Vacuum Science and Technology in Accelerators

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Session 5

Materials Properties Relevant to Vacuum

Aims

To understand which mechanical properties of materials are relevant for their use in vacuum

To understand the role of vapour pressure and of gases in materials in vacuum

To understand the role of stimulated desorption

Introduction

Relevant Mechanical properties

- Strength (over desired range of temperatures)
- Hardness
- Expansion coefficients
- Machining and joining properties
- Corrosion resistance

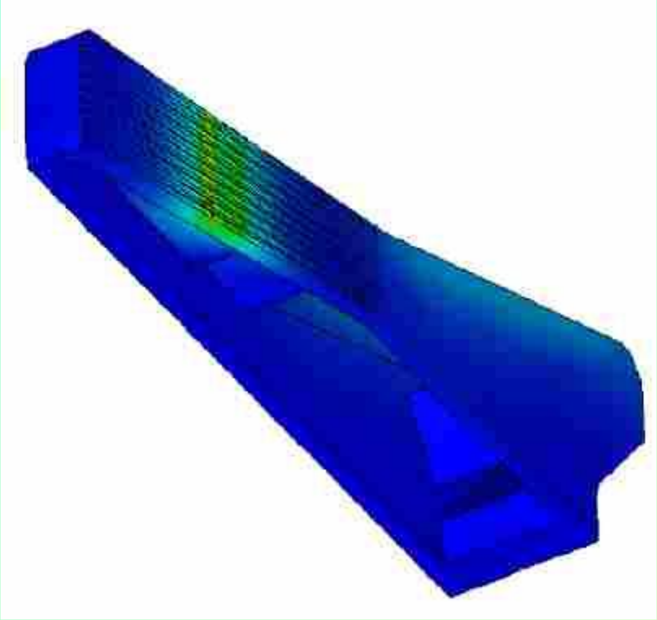
Relevant Physical Properties

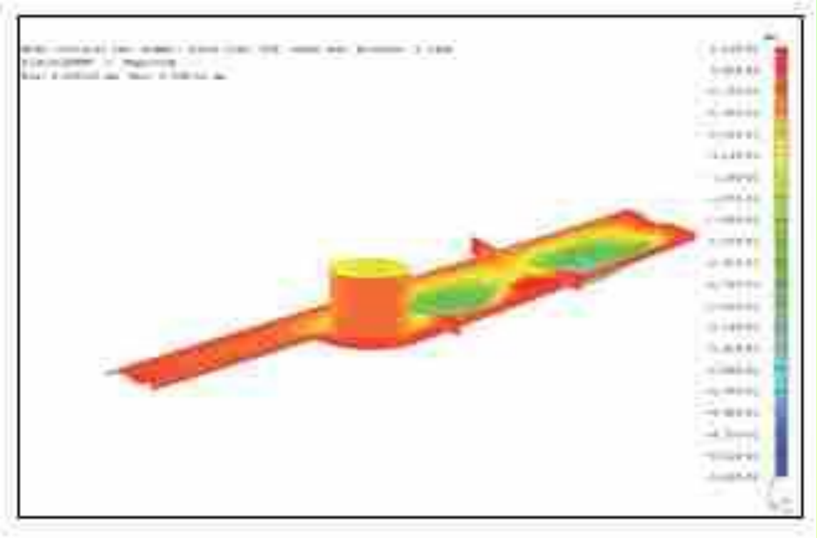
- Electrical conductivity
- Thermal conductivity
- Magnetic properties
- Permeability
- Residual Activity

Mechanical Properties

Wall loading is ~10.4 kg m^2

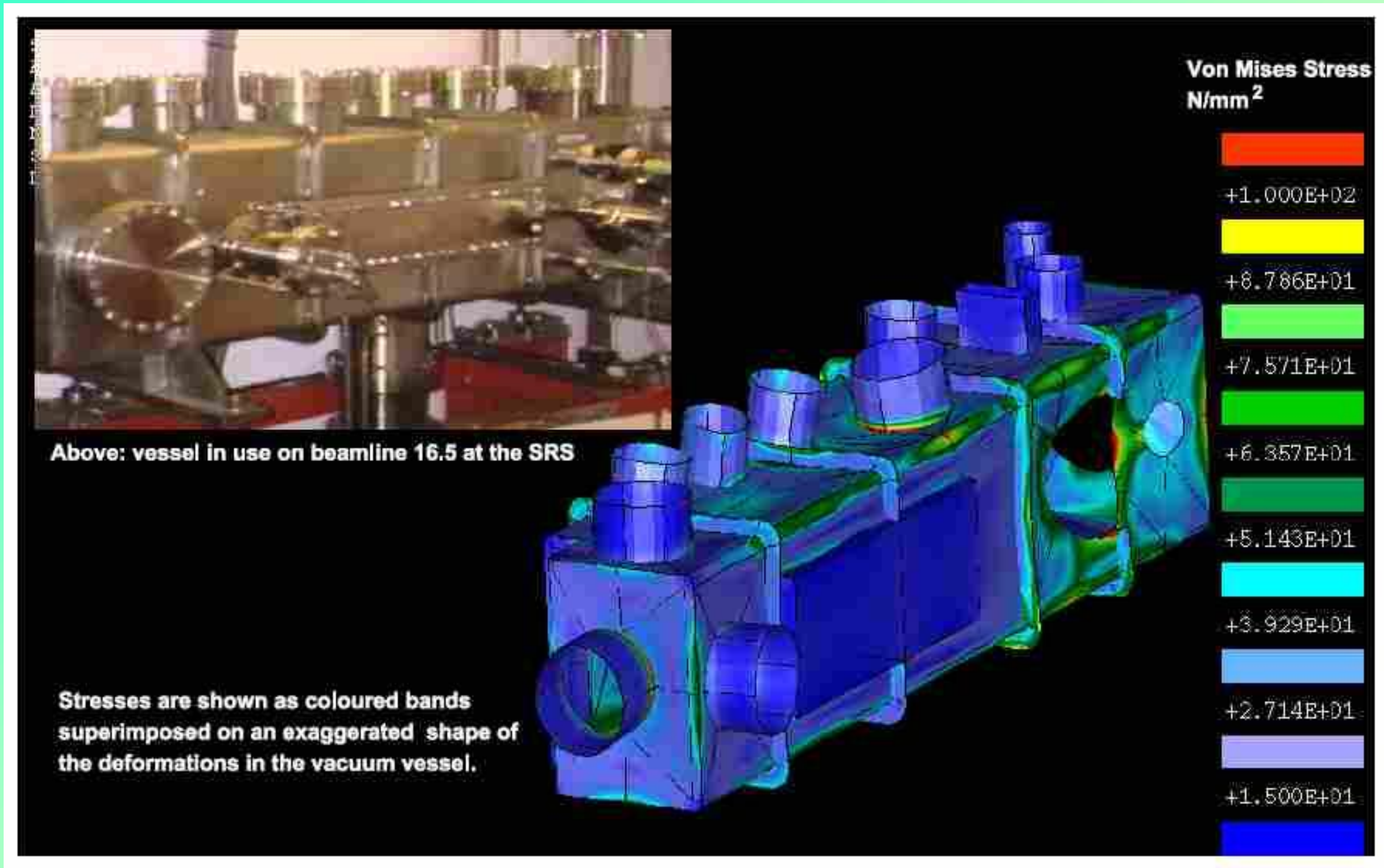
- Need to consider deflection of thin wall vessels
- FEA calculations





Materials Properties for Vacuum

Mechanical Properties



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

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

GRP Epoxy (low vacuum machines)

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.

