1	0.51
10	0.11
50	0.025

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:

$$P = Q/S$$

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!

Material	η_t (mbar ·lt/s/cm^2)
Aluminium (fresh)	$9\cdot10^{-9}$
Aluminium (20h at 150°C)	$5\cdot10^{-13}$
Copper (24h at 150°C)	$6\cdot10^{-12}$
Stainless steel (304)	$2\cdot10^{-8}$
Stainless steel (304, electropolished)	$6\cdot10^{-9}$
Stainless steel (304, mechanically polished)	$2\cdot10^{-9}$
Stainless steel (304, electropolished, 30h at 250°C)	$4\cdot10^{-12}$
Stainless steel (316, vacuum fired, 950°C 2-4 hours)	$5\cdot10^{-14}$
Perbunan	$5\cdot10^{-6}$
Pyrex	$1\cdot10^{-8}$
Teflon	$8\cdot10^{-8}$
Viton A (fresh)	$2\cdot10^{-6}$

Outgassing rate v 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** (≈$10^{-5} \rightarrow 10^{-15}$ mbar l/s/ 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.

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Part 3 – Processing Techniques for improved vacuum performance

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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 Number^2**

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

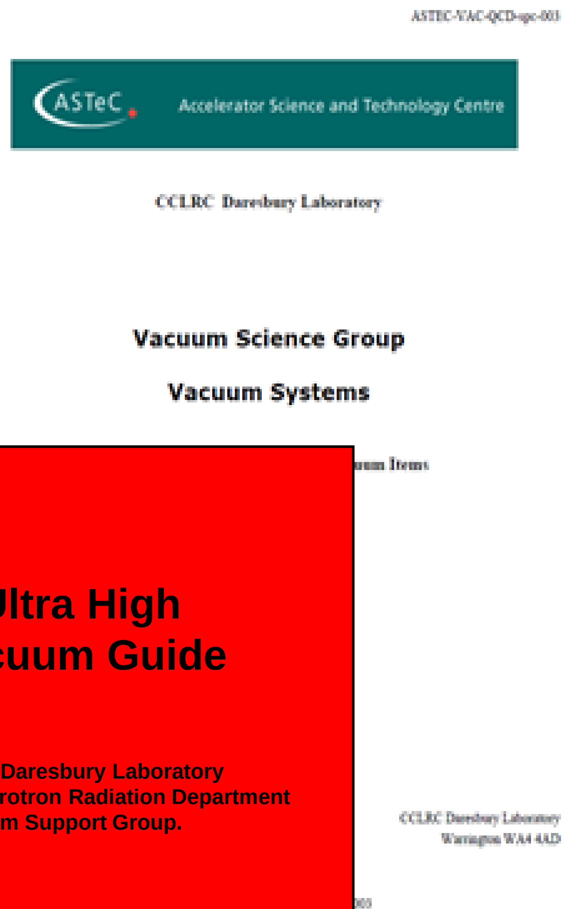
- **Stimulated desorption – Usually the MAJOR Gas Load**
  - Photon Stimulated **Desorption** (PSD)
  - Electron Stimulated **Desorption** (ESD)
  - Ion Impact **Desorption**
  - Increased Thermal **Desorption**

- **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**

Define your requirements

- F … is just a
- N … t to hav
- D … n you ne … o
  i … rds.
- S … ocuments:
  - JHV
  - essels
  - um vessels
  - mechanica
  - ges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ASTeC Accelerator Science and Technology Centre

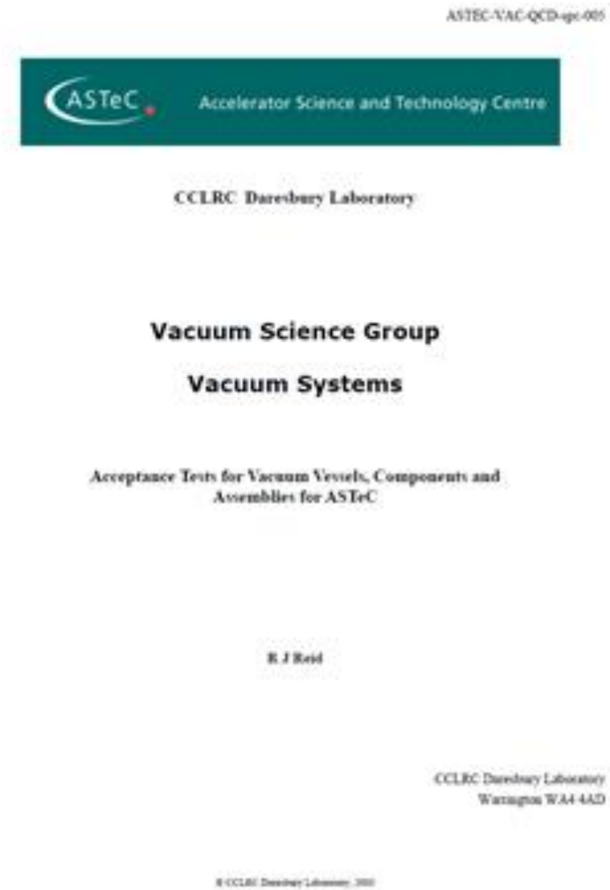
CCLRC Daresbury Laboratory

Vacuum Science Group

Vacuum Systems

…um Items

CCLRC Daresbury Laboratory
Warrington WA4 4AD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ASTeC Accelerator Science and Technology Centre

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Vacuum Science Group

Vacuum Systems

Acceptance Tests for Vacuum Vessels, Components and Assemblies for ASTeC

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Ultra High Vacuum Guide

CLRC Daresbury Laboratory
Synchrotron Radiation Department
Vacuum Support Group.

A compendium of Proceedures and Specifications
<Issue 3>
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Quality Control - Vacuum Database

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ASTeC - Vacuum Solutions

All Job Cards

Return to : DLAppWebServices Main Page   **Tuesday, December 1, 2020**   **Welcome : Keith Middleman (kjm56)**   Logout

New Job Card | Outstanding Jobs | Completed On Time | Return to Main Menu

Job Number		**Job Card Process**	Select job card type...
DM Officer		**DM Officer**	Select from existing DM Officers...
Date In		**Date In Month**	Select Date In Month...
Description		**Date In Year**	Select Date In Year...
Job Card Priority	Select....	**Select Job CardOrder**	Select Order...
Query Job Cards	i*		Export Results to Excel

Number of Records : 183.

Incompleted and missed required date Job Cards, show date required in red text

Job No	Demanding Officer	Date In	Date Req	Date Comp	Booking Code	Job Description	Priority Status
184	Hanley, Thomas, TF	30/11/2020	07/12/2020		STJA08106 05.04	tee fitting x 5 / elbow fittings x 5	Normal
183	Sian, Taaj, TS	30/11/2020	01/12/2020		STGA00500 01	ALUMINIUM SAMPLE PLATE....	Urgent
182	Conlon, James, JA	27/11/2020	03/12/2020	01/12/2020	STGA00500 01	CRYOLAB; 1 X KF 16 FLEX BELLOWS....	Normal
181	Conlon, James, JA	27/11/2020	30/11/2020	01/12/2020	STGA00028 01	VSG; VISTA; 1 X 8" WINDOW / 1X 8"BLANK....	Urgent
180	Foster, Rhys, RM	26/11/2020	04/12/2020		STGA09000 200	CLARA; 6 x YAG holders....	Normal
179	Foster, Rhys, RM	26/11/2020	30/11/2020		STGA09000 200	CLARA; (m5 x 12) x 100 / m4x12 set screw) x 100 / (5x12 dowel) x 50 / (m2x 12)x 100 /....	Normal
178	Clarke, Katherine, KC	26/11/2020	03/12/2020		STFC00470 01	CARME; Rest of Cable harnesses to be cleaned and baked. x3 one bolt is separate on one h....	Urgent
177	Bladen, Luke, LK	25/11/2020	27/11/2020	26/11/2020	STJA08106 05.01	ESS LWU 16-003 Vacuum Checks Outer Cleanroom....	Elevated
176	Murphy, John, JTM	01/11/2020	30/11/2020			John Nov 20....	Normal
175	Conlon, James, JA	23/11/2020	01/12/2020	01/12/2020	STGA00501 01	VSG; VISTA; s x CF38 window 1 x CF38 spacer....	Normal
174	Headspith, Alex, AD	17/11/2020	01/12/2020		STFC00470 01	CARME; Cable harness for ISOL UHV chamber - 5 off. K. Middleman for process requirements....	Urgent
173	Foster, Rhys, RM	18/11/2020	30/11/2020	30/11/2020	STGA09000. 200	CLARA; 14x viewports DN63 1x 114>70 ZL 5x 6>4 1/2 ZL....	Normal
172	Bladen, Luke, LK	17/11/2020	20/11/2020	19/11/2020	STJA08106 05.04	1 zero legth flange 8 inc[illegible]	Elevated
171	Birkenhead, Jacob, JT	17/11/2020	03/12/2020		STJA08106 05.04	ESS, Beam Transport Pr[illegible]nge 4 x 6 inch flange 5 x 4 inch flange 4 x....	Normal

Screenshot

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

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Quality Control

- General vacuum specification
  - Materials
  - Techniques
  - Processes
  - Handling
  - Inspection
- (In addition to vessel drawings, mechanical specification, etc.)

Accelerators + Vacuum

•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$_{o}$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$_{o}$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 Hydrofluoroethee for at least 15 minutes, or until the item has reached the temperature of the bath, whichever is longer.

