

Vacuum & Surface Science

Lecture 9

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Science & Technology Facilities Council

ISIS



ISIS from above



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ISIS

ISIS is a high power accelerator that fires high energy protons into two targets to release neutrons for experiments.

The ISIS synchrotron accelerates protons to 84% of the speed of light then fires them into two tungsten targets.



Target Station 1

Neutrons are released from both targets via spallation. Using neutrons, scientists can study the atomic structure of materials and can even measure the forces between atoms.



Target Station 2

The second target station is optimised for low energy neutrons providing greater capacity at ISIS and opening up new areas of research.



Part 1 – Vacuum for Accelerators

Part 2 – Surface Science



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ISIS

Part 1 - Vacuum for Accelerators

- In the UK – ISIS, Diamond, CLARA
- Also – CERN, ESRF, SNS, SOLEIL, ELLECTRA, DESY, BESSY etc



Part 1 - Vacuum for Accelerators

The Vacuum Spectrum

Extreme High Vacuum (XHV)	Ultra High Vacuum (UHV)	High Vacuum	Rough Vacuum
below 10^{-10}	10^{-10} to 10^{-8}	10^{-8} to 10^{-3}	10^{-3} to 10^{+3}
Pressure (mbar)			
← Cost (£)			



Part 1 - Vacuum for Accelerators

In general the vacuum level required for an accelerator is determined by its operation.

- Single pass – 1 to 1000 μ s (10^{-7} to 10^{-9} mbar)

OR

- Storage ring – 10 hours or more (10^{-9} mbar or less)



Part 1 - Vacuum for Accelerators

So what happens if you fail to achieve the required vacuum level ?

- Beam scattering occurs – increased radiation levels in work area
- Beam shape changes
- Beam lifetime reduced
- **Beam Loss Occurs**



Part 1 - Vacuum for Accelerators

How to ensure you achieve the required vacuum level and avoid beam loss.

- Correct selection of materials for the external vacuum vessel and also components used internally (including surface finish).
- Ensure that vacuum vessels and components are kept clean to reduce outgassing and desorption effects.
- Adequate pumping, leak testing and conditioning.



Part 1 - Vacuum for Accelerators

Any accelerator vacuum system needs the following ingredients;

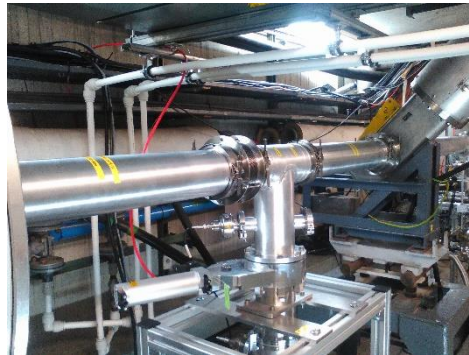
- Vacuum Vessel
- Internal vacuum components (eg. beam monitors)
- Cleaning procedure for vessel and components
- Vacuum Seals & Clamps
- Gate valves & bellows – RF screened if necessary
- Pumps and Gauges
- Residual Gas Analyser (RGA's)
- Modelling software
- Control system



Part 1 - Vacuum for Accelerators

The Vacuum Vessel – Common Materials

- Stainless steel (grades 304L or 316L and N)
- Most grades of Aluminium
- Titanium
- Copper
- Ceramic



Part 1 - Vacuum for Accelerators

1 of 10 Ceramic vacuum vessel used on ISIS, 4m long, 19 segments (Dipole Magnet)



Challenge is joining the metal flange to the ceramic – use a filler material



Part 1 - Vacuum for Accelerators

Materials for internal vacuum components

- Aluminium
- Oxygen Free Copper – excellent heat dissipation
- Stainless Steel
- Glidcop (Copper and Aluminium oxide alloy)
- Gold
- Silica

**Avoid materials containing brass or cadmium
and PVC insulated wire**



Part 1 - Vacuum for Accelerators

Cleaning of vacuum vessel and components

- Cleaning procedures will help to reduce thermal outgassing and also electron, photon and ion desorption, but difficult to stop these completely.
- Typically cleaning involves washing the vessel or component in solvent, then drying following by baking to remove solvent residue.



Part 1 - Vacuum for Accelerators

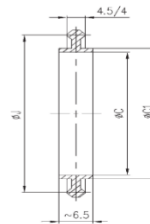
- Most accelerators will have an on-site cleaning facility – solvent baths and ovens



Part 1 - Vacuum for Accelerators

Vacuum Seals – Usually soft metals that are easily squashed (avoid elastomers eg. Viton and Kalrez of Chemraz).