Properties which influence the vacuum

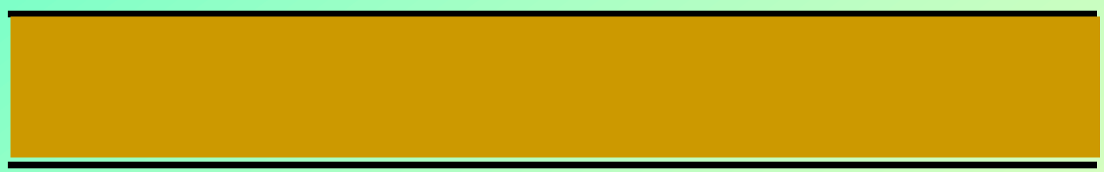
Outgassing

Desorption

Secondary Electron Yield

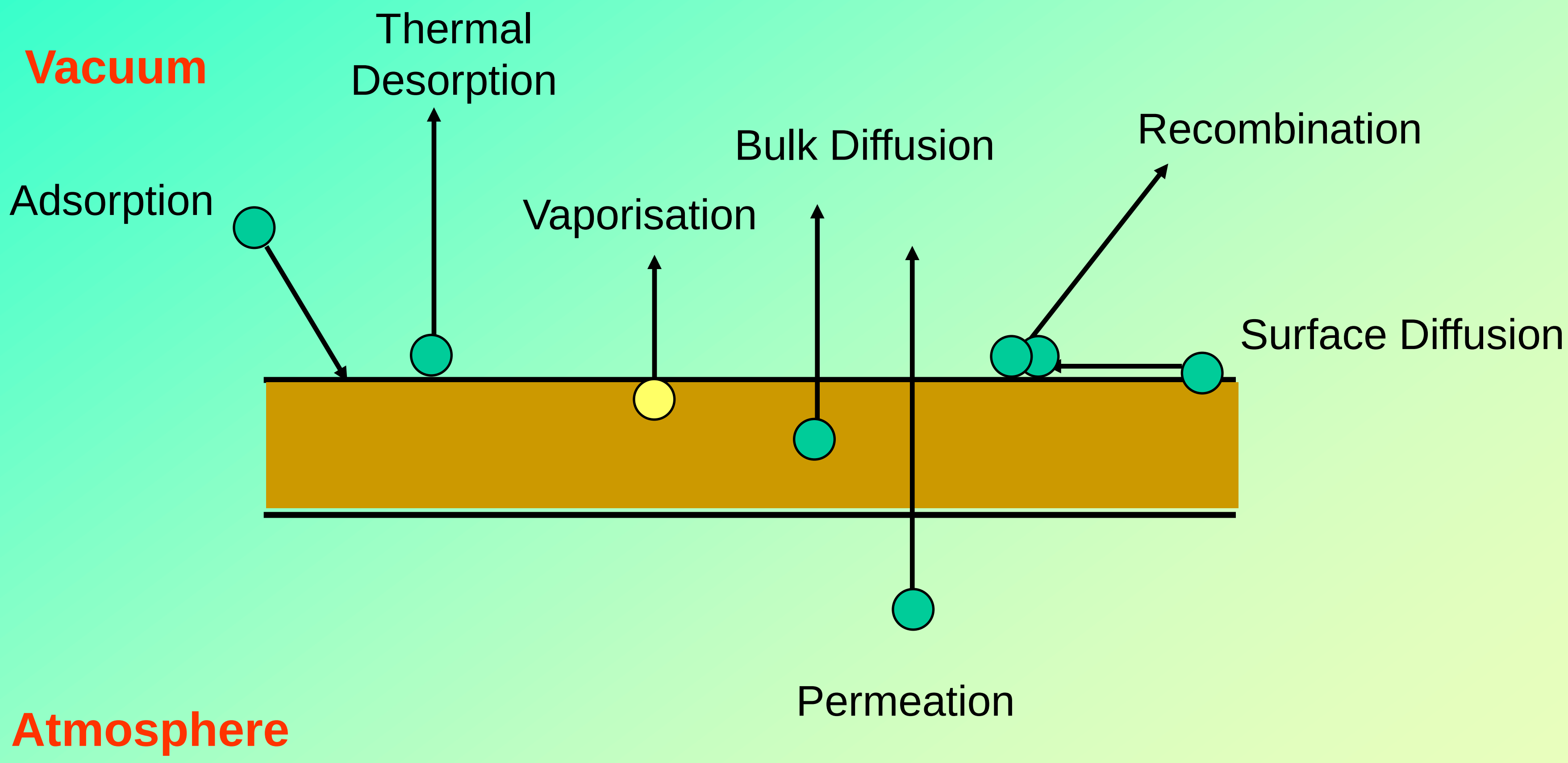
Mechanisms Contributing to Outgassing

Vacuum



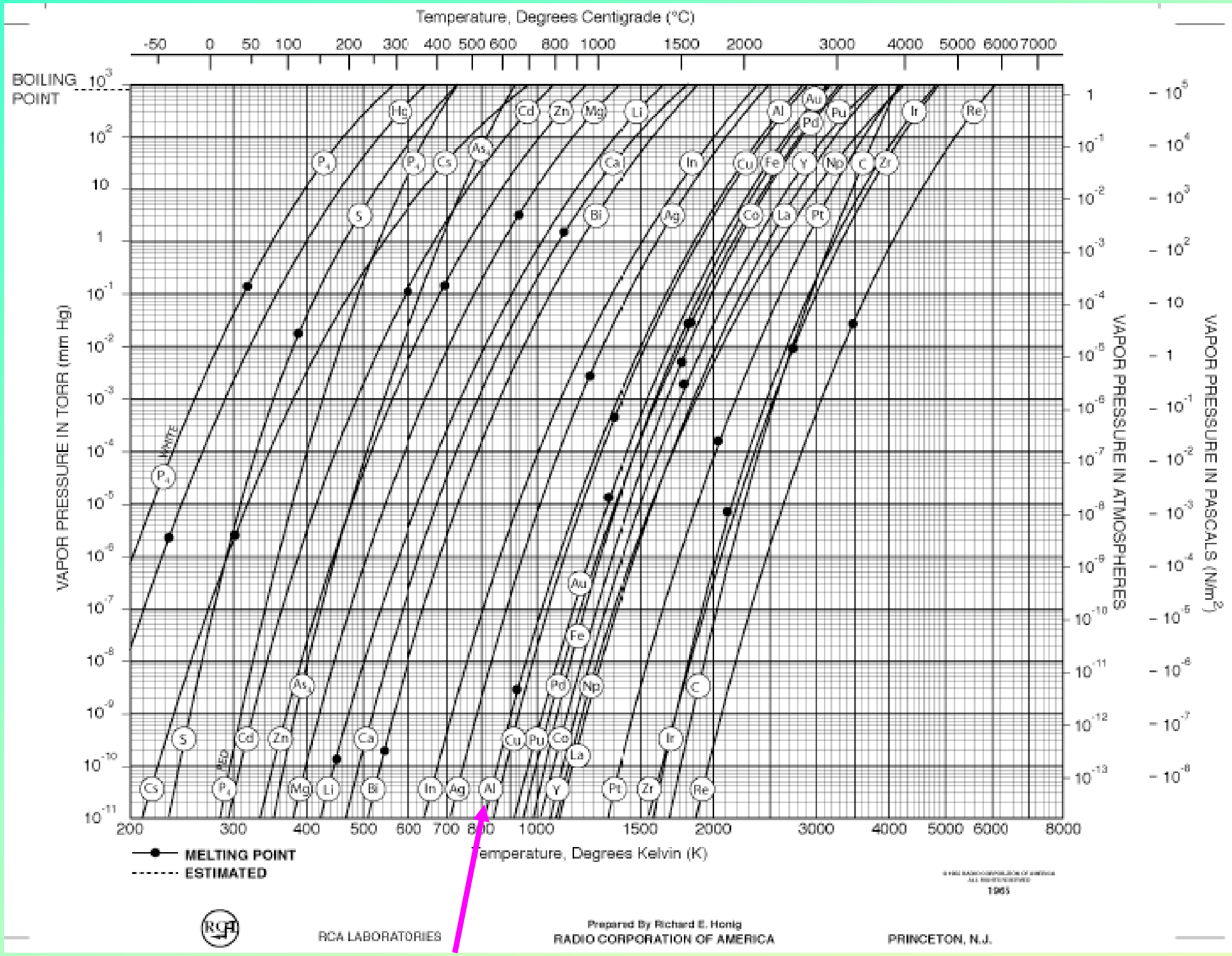
Atmosphere

Mechanisms Contributing to Outgassing



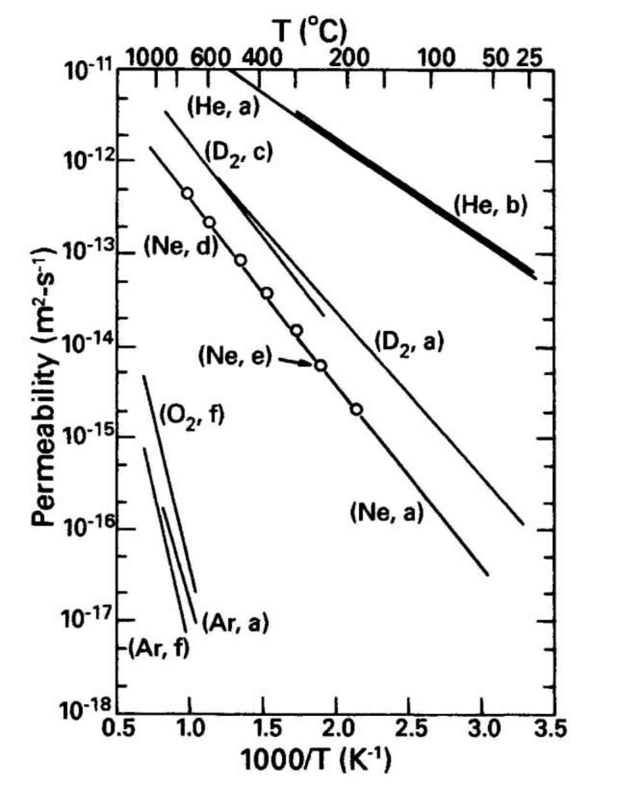
So to reduce outgassing, we must inhibit or reduce these processes