2.Vapour wash in Hydrofluoroether 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$_{o}$C) water jet, using clean demineralised water. Detergent must not be used at this stage.

5.Immerse in a bath of hot (60$_{o}$C) alkaline degreaser (P3 Almeco™ 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$_{o}$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^{o}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.

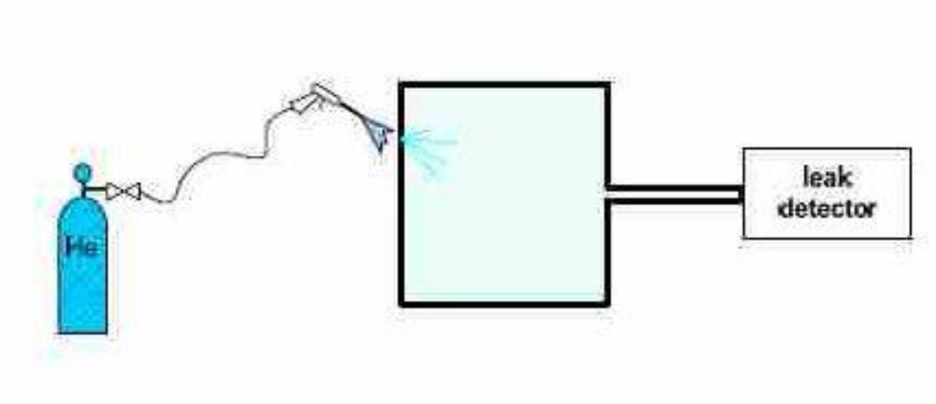
2.Pack and protect as in 5.6.3 above.

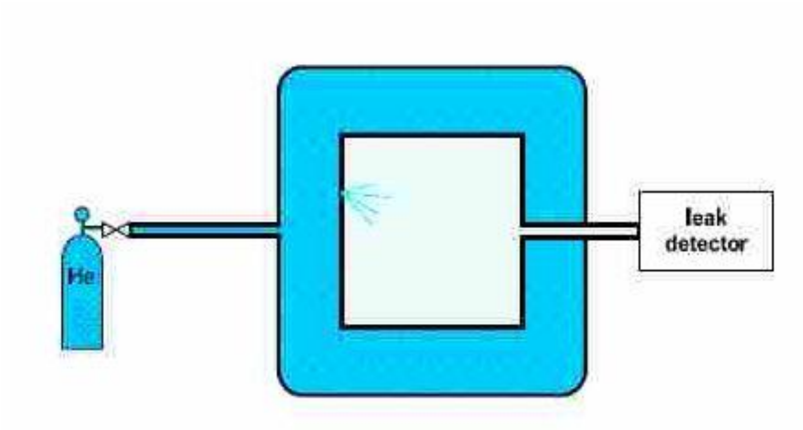
Quality Control

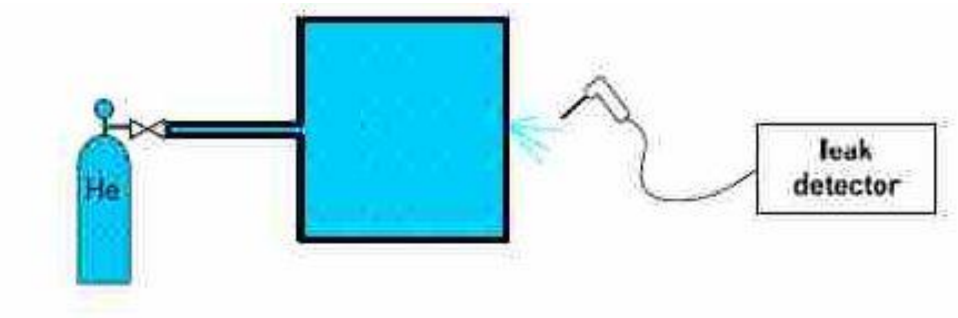
- Assessment (Tests)
  - Leak test
  - Performance tests
    - Base pressure
    - Outgassing rate
    - Cleanliness

Leak tests

- Specify a realistic leak rate
- Specify testing method



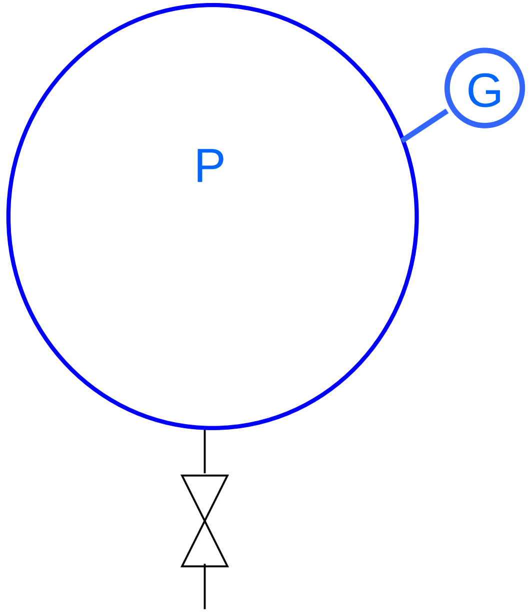


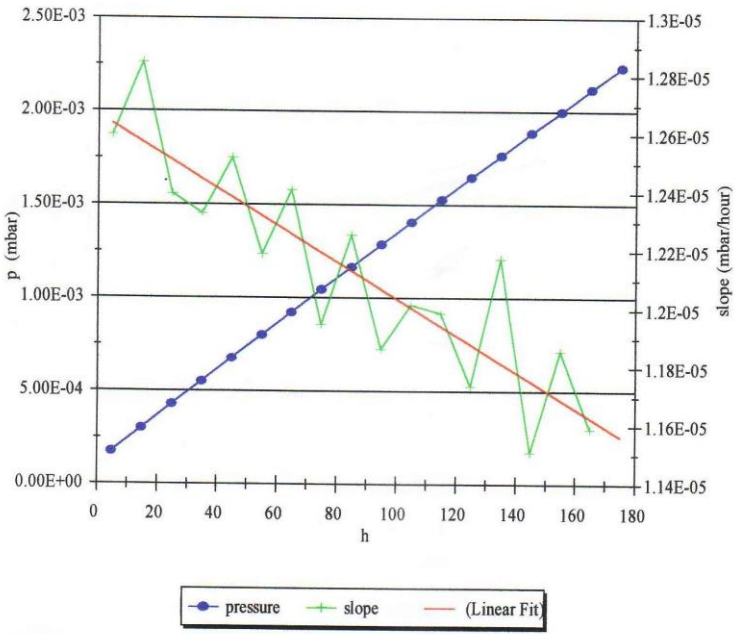


Outgassing test

- Rate of Rise (gas accumulation)

In a sealed chamber, $$Q = \frac{dP}{dt}_{t=0} \cdot \frac{V}{A}$$





Nemanic & Setina

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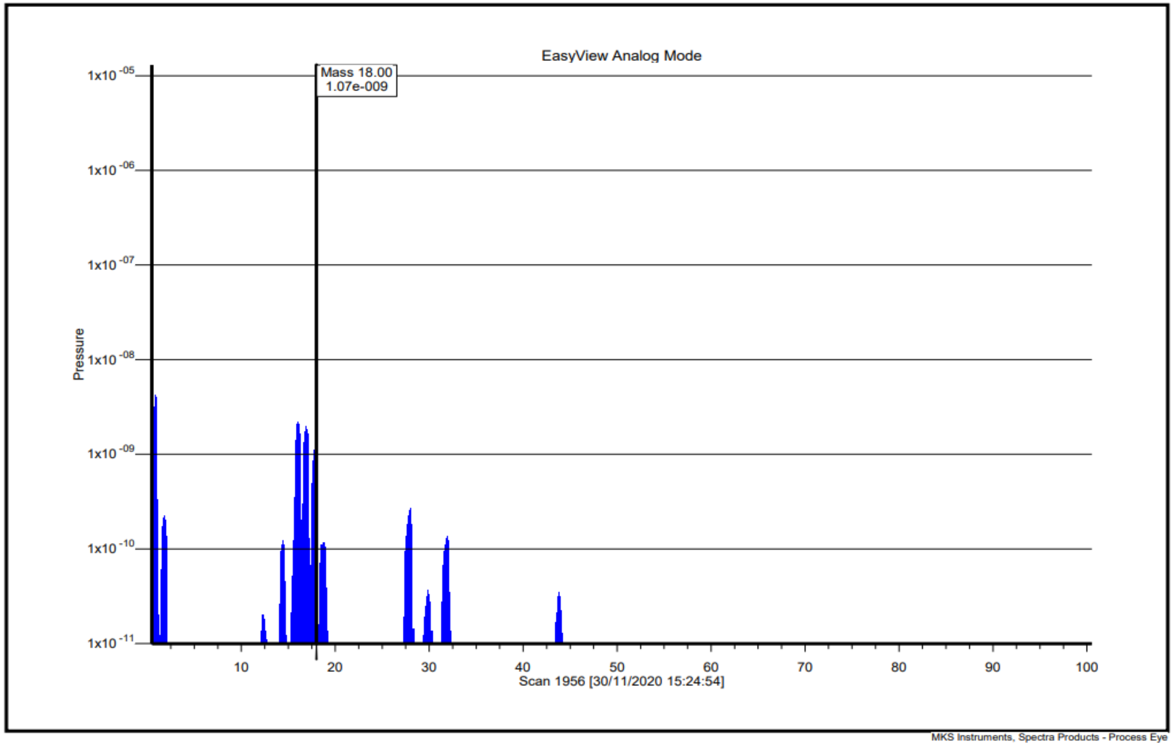
Acceptance Criteria

Pressure Region	General Contaminants (%)	Perfluoropolyphenylethers Sum of (peak at 69 and 77 amu) (%)	Chorinated species (Sum of peaks at 35 and 37 amu) (%)	Comment
UHV	0.1	0.01	0.01	Assuming system baked. **Calculation to be done at 10^{-9} mbar or below**



- RGA scans used to determine relative contamination within vacuum
- Sum of partial pressures of contamination should not be above 0.1% of total pressure

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Broad Range of Methods Available

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

Installation Cleanliness

•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 X 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.