- Copper
- Aluminium
- Gold
- Indium



Part 1 - Vacuum for Accelerators

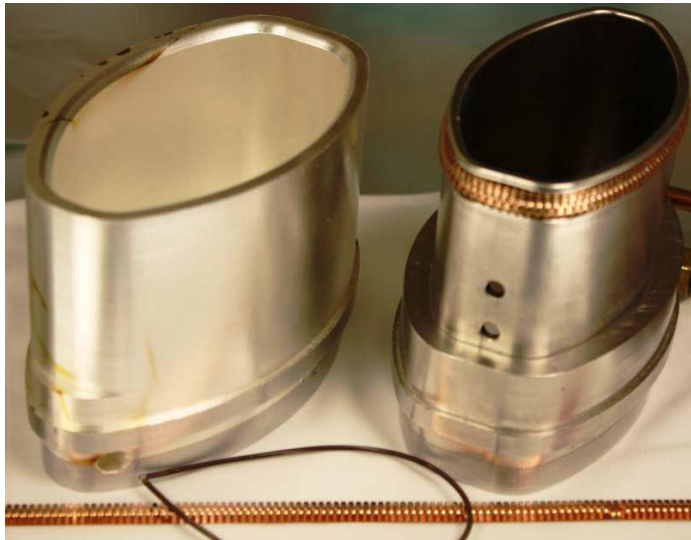
Vacuum Clamps – depends on flange dimensions

- Conflat
- ISO
- Quick Release
- Remote operation for high radiation areas.



Part 1 - Vacuum for Accelerators

Vacuum Valves & Bellows – RF Screened (springy fingers)



RF screened bellows (sliding arrangement) – CESR , US

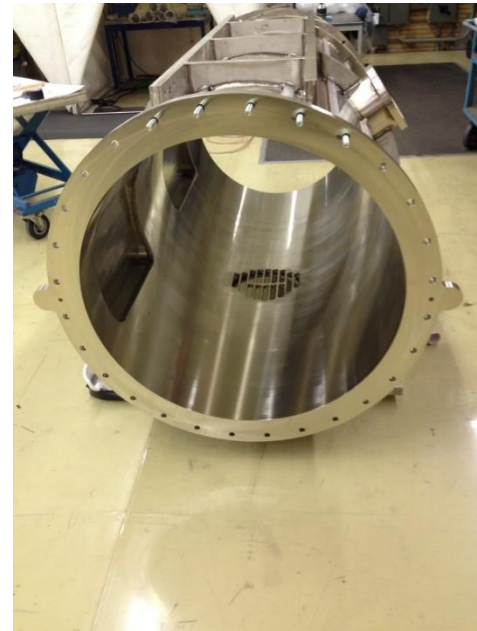
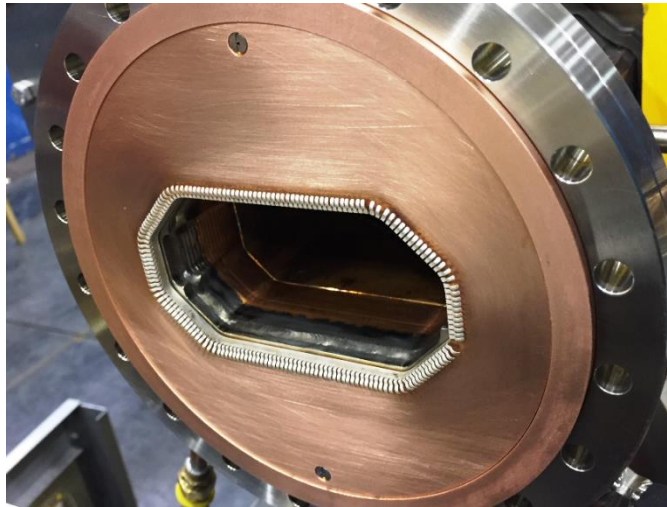


RF screened bellows – KEK, Japan

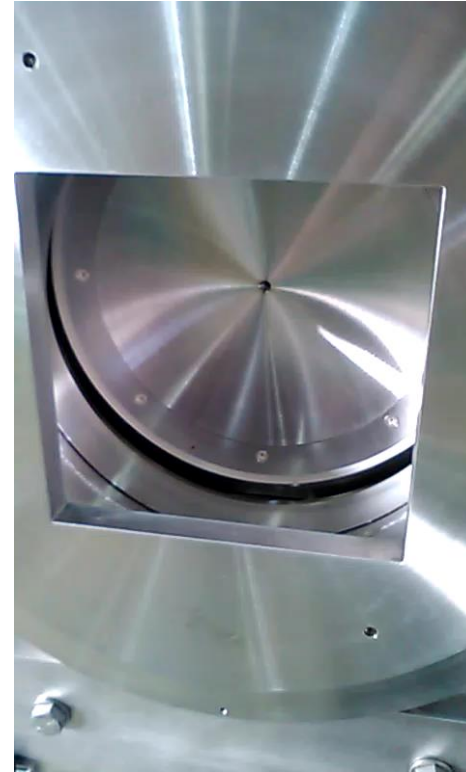
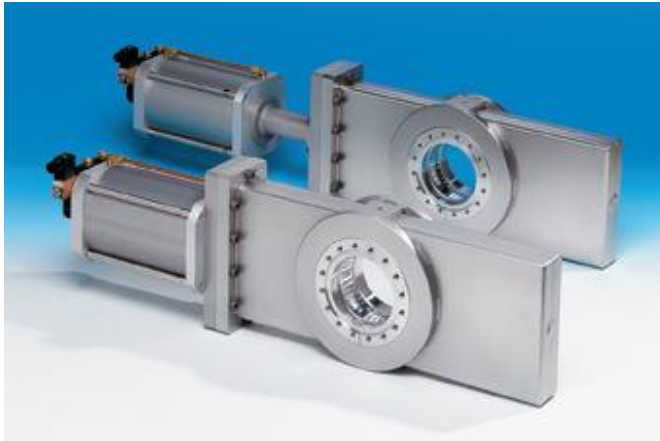