Vacuum Properties – Vapour Pressure

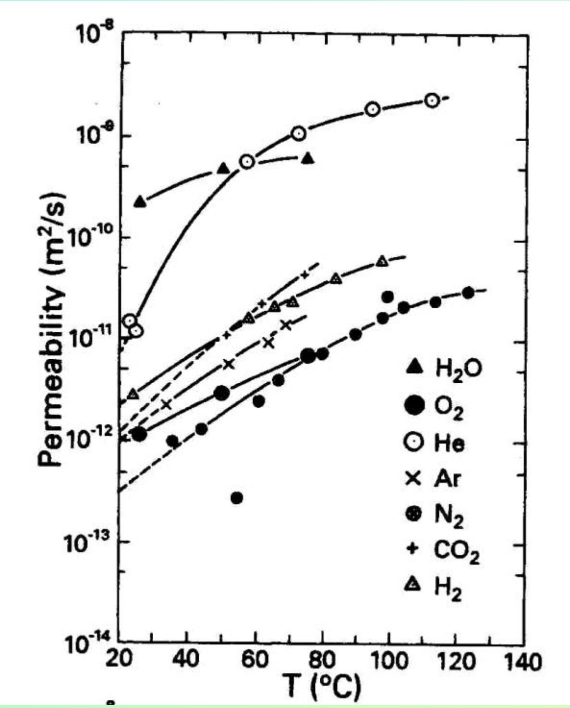


Materials Properties for Vacuum

Permeability

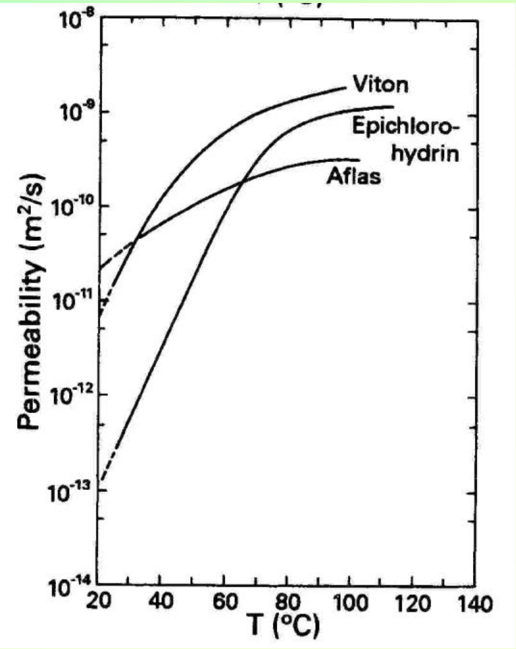


Permeability of gasses through glass

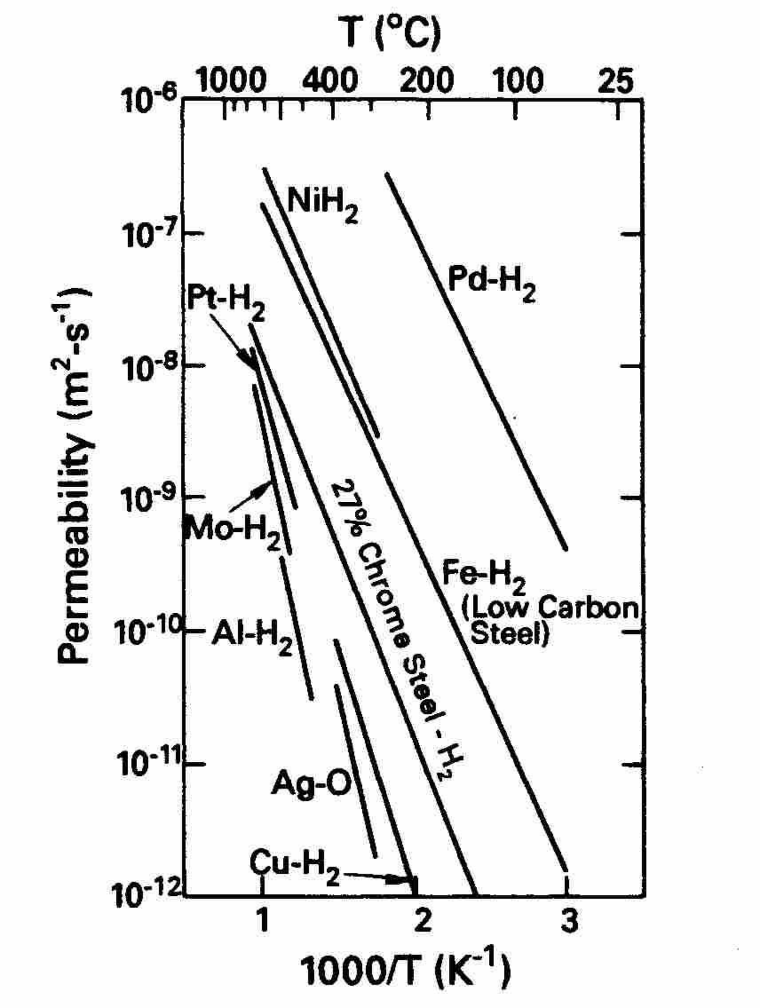


Permeability of gasses through Viton®

Permeability of helium through elastomers

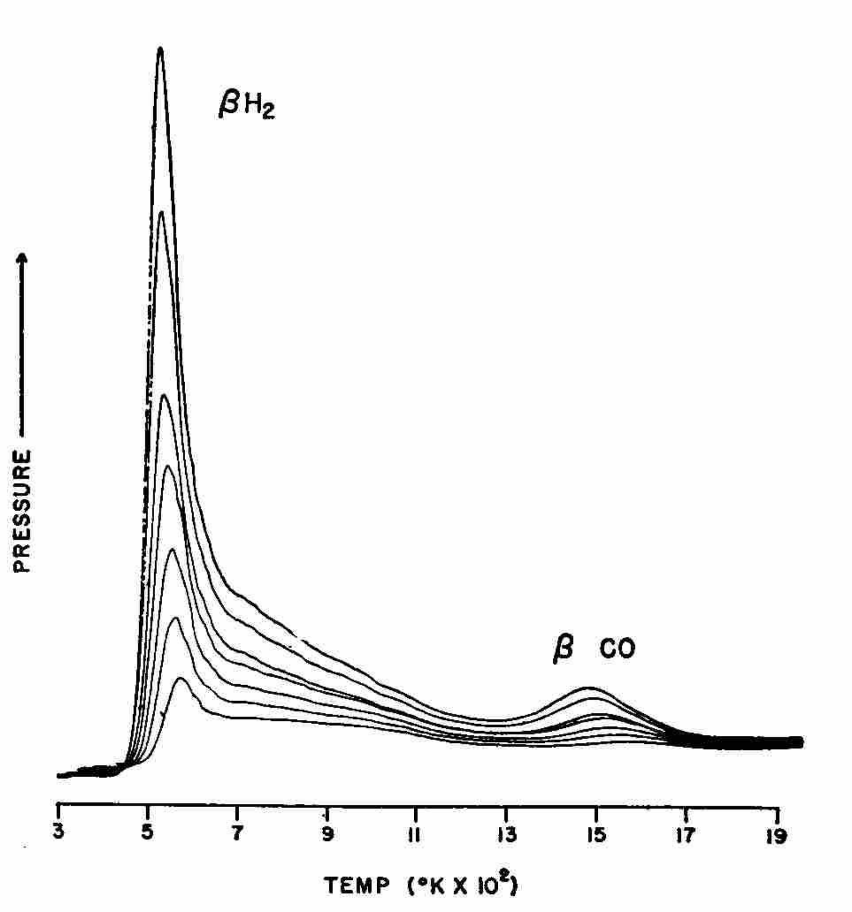


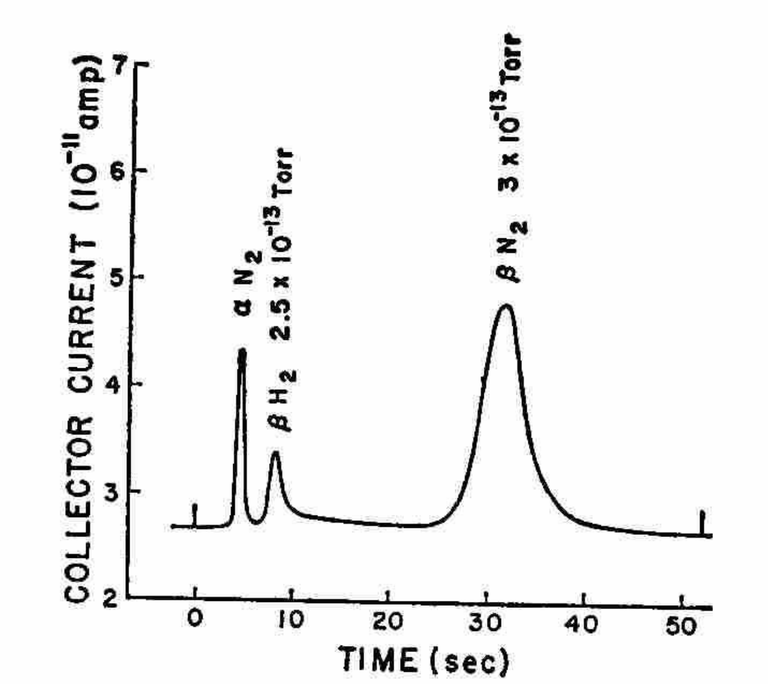
Permeability



Permeability of hydrogen through metals

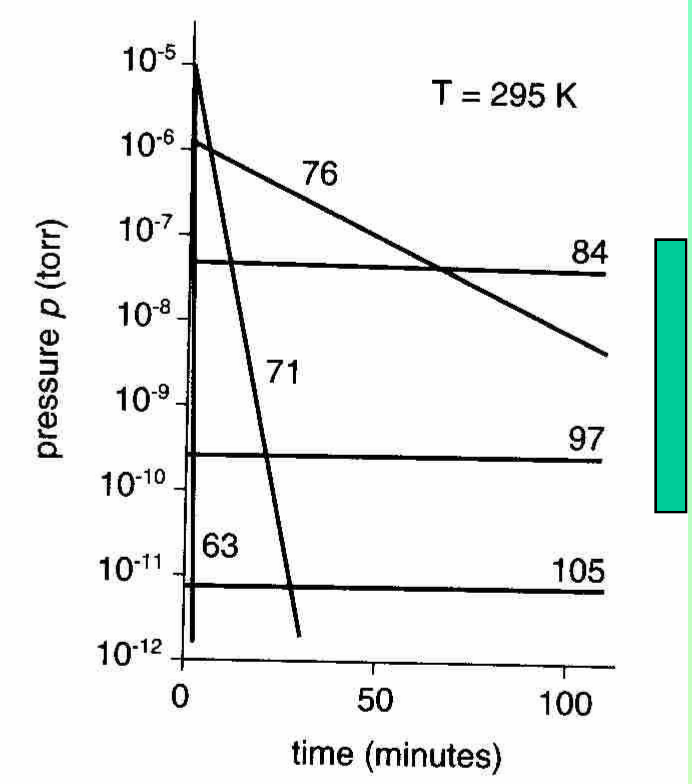
Vacuum Properties – Thermal Desorption



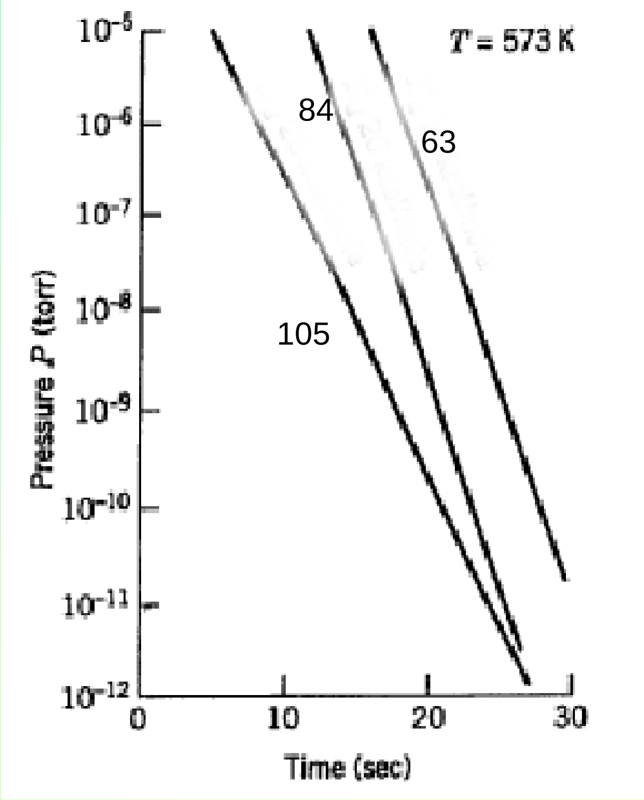


Vacuum Properties – Thermal Desorption

Under many circumstances, limit to pressure in a vacuum system will be be outgassing of water