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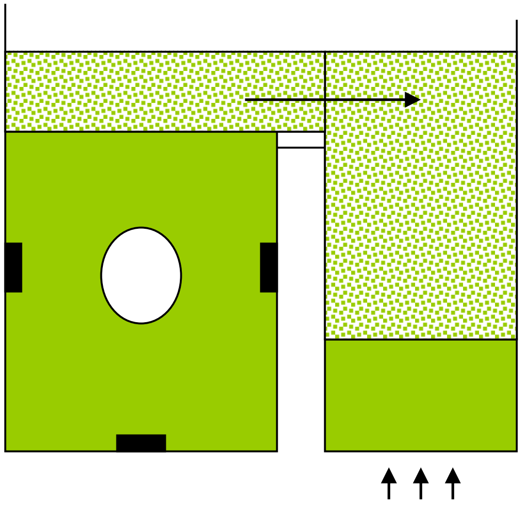
Typical Cleaning Agents

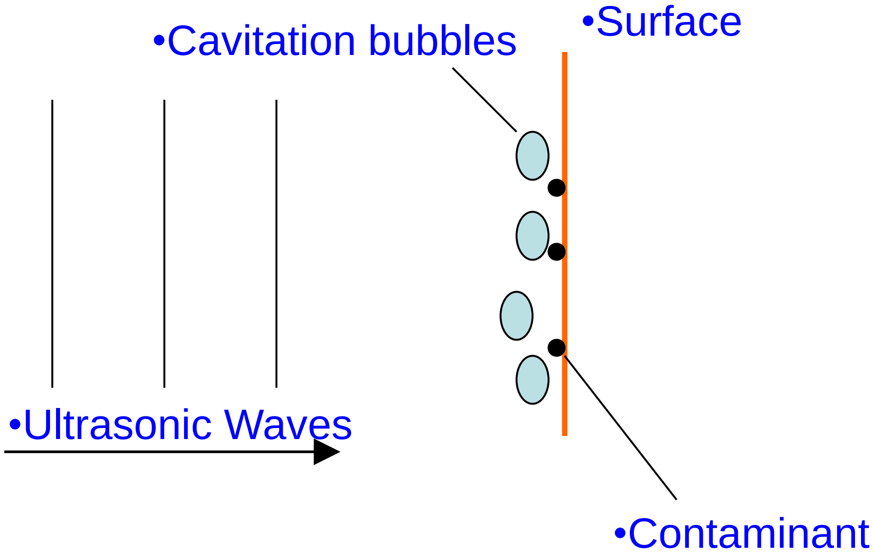
Agent	Examples	Advantages	Disadvantages	Disposal
Water		Cheap, readily available	Need de-min for cleanliness. Not a strong solvent	To foul drain
Alcohols	Ethanol, methanol, iso-propanol	Relatively cheap and readily available. Quite good solvents	Need control – affect workers; some poisonous; some flammable; stringent safety precautions.	Evaporate or controlled disposal.
Organic Solvents	Acetone, ether, benzene	Good solvents, evaporate easily with low residue.	Either highly flammable or carcinogenic	Usually evaporate
CFC's	Freon™ (CFC-113)	Excellent solvents; evaporate easily with low residue	Banned	Strictly controlled, must not be allowed to evaporate
Chlorinated hydrocarbons	Trichloroethylene (Trike™)	Excellent solvents. Non-toxic. Low boiling point. Low residue	Toxic, requires stringent safety precautions.	Strictly controlled
Detergents		Aqueous solutions, non toxic. Cheap and readily available. Moderate solvents.	Require careful washing and drying of components. Can leave residues.	To foul drain and dilution
Alkaline degreasers	Almeco™ sodium hydroxide	Aqueous solutions, non- toxic. Moderate solvents	Can leave residues and may throw particulate precipitates	Requires neutralisation, then dilution to foul drain.

Aqueous & Solvent Cleaning

- **Special Cleaning Techniques**

  Ultrasonic cleaning - widely used





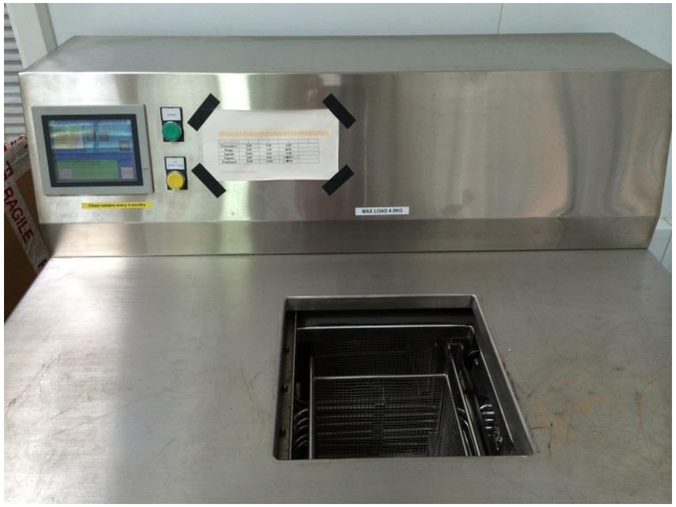
Cleaning Process

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

•Power wash booth for large items

•Auto washers for small items

Solvent wash, HFE72DE



•**<u>2 x Solvent cleaning plants:</u>**

•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.

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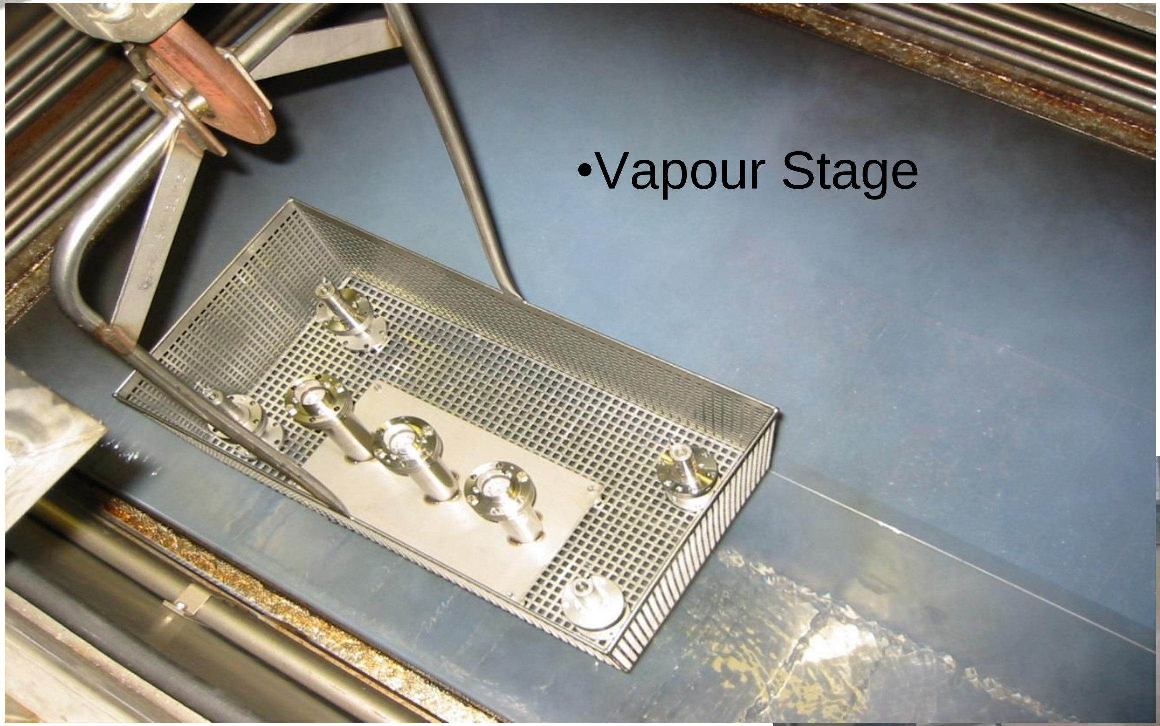
Solvent Cleaning



- Vapour Stage

- Alkaline degreaser

Drying

- Hot drying cabinet.