Part 1 - Vacuum for Accelerators

Minimising beam disturbance – more examples of screening






Part 1 - Vacuum for Accelerators



Part 1 - Vacuum for Accelerators

Types of Vacuum Pump

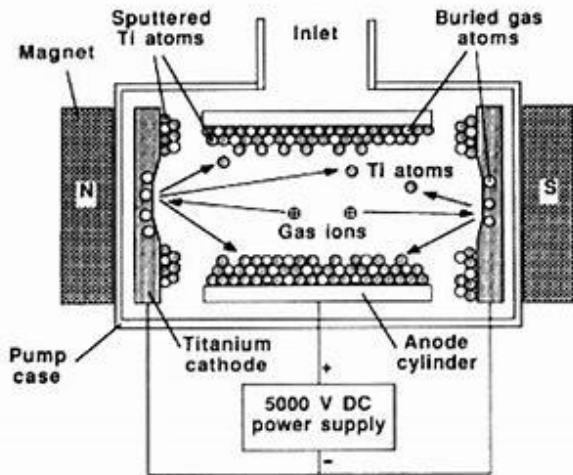
Rotary	Roots	Ion
Diffusion	Scroll	Getter
	Turbo	
	Screw	
	Claw	
	Cryo	
		



Part 1 - Vacuum for Accelerators

Vacuum Pumps – Ion Pumps (Diode or Triode) (From 0.2 l/s to 500 l/s)

Operation based on the penning discharge (HV applied in presence of a strong magnetic field). Cathode bombarded resulting in sputtering and trapping in or on the surface.



Part 1 - Vacuum for Accelerators

Ion Pumps Lifetime – 50 to 80,000hrs based on pump operation at 1×10^{-6} mbar

Lifetime depends on

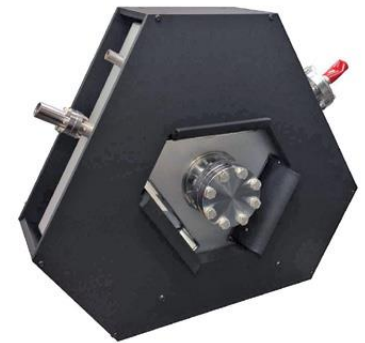
- Starting pressure (lower pressure is better)
- Gases being pumped
- Operating vacuum (lower pressure = longer life)
- On/Off cycles



Conventional Arrangement - DIAMOND



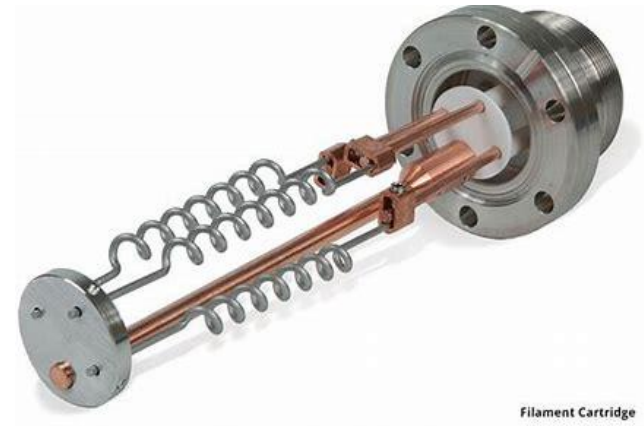
In Line Pumping – XFEL, DESY



Part 1 - Vacuum for Accelerators

Getter pumps - Titanium Sublimation (TSP) and Non Evaporable Getter (NEG)

TSP – Evaporates titanium over nearby surfaces.
“Trapping effect” can be improved by cooling. Other common evaporable getter materials are barium, thorium and manganese.



Part 1 - Vacuum for Accelerators

“NEG coatings are thin films, of getter material, which once activated has the ability to pump a vacuum vessel. It can also minimise outgassing and secondary electron yield from the internal surface of the vessel”.

Typically activation temperature is between 150 and 400°C.