Water



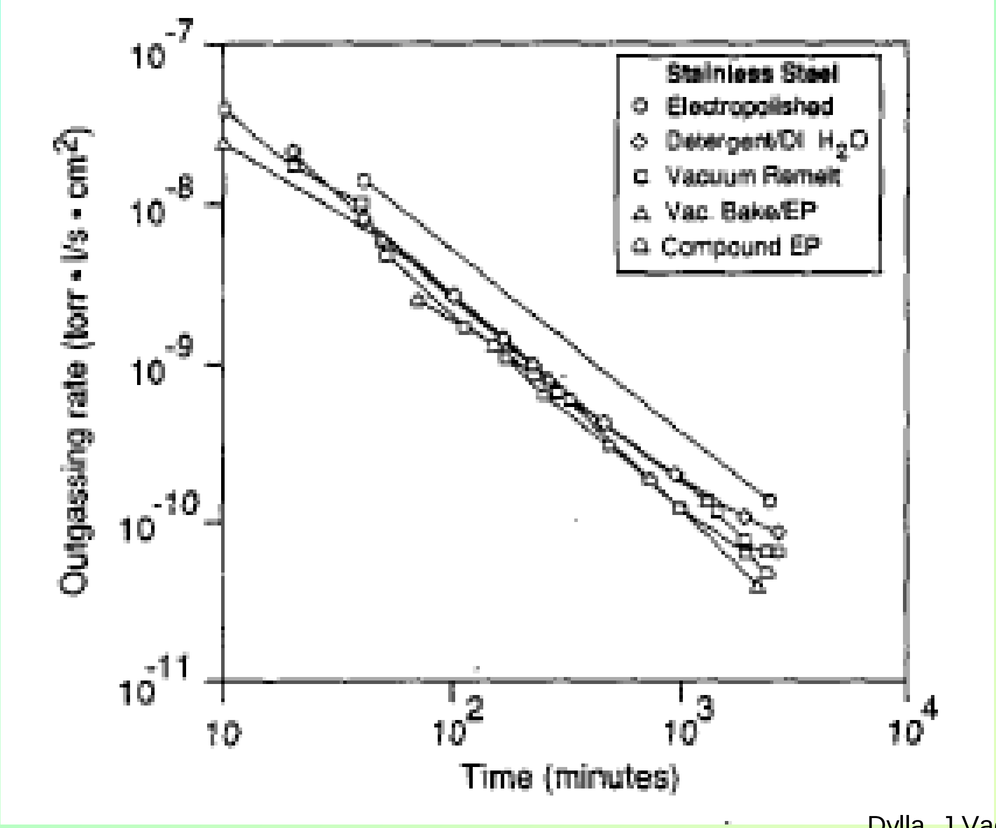
Energies of desorption in kJ mol^{-1}

Reducing outgassing

Permeation :	barrier layer on surface (internal or external)
Bulk diffusion :	reduce dissolved hydrogen, induce trapping states (bulk or surface)
	reduce or increase grain boundary density
Desorption :	reduce surface concentration
Adsorption :	reduce or fill surface binding sites
Recombination :	reduce surface mobility by introducing surface trapping sites

What affects Hydrogen outgassing from Stainless Steel?

- Conventional surface treatments (e.g. detergents, solvents, glow discharge) have only a small effect



Dylla, J Vac Sci Tech A11 (1993) 2623

Hydrogen Outgassing from Stainless Steel

For technological surfaces, surface roughness has little effect

Baking at moderate temperatures reduces the outgassing rate

Successive bakes reduce the rate further

Baking in air reduces the rate considerably

High temperature vacuum firing reduces the rate considerably

Thin wall vacuum vessels have lower outgassing rates than thick wall vessels

Hydrogen Outgassing from Stainless Steel

Possible effect of barrier layers (surface oxides)

Possible effect of depleting bulk hydrogen

Possible effect of inducing depletion layer at surface

Possible effect of changes in composition and/or structure

Possible effect of changing number or distribution of trapping states (bulk/surface)

Possible role of recombination/dissociation processes

Current Models

There is no *a priori* method at present for calculating outgassing. Current models are presented in-

Outgassing rate determined by bulk diffusion, Calder & Lewin, Brit J Appl Phys 18 (1967) 1459

Outgassing rate determined by recombination at the surface, Moore, J Vac Sci Technol A13 (1995) 545

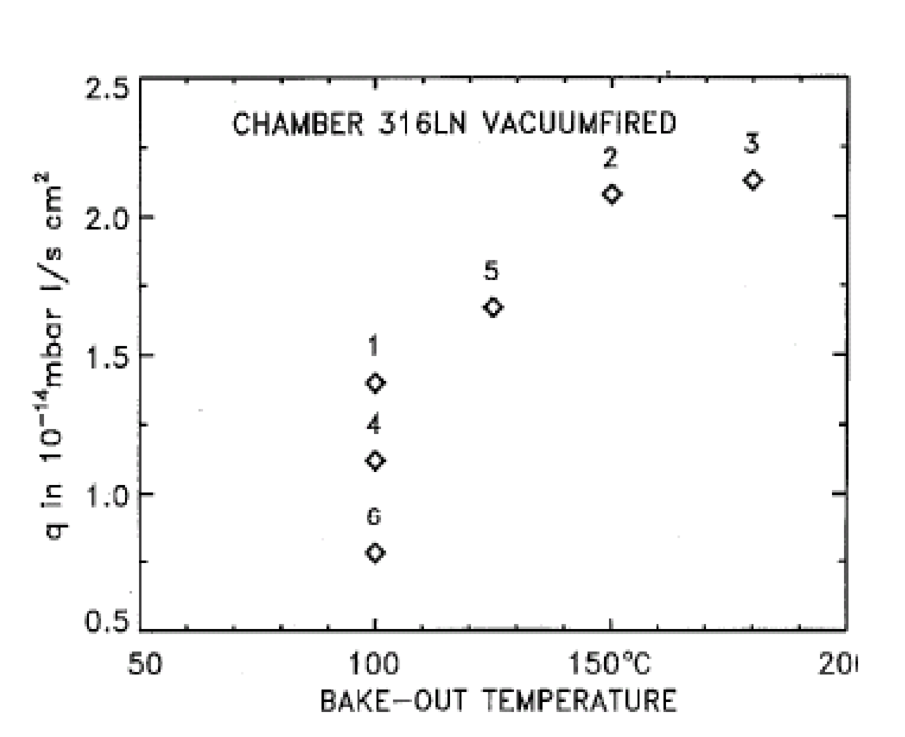
P Chiggiato *in* CAS - CERN Accelerator School : Vacuum in Accelerators, Platja d'Aro, Spain, 2006, to be published (http://cas.web.cern.ch/cas/Spain-2006/Spain-lectures.htm)

Bakeout temperature

Conventional wisdom -

Within limits, the higher the temperature of bakeout the better (time*temperature ~constant)

However



Jousten, Vacuum 49 (1998) 359

What can we conclude?

- The outgassing of hydrogen from stainless steel is complex
- Several different processes are involved
- Fundamental properties, e.g. diffusion rates, not known to sufficient accuracy
- Better controlled, systematic studies are required

What can be achieved?

Experience shows that an outgassing rate of the order of 10^{-11} mbar l s^{-1} cm^{-2} is readily obtainable

An outgassing rate of 10^{-13} mbar l s^{-1} cm^{-2} is achievable with conventional techniques

Rates lower than 10^{-14} mbar l s^{-1} cm^{-2} are achievable with care, particularly in thin walled vessels

Published values of outgassing rates

If we now consider outgassing from various materials, and not limit the species to hydrogen

- There is a great deal of data available in the literature
- Few systematic compilations
- One can almost select a value for a particular system of whatever one wants
- Certainly great spread in data and it is inconsistent
- Measurement conditions not standardised
- Measurement technique not standardised
- Pretreatments often not stated
- Materials often not well characterised

Published values of outgassing rates

So is this a council of despair?

Clearly, absolute values not well founded

Number of valuable studies looking at *trends*

Folklore can be of considerable use

However, design work often based on unreliable and/or extrapolated data

Designs tend to be conservative and therefore not optimised.

Strategies for Reducing Outgassing

Thorough clean and bake – remove contamination

Vacuum fire - depletion

Air bake – oxide film – barrier layer or depletion

Film such as TiN - barrier layer

Getter film – barrier? trap?

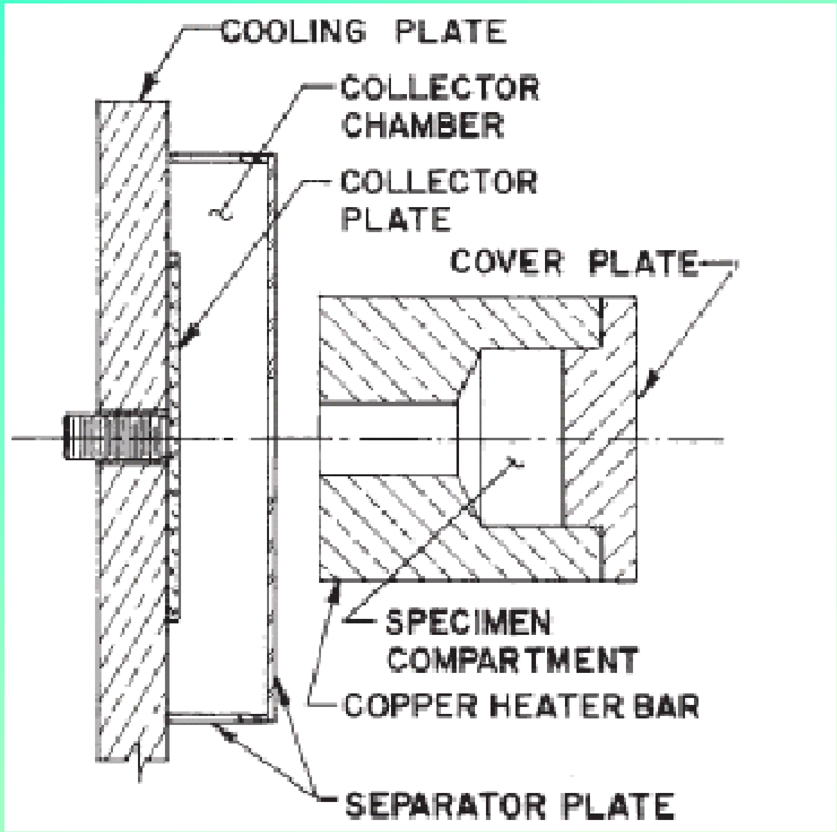
Methods of Measuring Outgassing

Three basic techniques

- Weight loss
  - Space Industry
  - Most extensive compilations – Nasa, ESA (LIGO)
- Throughput
- Rate-of-rise (gas accumulation)