Cleaning Process Scientifically Developed

•**Publications:**

1. K.J. Middleman, J.D. Herbert, R.J. Reid, Vacuum 81 (2007) p793-798
2. J.D. Herbert and R.J. Reid, Vacuum, Vol. 47, 6-8, p693 (1996)
3. J.D. Herbert, R.J. Reid, A.E. Groome, J. Vac. Sci. Technol. A12(4), p1767, (1994)

- 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

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

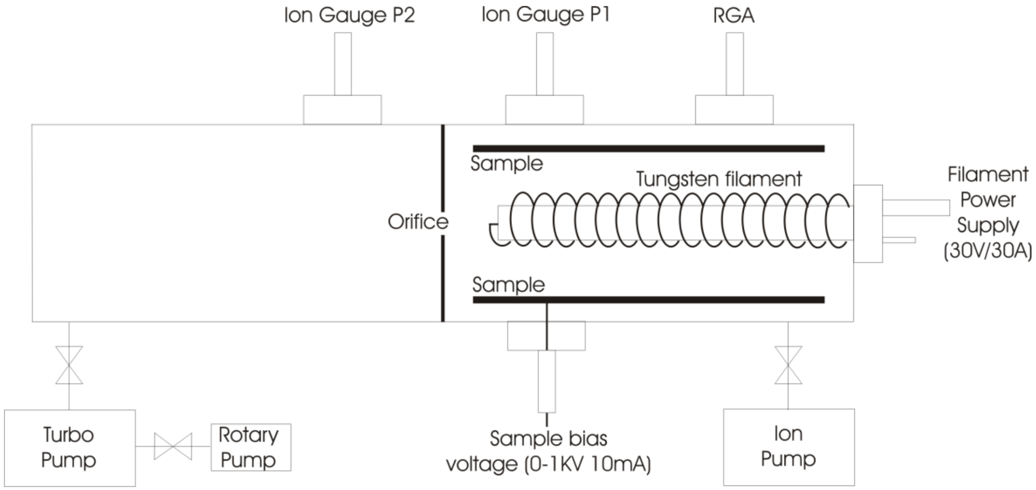
✓ **Trichloroethylene** selected (comparable to Trichloroethane)

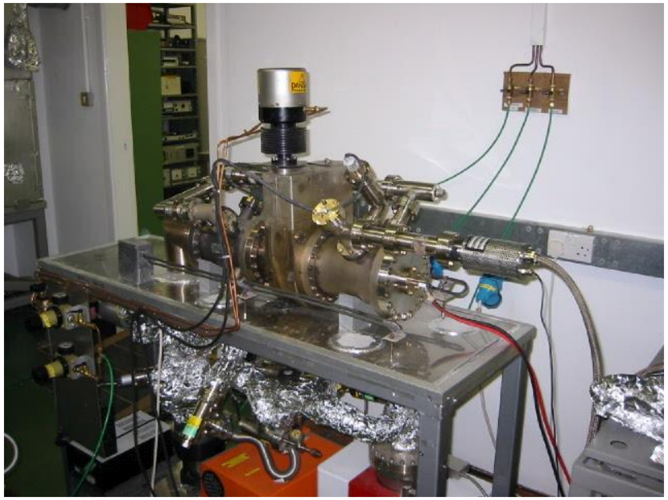
✗ **Aqueous** cleaners NOT SUFFICIENT alone but OK in combination with solvent.

✓ **Glow Discharge** – Dropped

Replacement of Trichloroethylene

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





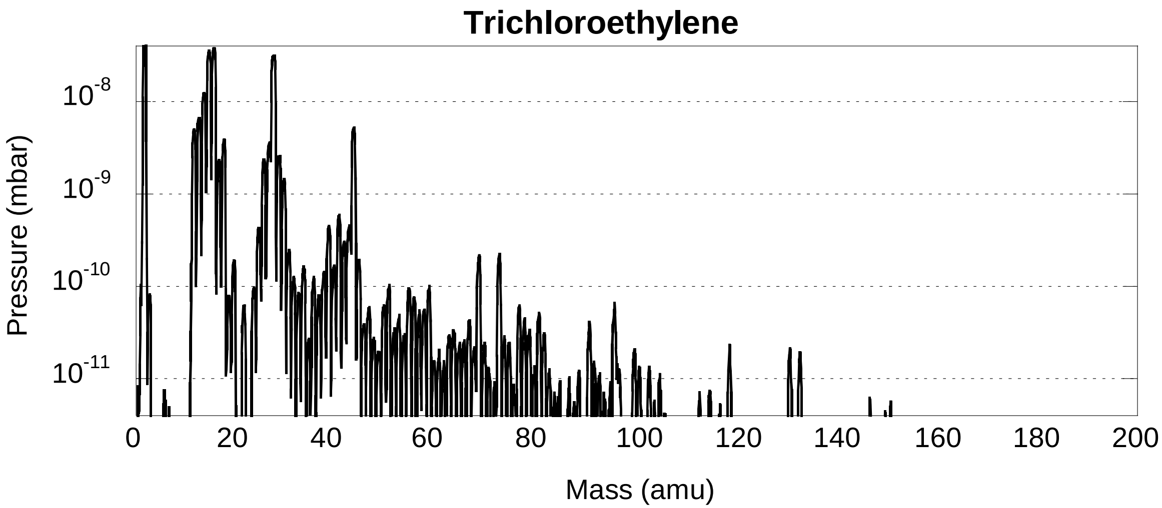
$$Q = \frac{P1 - P2}{A} \cdot C$$

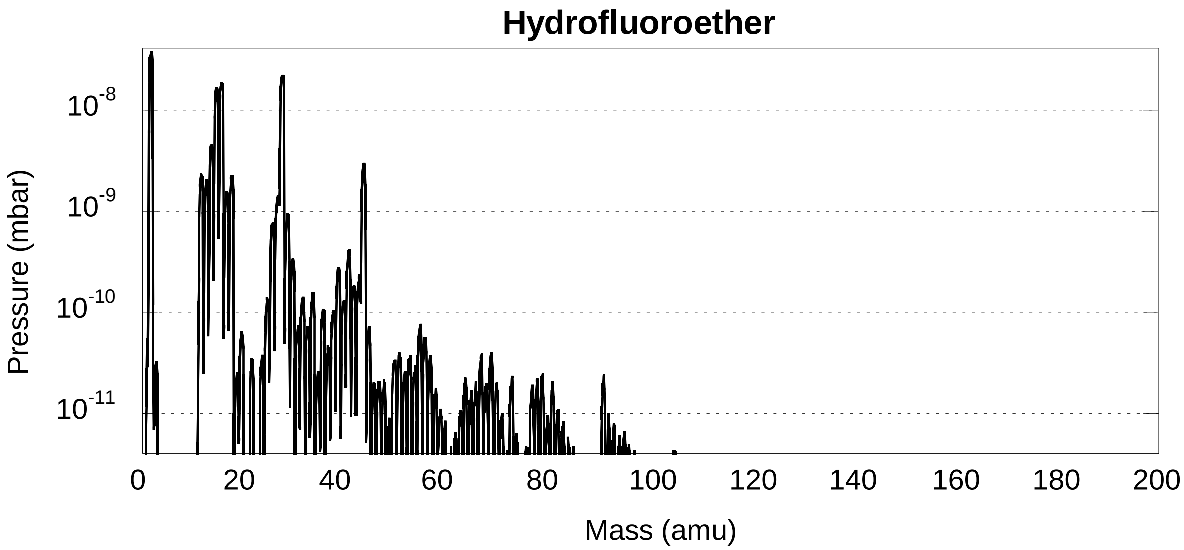
- Comparative Tests - existing procedure proven for 20 years

Cleaning Project Results

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

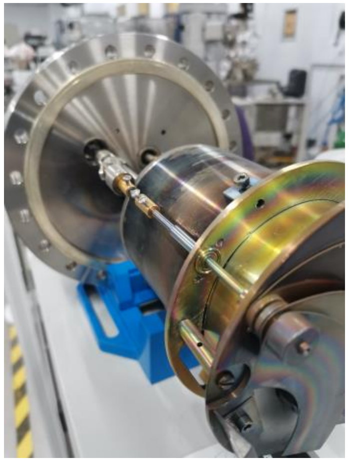
ESD RGA data for HFE and Trike

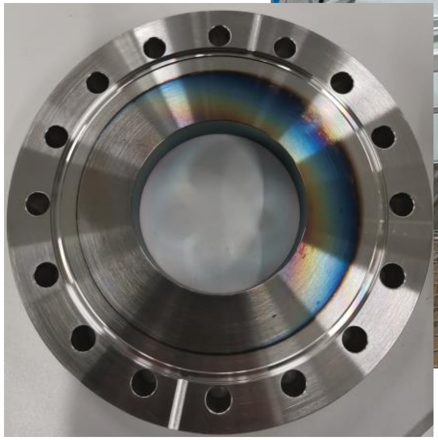




Slurry Blasting

- Slurry Blasting or Wet Blasting is the process of mixing an abrasive with pressurised water to clean a surface
- Forcibly propelling abrasive material at a surface to remove heavy contamination such as coatings or severe discolouration.
- Mainly used in heavily contaminated ion pump elements or coated components
- Can change the surface appearance due it its aggressive nature





Vacuum Firing

- The manufacturing process for steel means large quantities of H_2 are left in the bulk of the material.
- This H_2 is the limiting factor in achieving the best possible outgassing rates for UHV/XHV systems
- Vacuum firing (or anne… …terial is heated up to high temperature (~950° C)…
- This high temperature … H_2 from the bulk to the surface layers and allow…
- This process can improv… …s steel by up to 2-3 orders of magnitude.
- The high temperature t… …permeability of a material, something which is ver…