Extensive research carried out at CERN by Cristoforo Benvenuti

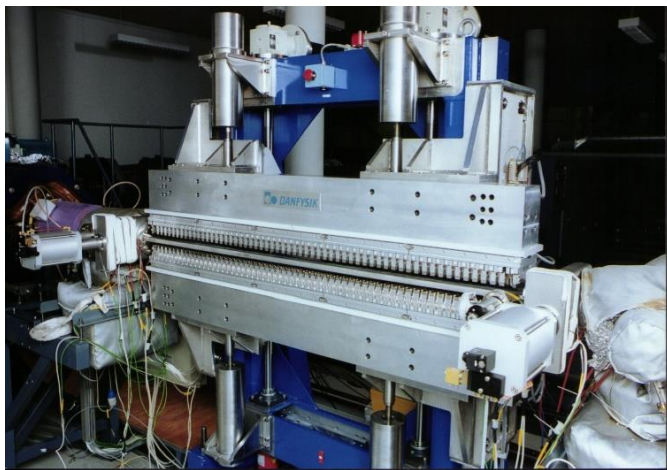
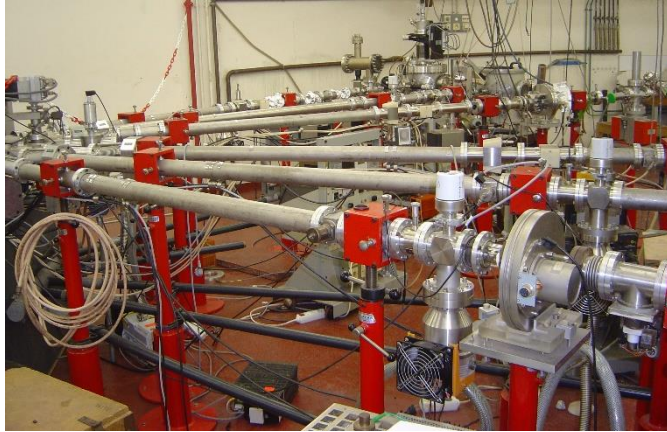


Part 1 - Vacuum for Accelerators

- Helps to improve the base pressure in the accelerator or vacuum vessel, which improves beam-lifetime.
- Helps to reduce outgassing and secondary electron yield from the vessel walls, which also improves beam-lifetime.
- Modern insertion devices have limited conductance for pumping by conventional means.



Part 1 - Vacuum for Accelerators



Part 1 - Vacuum for Accelerators

What are NEG coatings made from?

- Preference for elements that have high oxygen diffusion and solubility limits. Hence mainly elements in Group 4 and 5 of the periodic table (i.e. Ti, Zr, Hf, V, Nb and Ta).
- Usually binary (e.g. TiV), or tertiary alloys of the above used (e.g. TiVZr or TiVHf).



Part 1 - Vacuum for Accelerators

What makes a good NEG coating

- Good adhesion
- Low activation temperature
- High solubility and diffusion limit for oxygen
- High mechanical resistance
- Large pumping speeds
- Low photoelectron and secondary electron yields
- Non toxic



Part 1 - Vacuum for Accelerators

How do they work ?

- X-ray Photoelectron Spectroscopy shows that during activation surface composition changes from being oxygen rich to more metallic in nature. This provides active sites for adsorption.
- H₂ is adsorbed *reversibly* and can easily be removed by re-heated.
- CO and CO₂ are adsorbed *irreversibly* forming stable compounds with the NEG alloy such as titanium oxide. When heated these compounds migrate into the bulk of the material leaving new sites for adsorption.



Part 1 - Vacuum for Accelerators

What are the other benefits of using NEG coatings?

- When used in conjunction with ion and turbo pumps can help to attain extreme high vacuum conditions (XHV), by removal of CO, H₂ and CO₂.
- Vibration free, suitable for very delicate experiments.
- Not influenced by magnetic or electric fields.



Part 1 - Vacuum for Accelerators

What about the disadvantages of NEG

- Expect to pay more for a NEG coated vessel than a uncoated one.
- Risk of contamination or poisoning during the activation process and during bakeout which will reduce the lifetime of the coating.
- Not suitable for pumping noble gases e.g. methane and argon
- Should never be exposed to high concentrations of hydrogen.



Part 1 - Vacuum for Accelerators

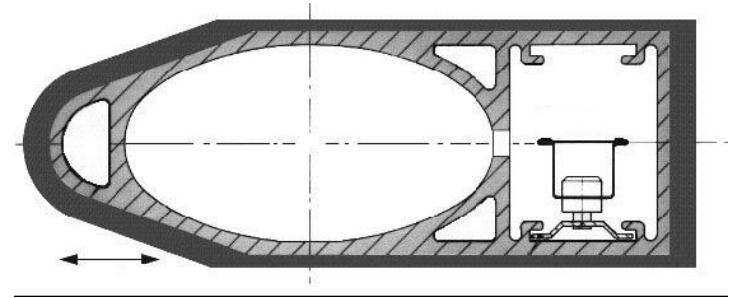
Options for adding NEG coatings

- Install cartridge with the getter material on it, e.g. SAES ST101 and ST707.