Methods of measuring outgassing

Weight Loss Technique



$$Q = \frac{dM}{dt} \cdot \frac{RT}{M}$$

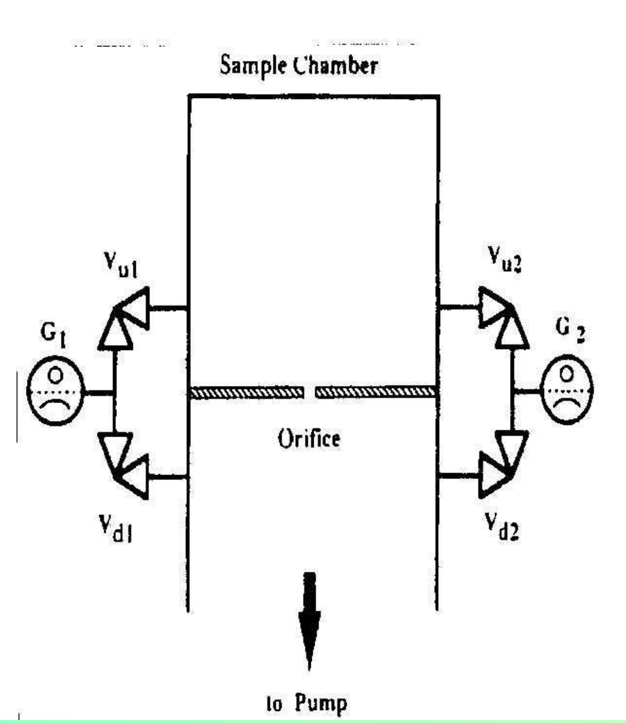
$$Q = 1.4 \times 10^{4} \cdot \frac{dM}{dT} \cdot \frac{1}{M} \quad \text{mbar l s}^{-1}$$

If the outgassing species is water, and we can detect 1µg s^{-1},

$Q = 6.10^{-4}$ mbar l s^{-1}

Methods of measuring outgassing

Throughput Method



$$Q = (P_1 - P_2)\cdot\frac{C}{A}$$

Conductance Modulation

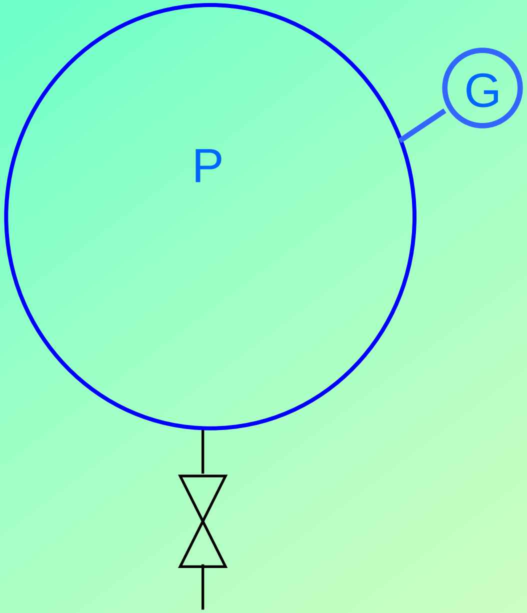
$$Q = \frac{p_1 - p_2}{A\left(\frac{1}{C_1} - \frac{1}{C_2}\right)}$$

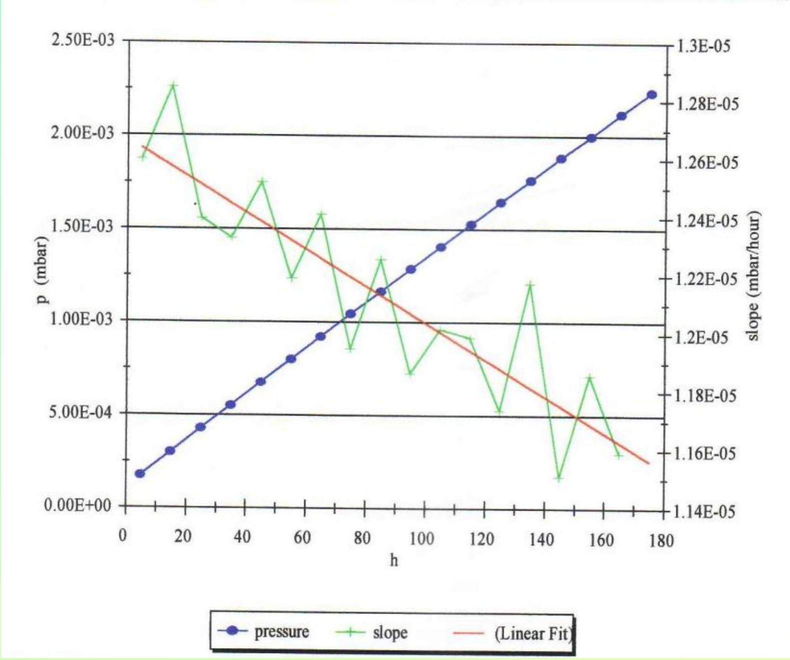
Jousten in Lafferty, *Foundations of Vacuum Science and Technology*, 1998

Methods of measuring outgassing

Rate of Rise (gas accumulation)

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





Nemanic & Setina

Real Samples

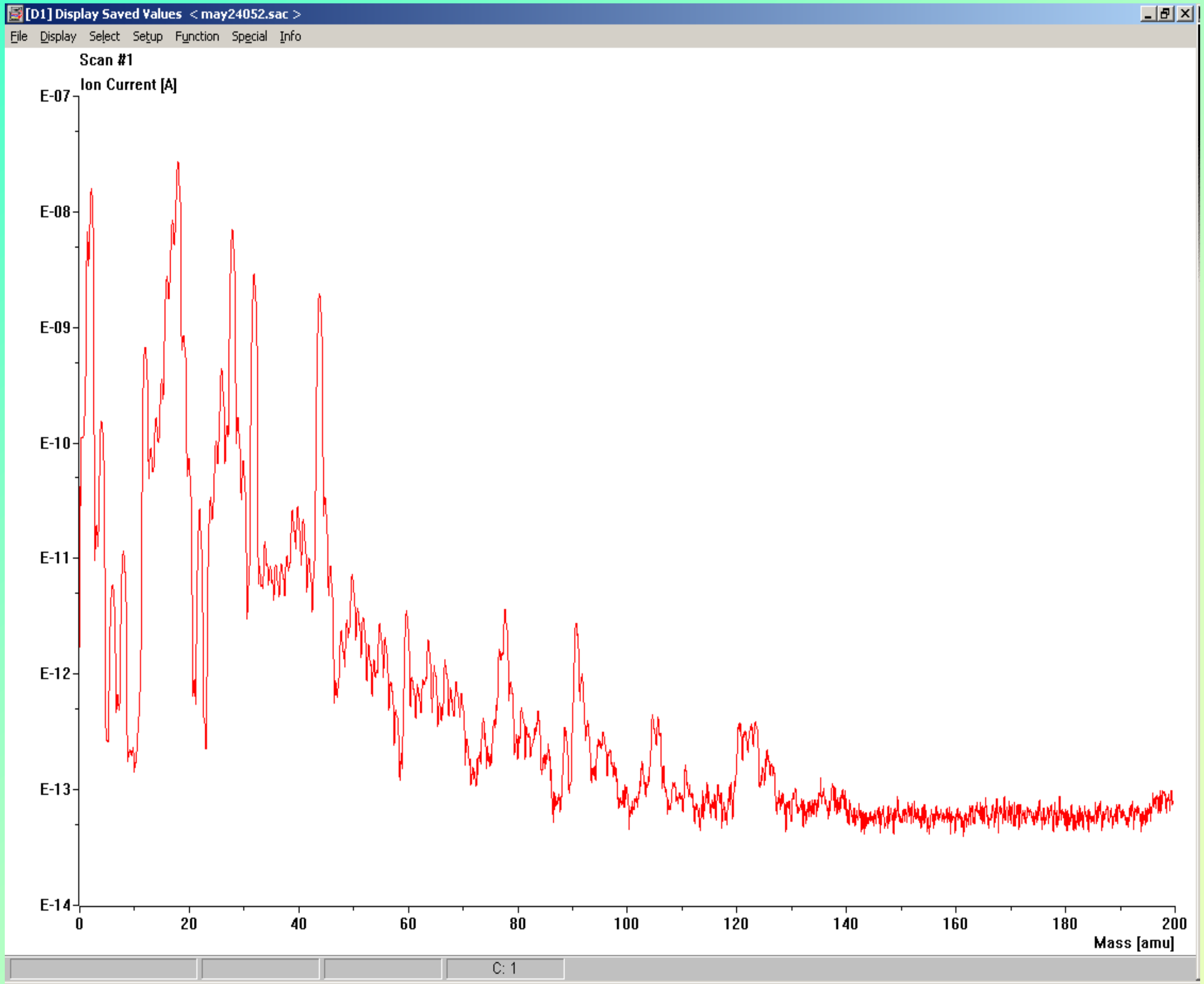
For real samples, outgassing can be complicated and we almost certainly want to know what species are being given off.