- <u>Main Uses:</u>
  - 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

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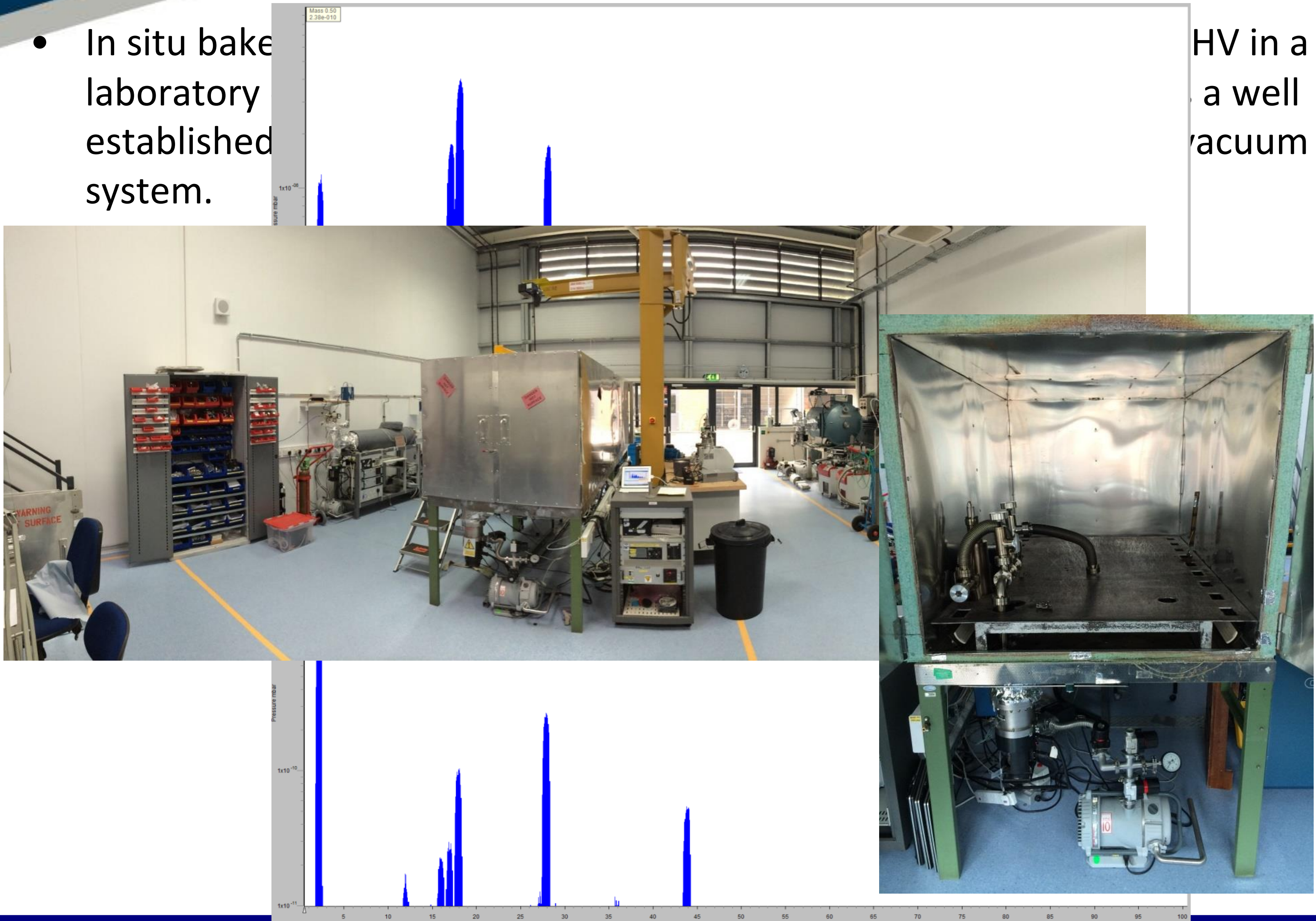
Bakeout In-Situ

- In situ bake … HV in a laboratory … a well established … vacuum system.



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Bakeout Ex-Situ

<u>What is an acceptable RGA Scan?</u>

- 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 **<u>MUST</u>** 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 **<u>NOT</u>** 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

Line Number	Pressure Region	General Contaminants (%)	Perfluoropolyphenylethers Sum of (peak at 69 and 77 amu) (%)	Chorinated species (Sum of peaks at 35 and 37 amu) (%)	Comment
1	UHV	0.1	0.01	0.01	Assuming system baked. Calculation to be done at 10^{-9} mbar or below
2	HV - UHV	0.75	0.075	0.075	Assuming system unbaked. Calculation to be done at 10^{-7} mbar or below

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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!

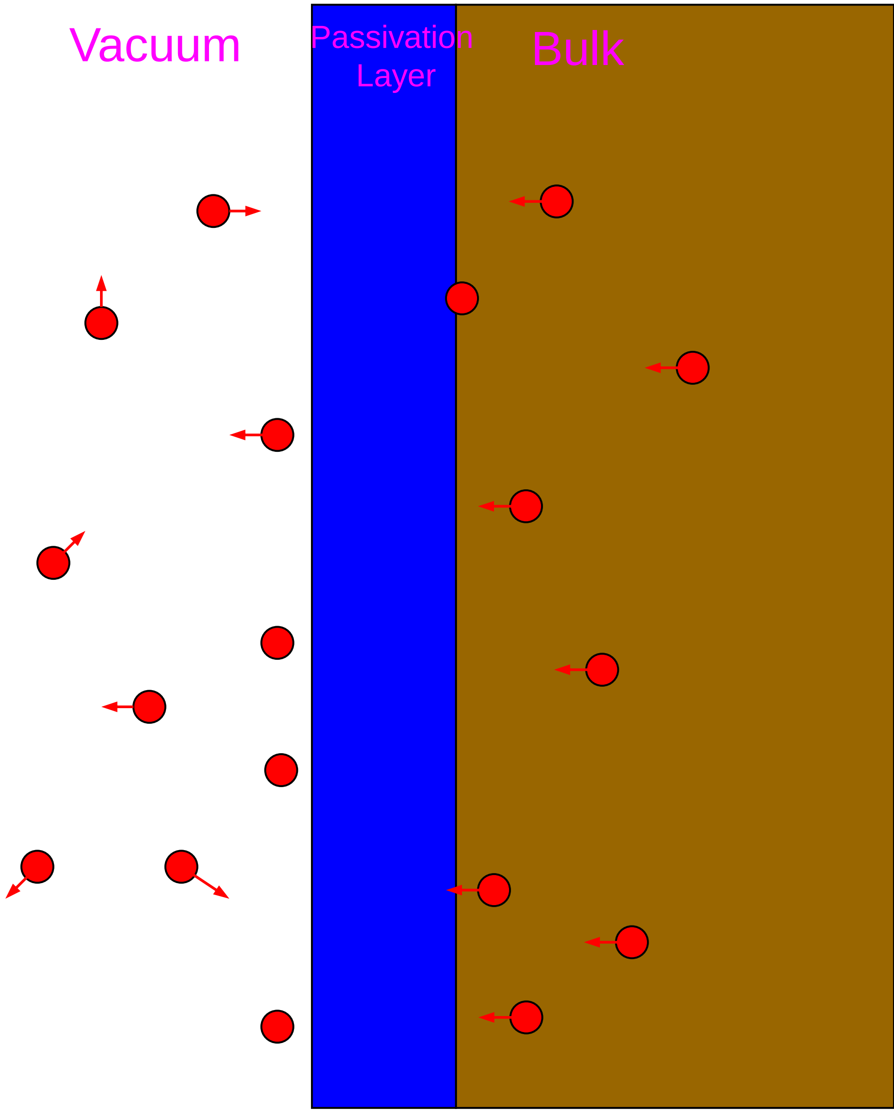
Dry Ice Cleaning

- 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.

- <u>Main Uses:</u>
  - Food industry
  - Semiconductor
  - Aerospace
  - RF structures for accelerators

What is Passivation?

- The use of a barriers to inhibit outgassing
  - Coatings
    - NEG
    - TiN
  - Surface modifications
    - Electropolishing
    - Acid Etching (Buffer Chemical Polishing – BCP)



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Air Baking

- The simple process of heating a vacuum chamber to a particular temperature in air.
- Typically baked to around 400^{o}C
  - Helps remove H_2 from the bulk but at a lower temperature the rate of diffusion is much lower, therefore not as effective at depleting H_2 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.

Air Baking

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.