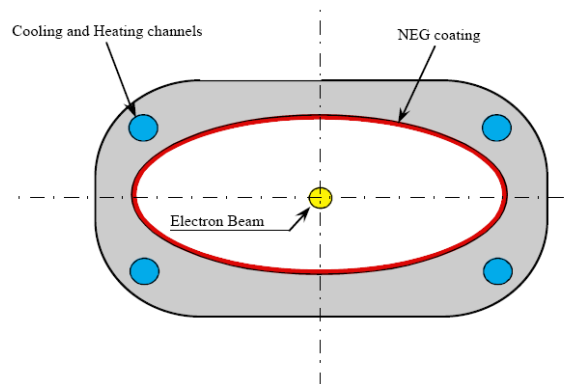


Part 1 - Vacuum for Accelerators

- Anti-chamber arrangement



- Vacuum coat the inside of the vessel typically using magnetron sputtering.



Part 1 - Vacuum for Accelerators



Part 1 - Vacuum for Accelerators

Vacuum gauges – no one gauge covers the entire vacuum spectrum.

Type	Range (mbar)
Spinning rotor gauge	10^{-1} to 10^{-7}
Dial gauge	1000 to 1
Diaphragm gauge	1000 to 10^{-5}
Pirani	1000 to 10^{-2}
Hot Cathode (Ion)	10^{-3} to 10^{-10}
Cold Cathode (Penning and Inverted Magnetron - IMG)	10^{-2} to 10^{-10}



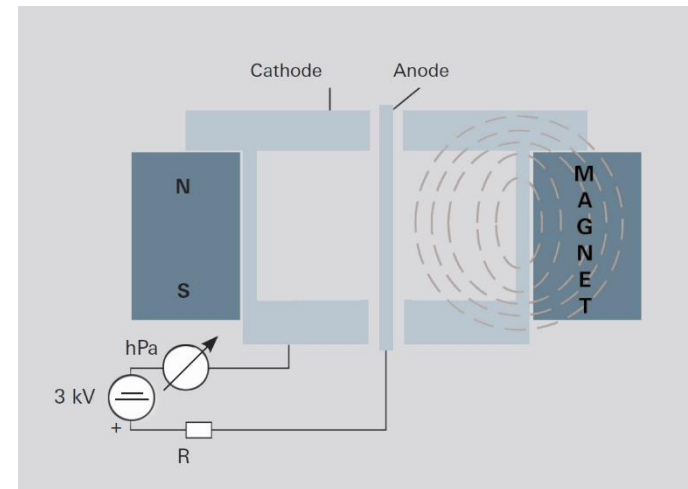
Part 1 - Vacuum for Accelerators

IMG – operation based on a penning discharge in the presence of a magnetic field.

Gas ionization used to
Measure pressure.

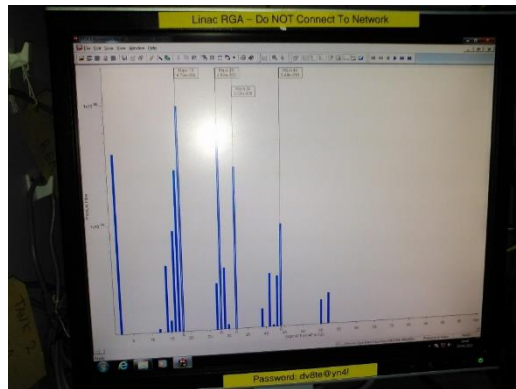
Gauge is gas sensitive due to
ionization probabilities.

Can be difficult to initiate
discharge at low pressures



Part 1 - Vacuum for Accelerators

RGA's – for diagnostics and leak testing



RGA ALARMS STATUS (Read Only)							
	GAS	MASS	READ LEVEL	TRIP LEVEL	TRIP TIME	CHECKING	STATUS
LOW ALARM	HELIUM	4 AMU	99.13 %	99.00 %	100 Sec	DE-ACTIVATED	NO ALARM
HIGH ALARM 1	HYDROGEN	2 AMU	0.00 %	1.00 %	100 Sec	DE-ACTIVATED	NO ALARM
HIGH ALARM 2	METHANE	16 AMU	0.01 %	1.00 %	100 Sec	DE-ACTIVATED	NO ALARM
HIGH ALARM 3	WATER	18 AMU	0.11 %	1.00 %	100 Sec	DE-ACTIVATED	NO ALARM
HIGH ALARM 4	NITROGEN	28 AMU	0.41 %	8.00 %	100 Sec	DE-ACTIVATED	NO ALARM
HIGH ALARM 5	OXYGEN	32 AMU	0.10 %	2.00 %	100 Sec	DE-ACTIVATED	NO ALARM
HIGH ALARM 6	USER ASSIGNED	1 AMU	0.00 %	1.00 %	0 Sec	DE-ACTIVATED	NO ALARM
HIGH ALARM 7	USER ASSIGNED	1 AMU	0.00 %	0.00 %	0 Sec	DE-ACTIVATED	NO ALARM
HIGH ALARM 8	USER ASSIGNED	1 AMU	0.00 %	0.00 %	0 Sec	DE-ACTIVATED	NO ALARM