We look at a few real samples

Materials Properties for Vacuum

Epoxy Resin - Used as Vacuum Sealant

Specific Outgassing rate (pre-bake) = 5.9×10^{-9} mbar l s^{-1} cm^{-2}

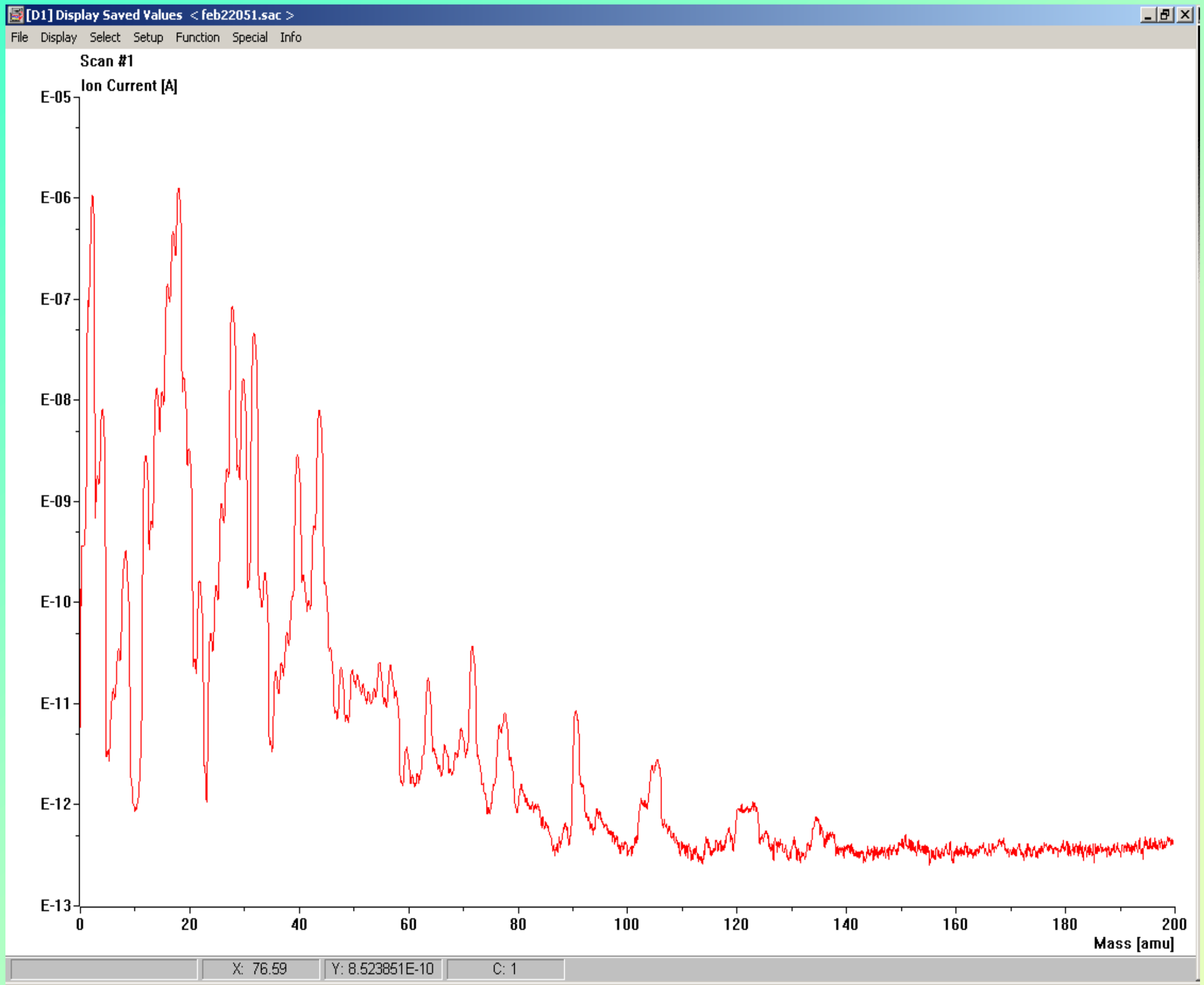


R J Reid

Materials Properties for Vacuum

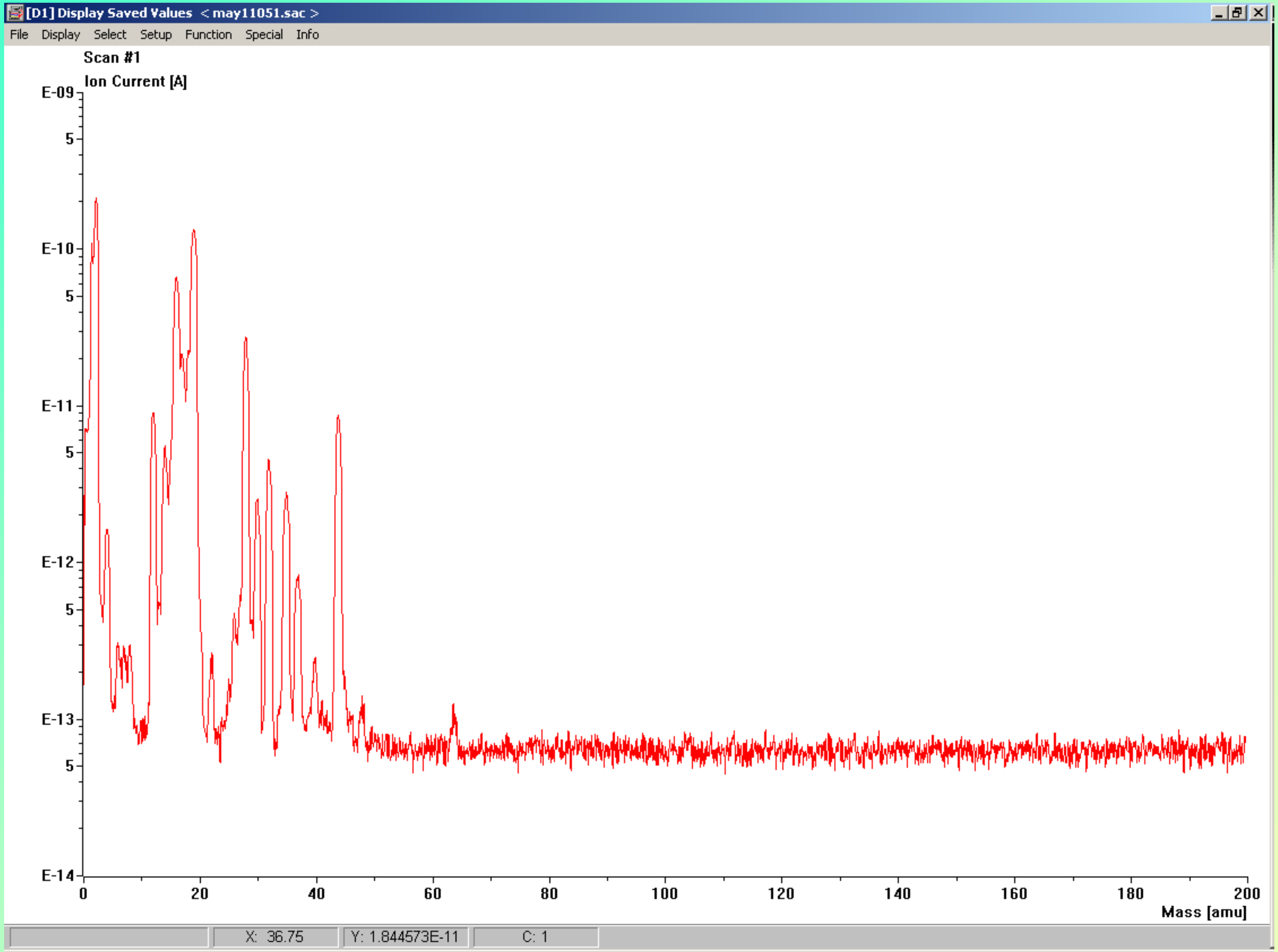
Boron Carbide - Used as Neutron Absorbers on ISIS