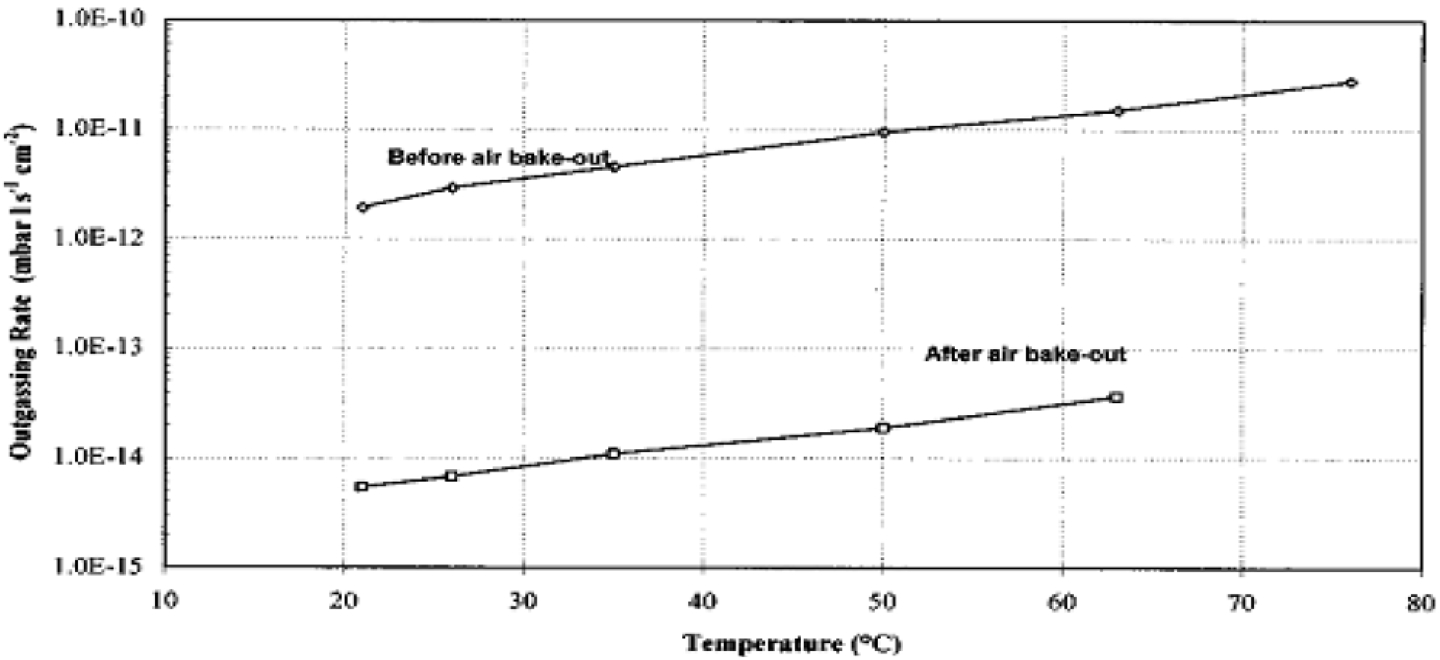


Figure 4. Hydrogen outgassing rate with and without air baking[7]

[7]M. Bernardini, S. Braccini, R. De Salvo, G. Genuini, Ž. Zhang, "Air bake-out to reduce hydrogen outgassing from stainless steel, "journal of vacuum and science technology, vol. 1, no. 16, pp. 188-193, 1998.

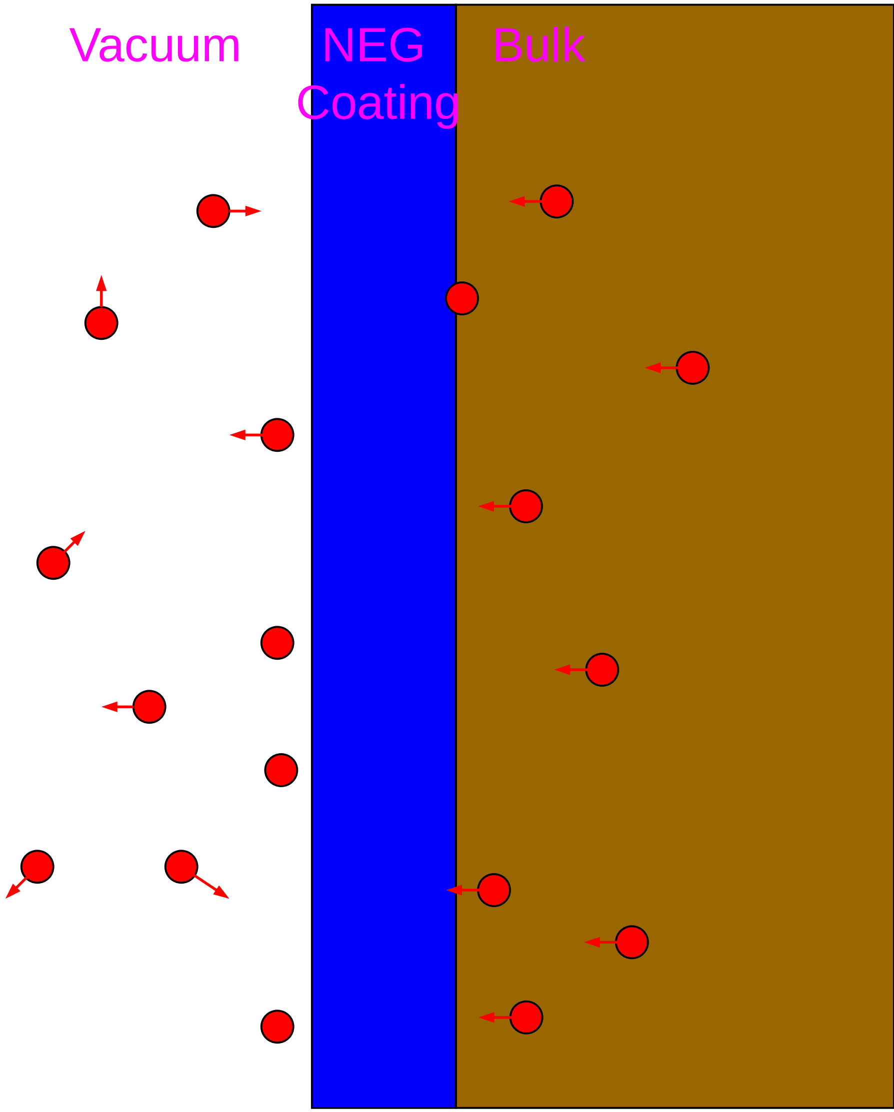
What NEG coating does

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

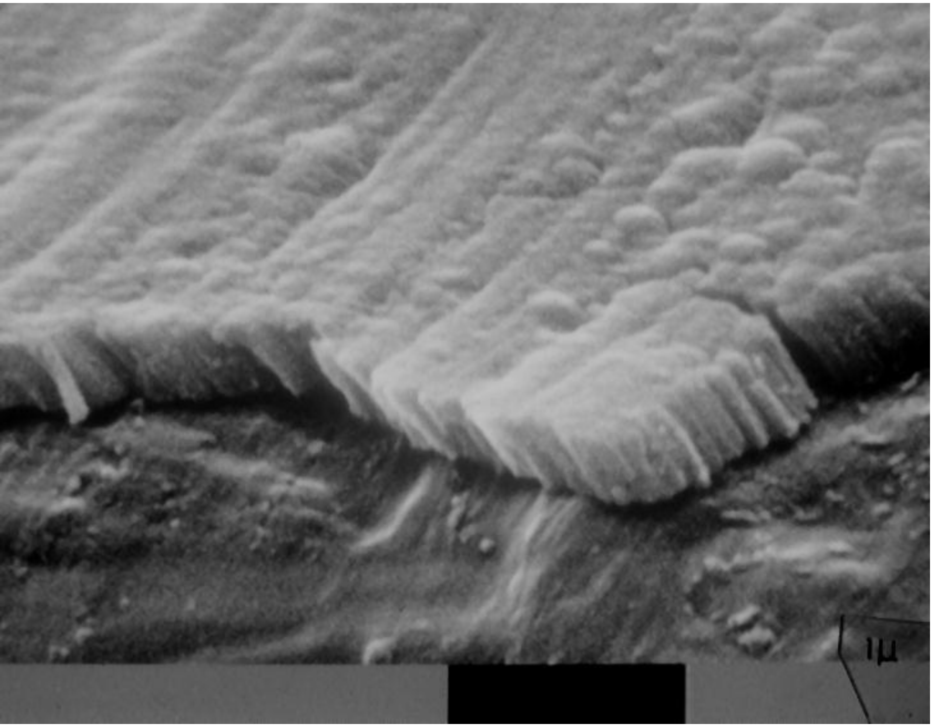
$$S = \alpha \cdot A \cdot v/4;$$

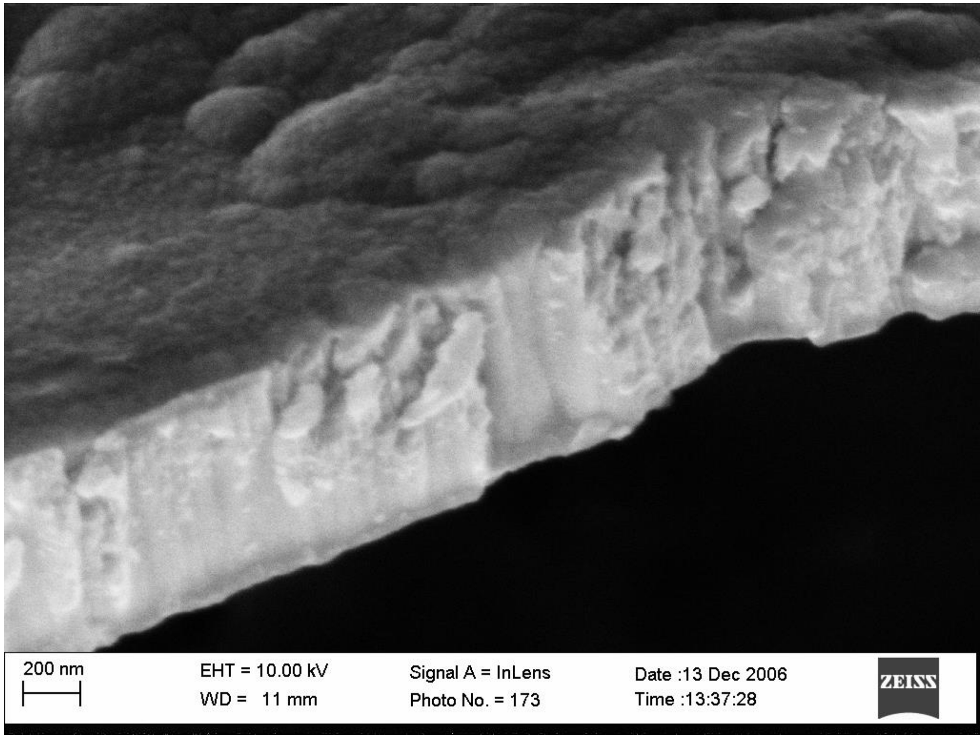
where α – sticking probability,
A – surface area,
v – mean molecular velocity