NOTE - ALARM LEVELS AND FUNCTIONS CAN ONLY BE SET FROM THE ALARM SET UP PAGE

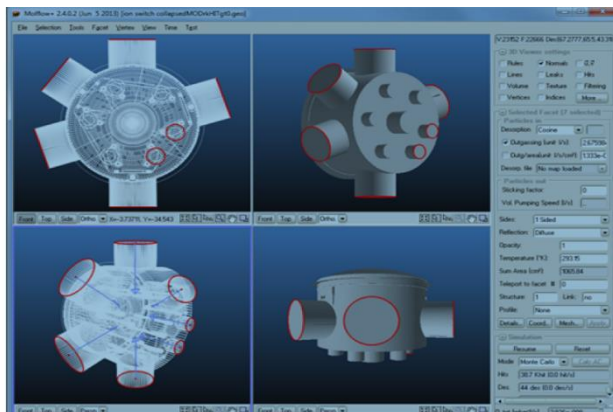
RGA MENU



Part 1 - Vacuum for Accelerators

Modelling software for accelerators

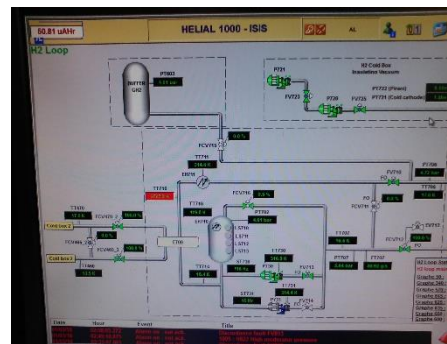
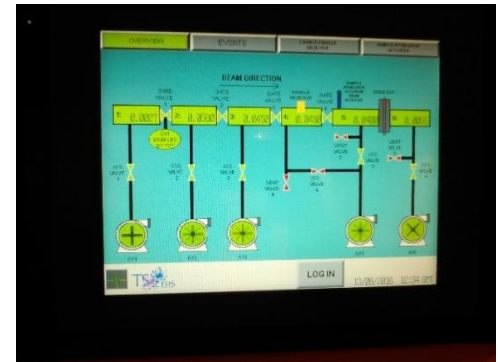
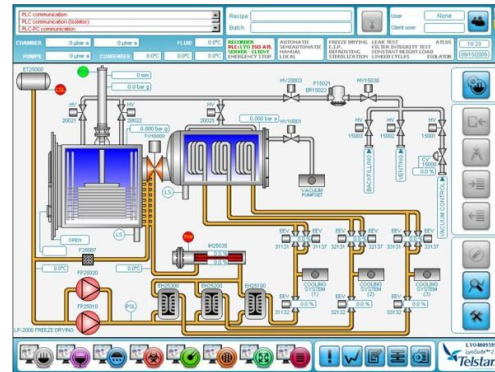
- Basic (Diffusion) Model – whilst quick and easy struggles with complicated vessel shapes.
- Better option is to use 3D test particle Monte-Carlo simulation eg. Molflow (R. Kersevan, CERN).



Part 1 - Vacuum for Accelerators

The Vacuum Control System

- LabView
- EPICS
- Device Net
- Profibus
- RS 485



Part 1 - Vacuum for Accelerators

Can be reduced by treating the internal surface of the vacuum vessel with a coating eg. Titanium or Titanium Nitride, NEG or Carbon, which reduce secondary electron yield. CSNS (China) uses 100nm thick coating on both quadrupole and dipole ceramic vessels.



Or

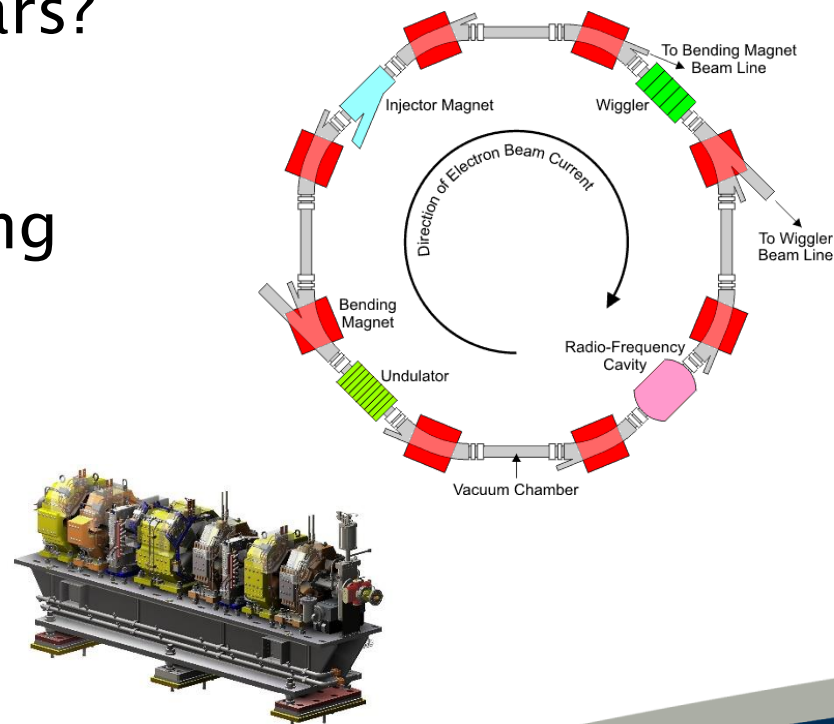
Apply an external field using a solenoid coil around the beampipe to keep stray electrons nearer the wall.