Specific Outgassing rate (pre-bake) = 4 x 10^{-9} mbar l s^{-1} cm^{-2}



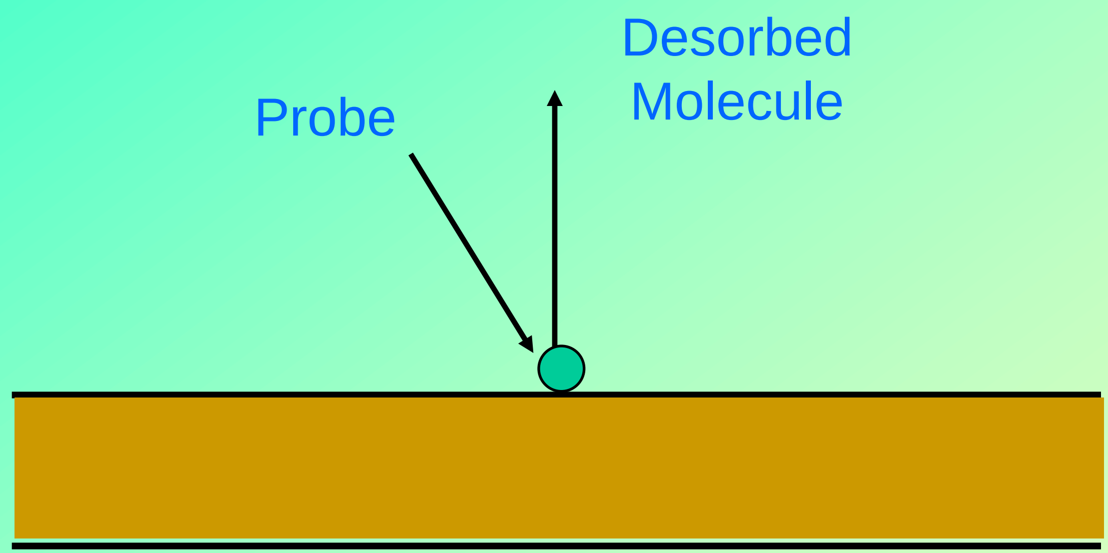
Viton Sealed valve

Leak Rate (post-bake after 10000 cycles) = 5×10^{-11} mbar l s^{-1}



Materials Properties for Vacuum

Stimulated Desorption



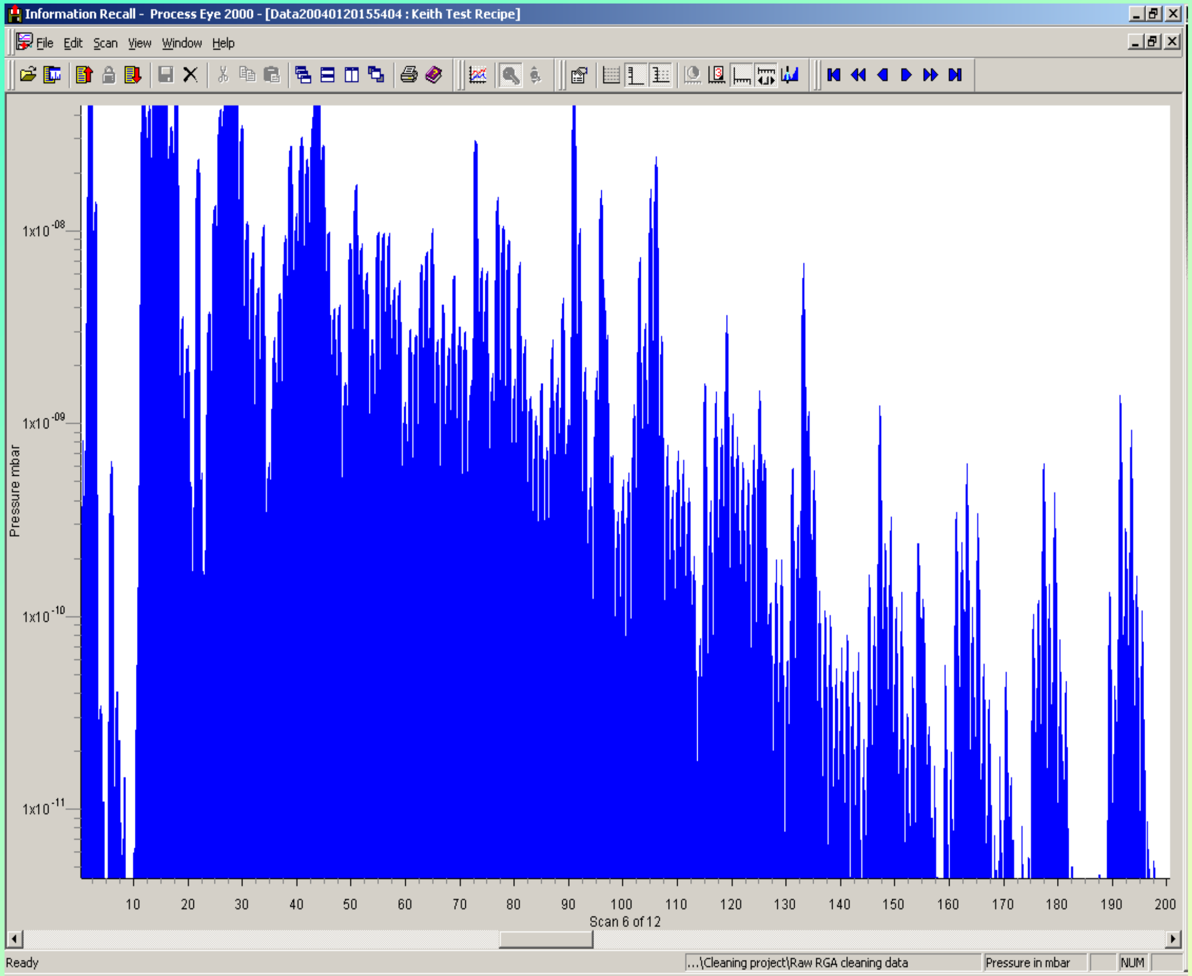
Probe can be an electron, photon or ion.

The processes are similar, but cross sections are different, so yields are different.

As for outgassing, there is no good theory for calculating yields at present.

Materials Properties for Vacuum

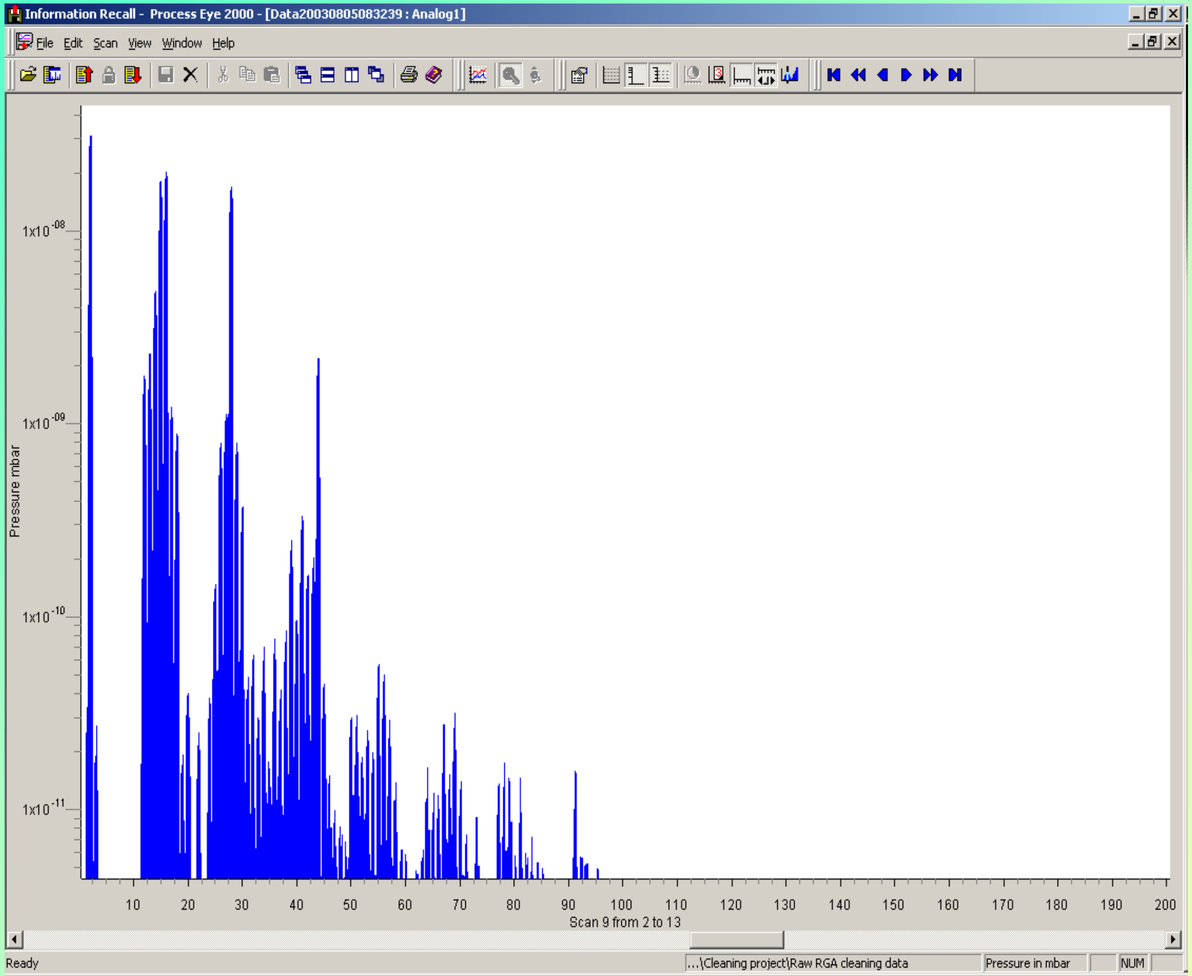
304L ST ST - Contaminated and then ultrasonically cleaned with aqueous cleaner.
RGA scan taken during ESD.
Specific Outgassing rate (post-bake) = 2.9×10^{-12} mbar l s^{-1} cm^{-2}



Materials Properties for Vacuum

304L ST ST - Contaminated and then ultrasonically cleaned with hydrofluoroether solvent. RGA scan taken during ESD.

Specific Outgassing rate (post-bake) = 5.7 x 10^{-13} mbar l s^{-1} cm^{-2}

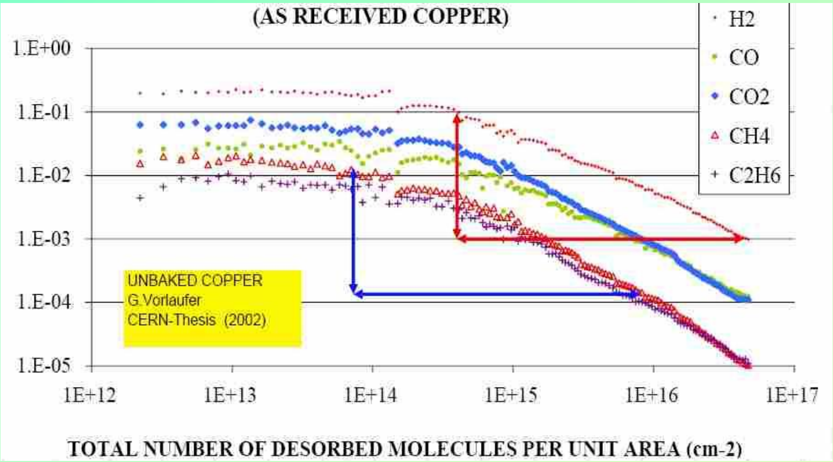


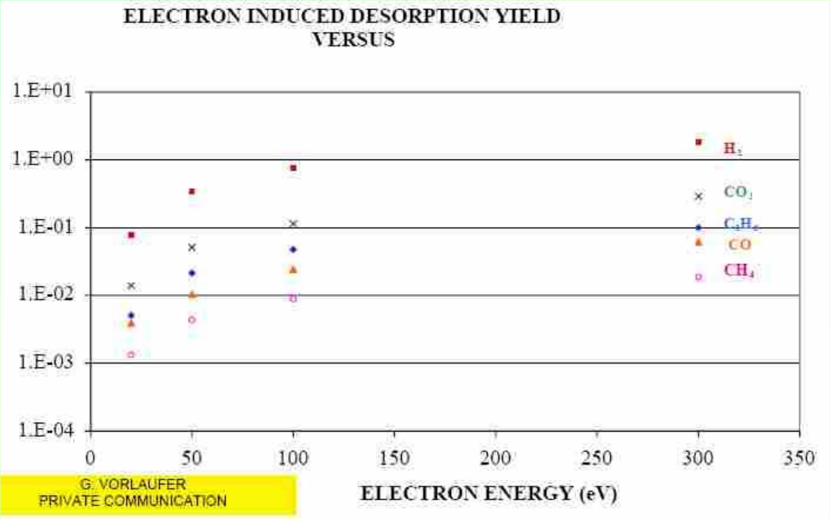
Materials Properties for Vacuum

Electron Stimulated Desorption

ESD yields can be high. It can be a problem in hadron and heavy ion machines, so care may have to be taken to ensure that secondary electron production is minimised.