SEM images of films (film morphology)

- columnar   dense

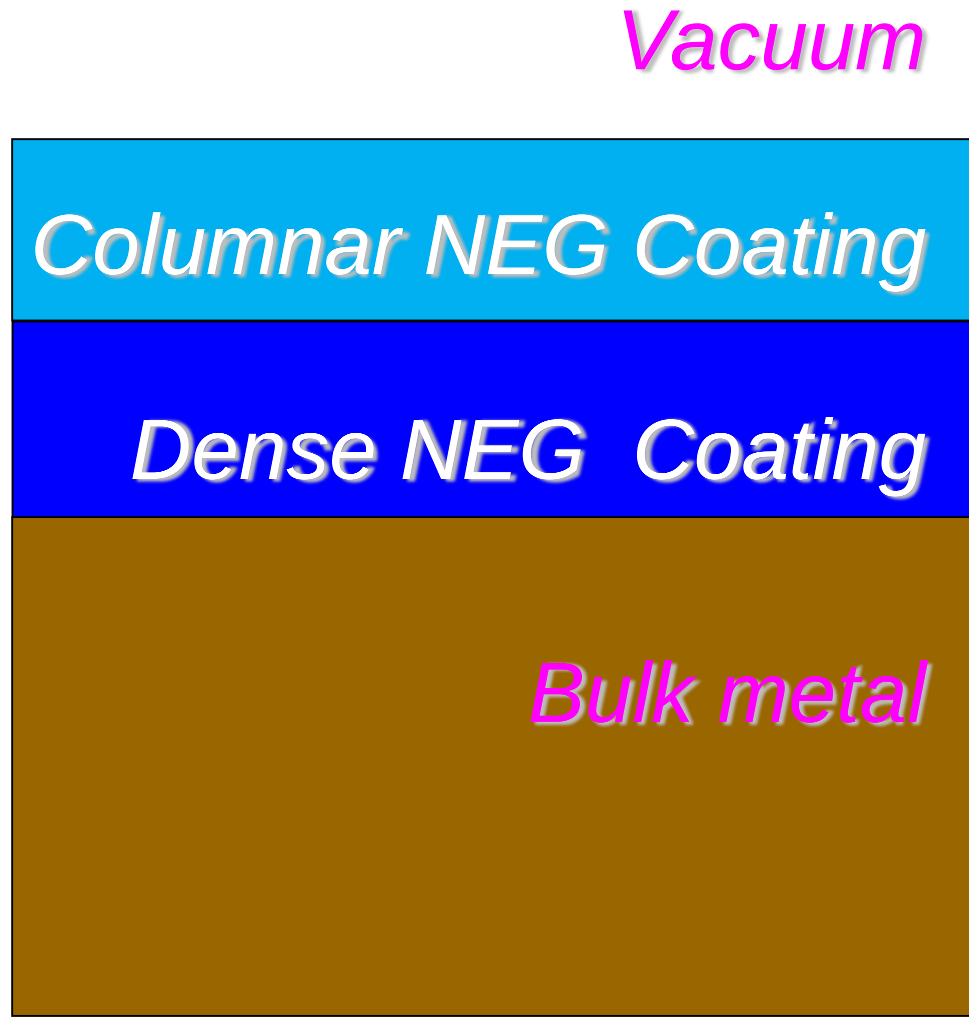




•O.B. Malyshev, R. Valizadeh, J.S. Colligon *et al*. J. Vac. Sci. Technol. A 27 (2009), p. 521.

Dual layer

- 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



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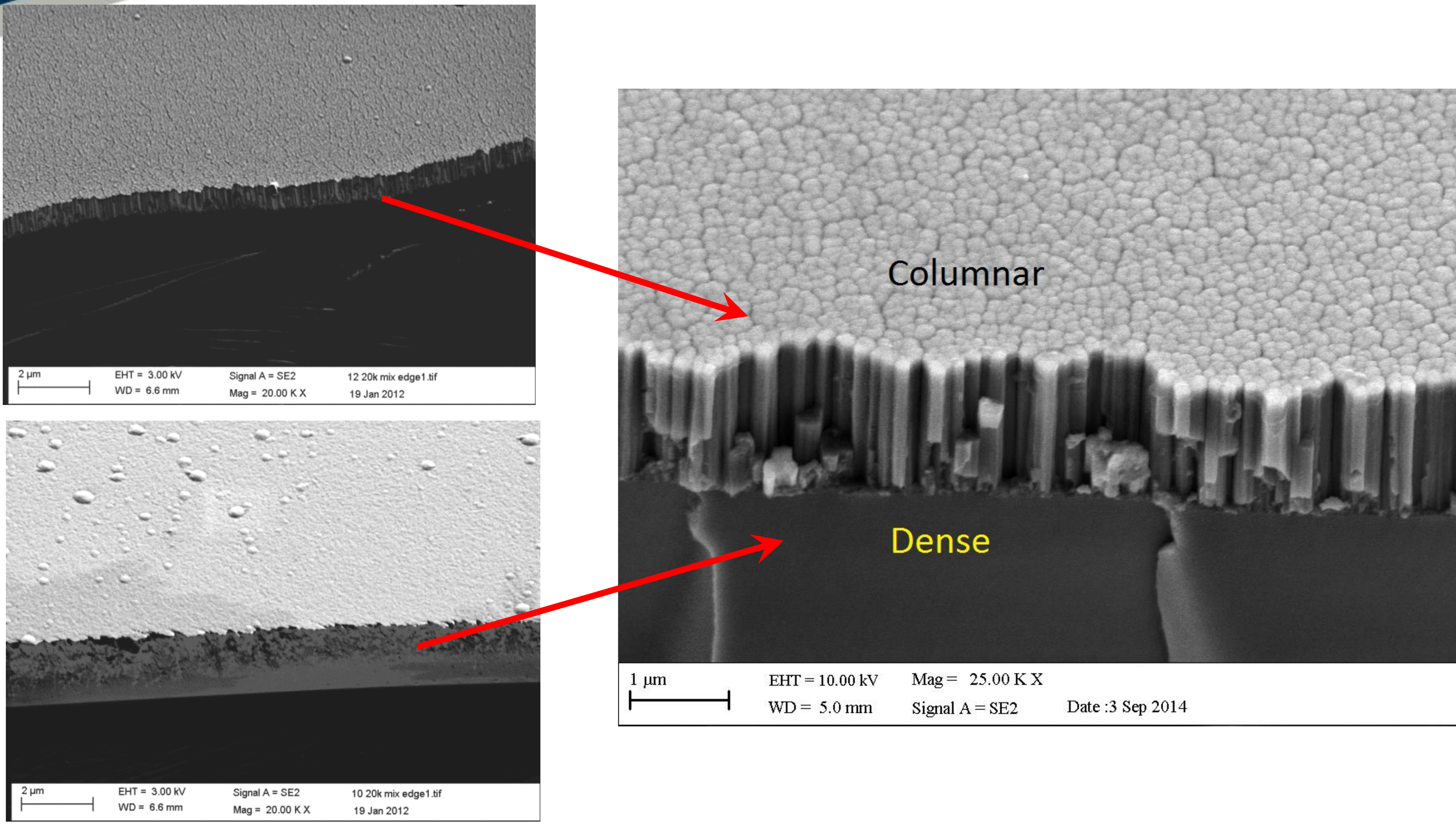
Dual layer



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 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/cm^2 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**