Part 1 - Vacuum for Accelerators

How has the design of accelerator vacuum systems changed over last 10 to 15 years?

- Control system and modelling system developments
- Vacuum modules (girders)
- Decommissioning costs need to be taken into consideration.



Part 2 – Surface Science

- Extensively used for studying catalysts, semiconductor and coatings.
- Need vacuum to eliminate gas scattering effects and maintain a clean surface for analysis.
- Basically for monolayer coverage

Vacuum level	Time (s) for monolayer coverage
Rough	10^{-3}
High	1
Ultra High	10^{+4}



Part 2 – Surface Science

Some surface science techniques – no one method is the best

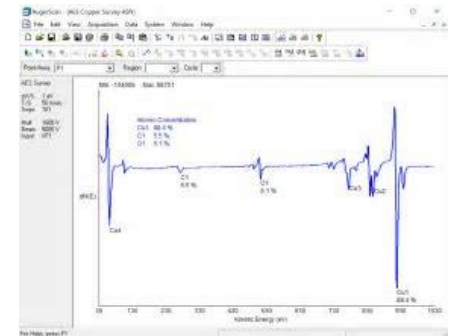
Auger Electron Spectroscopy	Ion Scattering Spectroscopy	Inverse Photoemission
Angle Resolved Photoemission	Low Energy Electron Diffraction	Rutherford Back Scattering
Atomic Force Microscopy	Near Edge X-Ray Absorption Fine Structure	Surface Enhanced Raman Spectroscopy
Electron Energy Loss Spectroscopy	Second Harmonic Generation	Surface Extended X-Ray Absorption Fine Structure
Field Emission Microscopy	Transmission Electron Microscopy	X-Ray Diffraction Spectroscopy
Field Ionization Microscopy	Thermal Desorption Spectroscopy	Fourier Transform Infrared Spectroscopy



Part 2 – Surface Science

Auger Electron Spectroscopy

- Elemental analysis of top 2nm of surface.
- Bombarding sample with a primary electron beam. This results in removal of an inner (K) shell electron and emission of an Auger electron.
- Use hemispherical analyser to analyse Auger electrons.
- Scanning Auger involves rastering beam over surface to build up an image



Part 2 – Surface Science

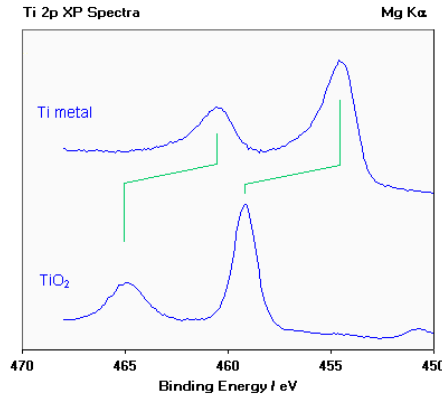
Photoelectron Spectroscopy (PES)

- X-rays (XPS) photon energy of 200 - 2000eV (core level analysis) or Ultraviolet (UPS) photon energy 10 – 45eV (valence bands)
- Need a monochromatic source of x-rays (X-ray gun), helium lamp or a synchrotron
- Like AES measure kinetic energy of emitted electron based on the following equation $KE = h\nu - BE$ (Binding Energy)
- Each element has a characteristic binding energy, peak intensity indicates concentration.

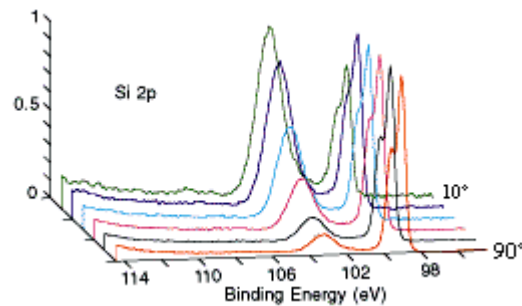


Part 2 – Surface Science

- Chemical shifts can be used to identify different oxidation states



- Angle dependent analysis can also be carried out by measuring the photoelectrons emitted at different angles