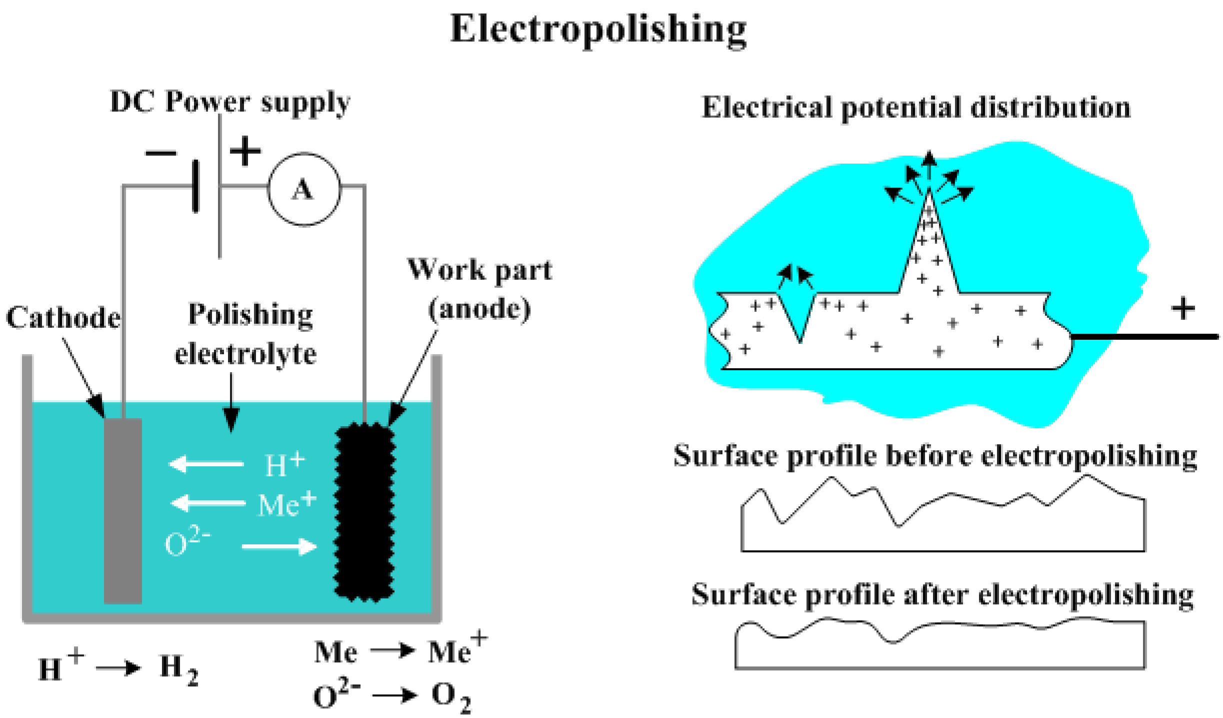
Buffered Chemical Polish

- 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

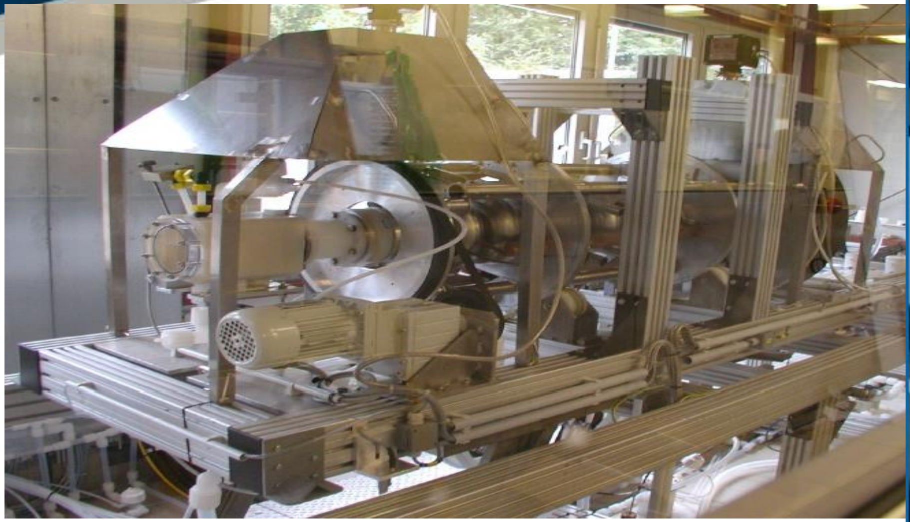
- Acid mixture
  - Hydrofluoric acid; HF (49%)
  - Nitric Acid; HNO_3 (65%)
  - Phosphoric Acid; H_3PO_4 (85%)
  - In a 1:1:1 mixture
- Risk of hydrogen contamination
  - Correct mixture should be used
  - Temperature of acid should be kept below <18 ºC, to control the exothermic reaction
  - Vacuum processing required
- Cavity is the high pressure rinsed (HPR) with ultrapure water

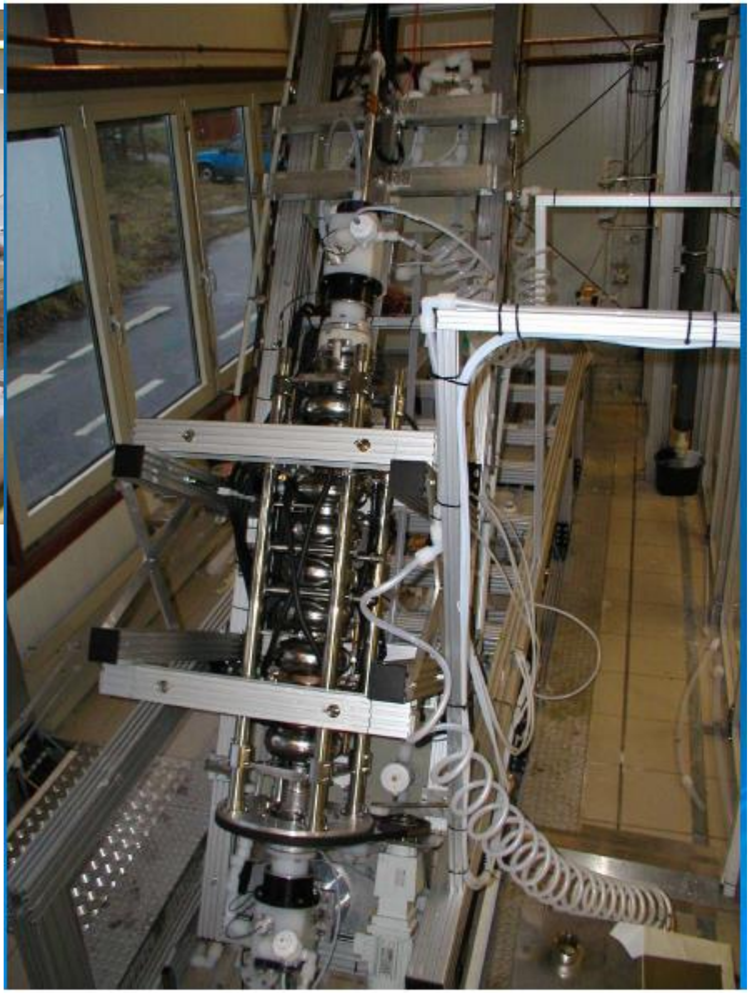
Electropolish (EP)



- 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

Electropolish (EP)





- 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 Accelerators

- 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^{-11} mbar**, **P_{O2}<10^{-14} 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.

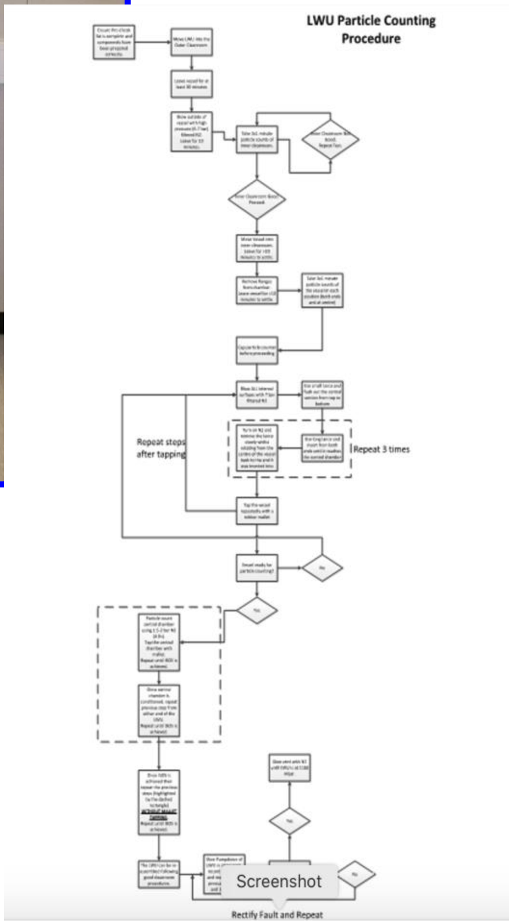
Dust particles in a vacuum chamber

- The dust *micro-particle* in the beam vacuum chamber might be ionised by photons or photoelectrons and then be *trapped be the beam* electric field.
- This may course *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 enviroment when vacuum chamber is open
  - Clean gas for venting (for example, boil-off nitrogen)

Particle Control



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**

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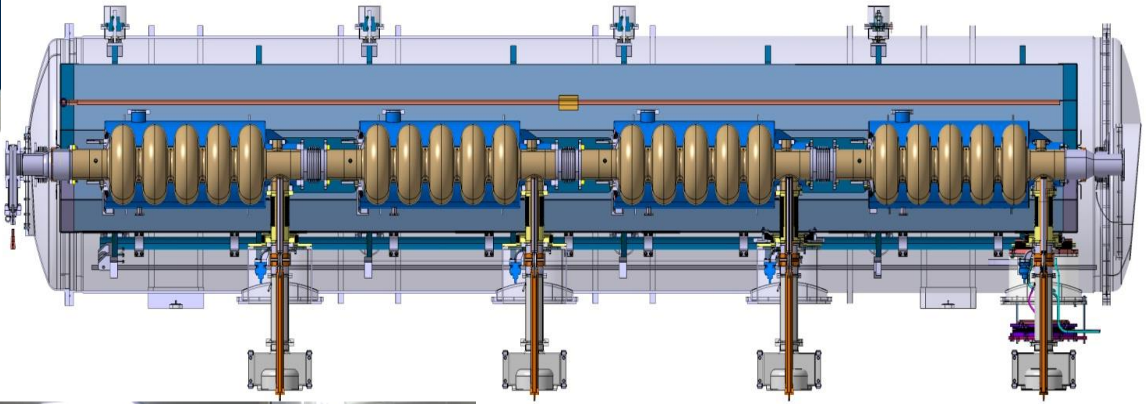
Particle Cleaning – Why it's important



- ESS LWU's have to be particle free
- They are directly connected to the SRF cavity.
- Any particles cause field emission within the cavity.
- Field emission process generates electrons and causes localised heating
- Such issues cause a degradation in cavity performance & cryogenic instability
- Can cause SRF cavity to Quench where the cavity loses its superconducting state

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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