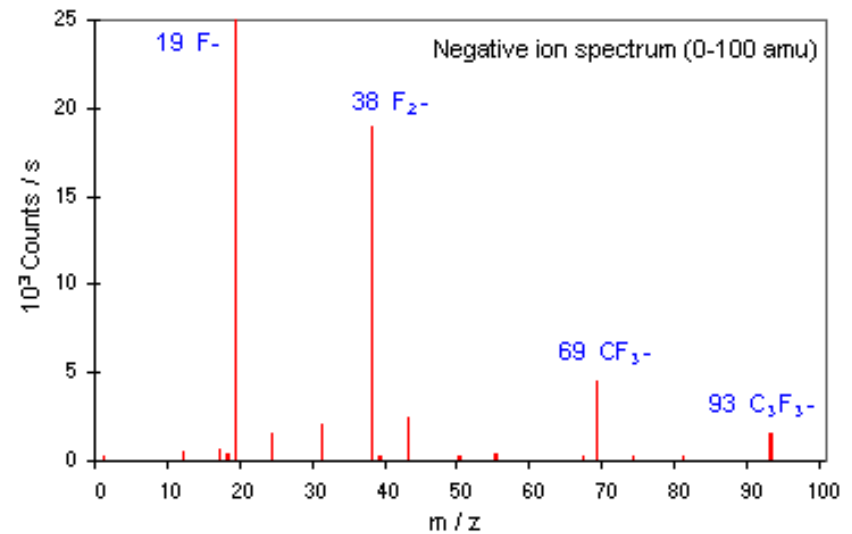
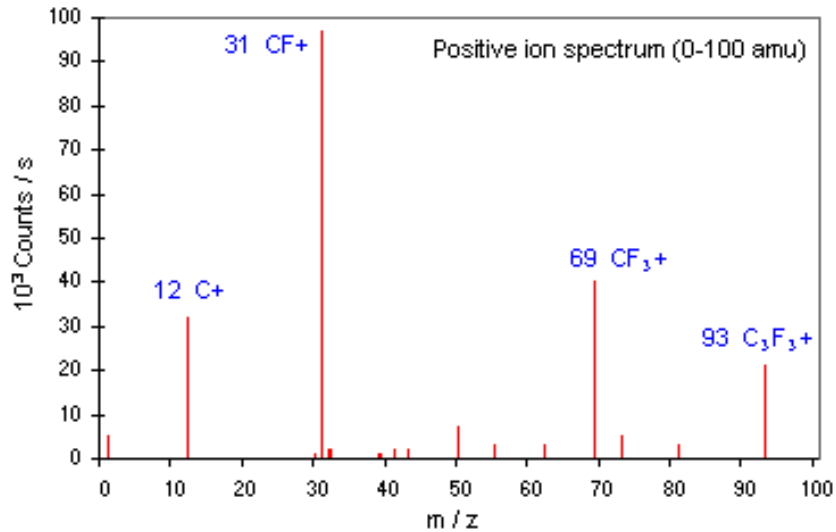
Part 2 – Surface Science

Secondary Ion Mass Spectrometry (SIMS)

- Sample bombarded with high energy ions (eg Ar^+), resulting in the emission of neutral and charged species that are then analysed using a mass spectrometer.
- Can carry out depth analysis of surface.
- Mass spectrometer could be quadrupole, magnetic sector or time of flight.



Part 2 – Surface Science



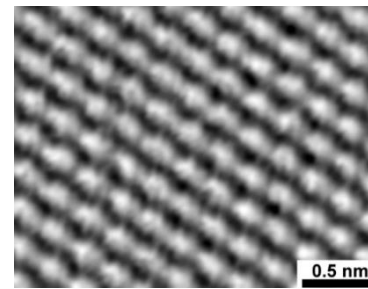
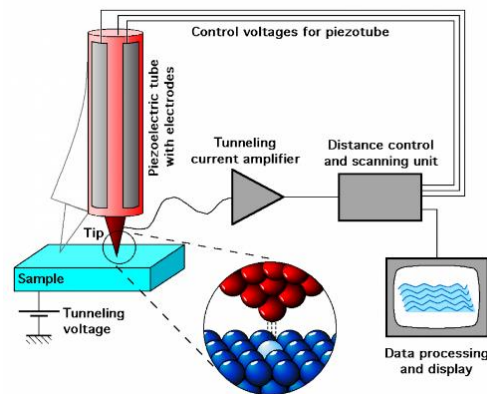
SIMS analysis of PTFE



Part 2 – Surface Science

Scanning Tunnelling Microscopy (STM)

- Requires a sharp tip (probe), prepared by cutting, chemical etching or surface “tapping” – single atom tip required for best resolution. Can be carried out at room or low temperatures.
- Normally the tunnelling current is kept constant whilst the tip moves along the surface. This results in an image of the surface.



Part 2 – Surface Science

Limitations of surface science techniques.

- Pressure Gap (real v's actual conditions)
- Probe beam effects – heating and sample charging



Sometimes Bad Things Happen On Accelerator Vacuum Systems !