However, the phenomenon of beam cleaning (“scrubbing”) is helpful.

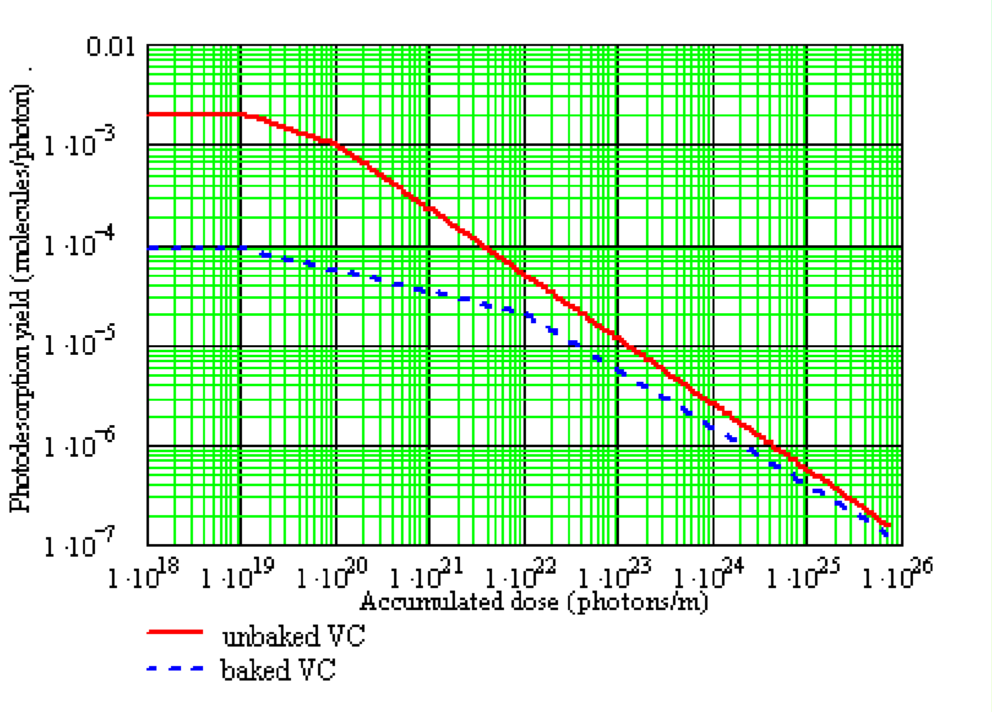




Materials Properties for Vacuum

Photon Stimulated Desorption

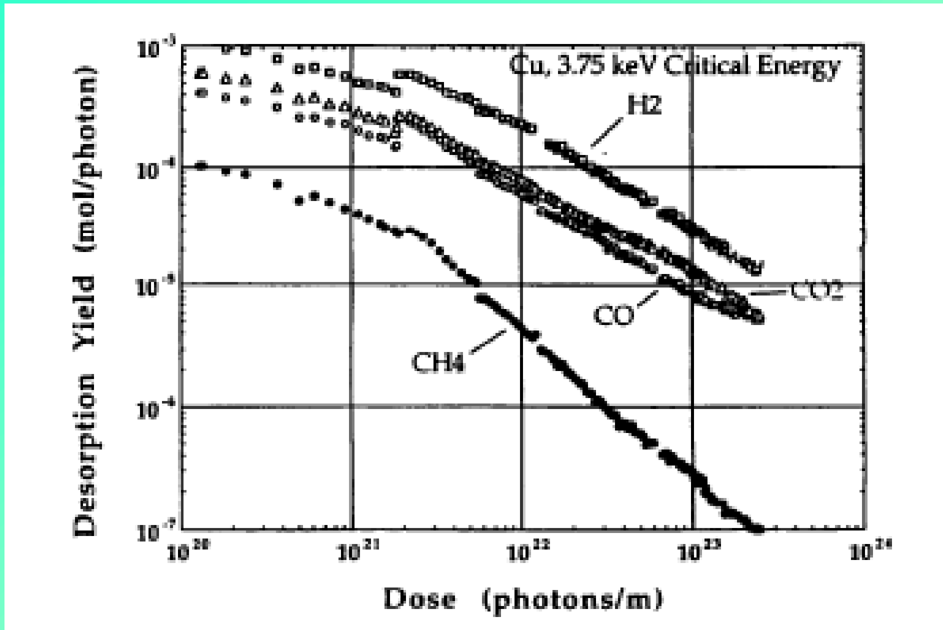
There is a wealth of pragmatic information from synchrotron light sources to help us to design accelerators.



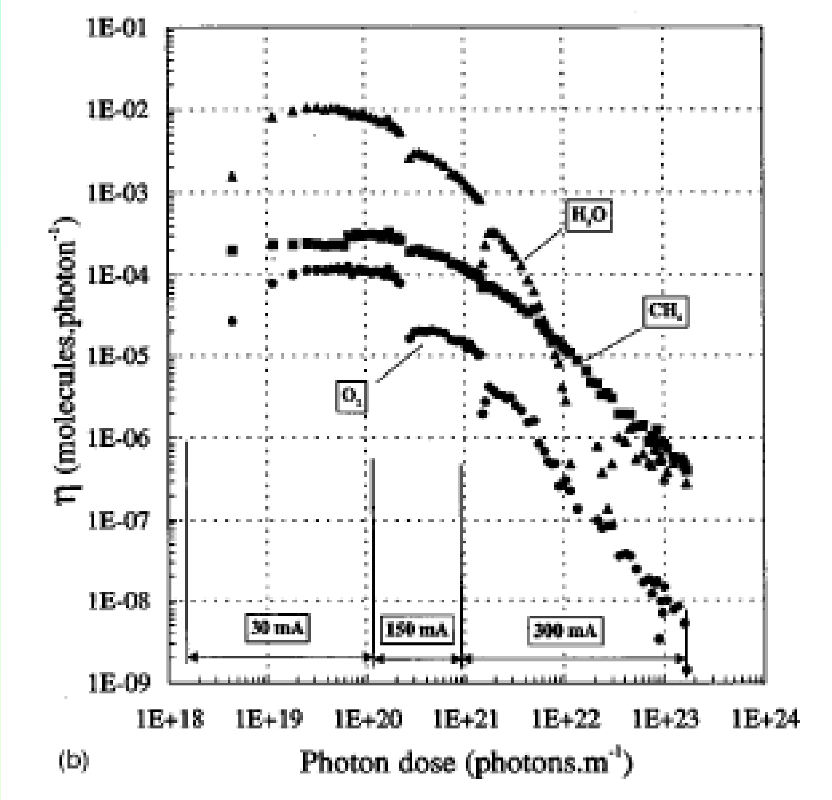
"Idealised" yield curves for baked and unbaked stainless steel

Materials Properties for Vacuum

Photon Stimulated Desorption



C Foerster, private communication

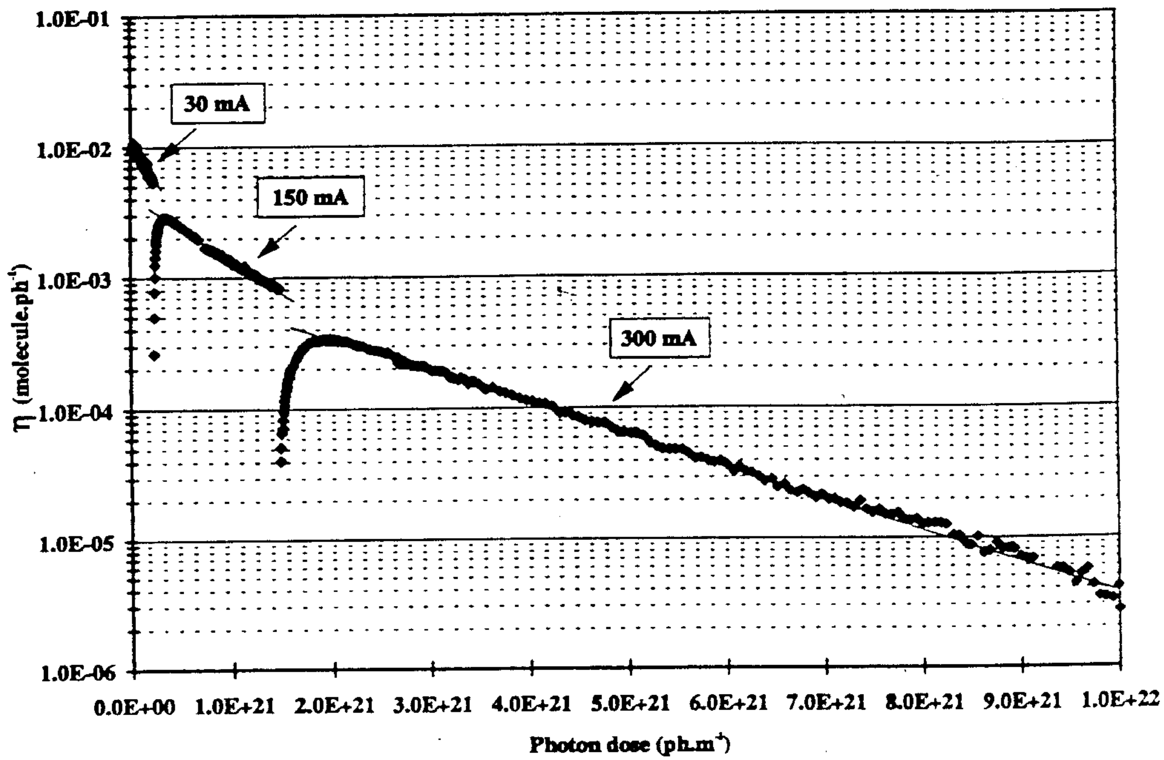


C Herbeaux & P Marin, J Vac Sci Technol **A17** 635 1999

Desorption yields are beam dose dependent

Materials Properties for Vacuum

Photon Stimulated Desorption



Photon stimulated desorption of H_2O, Showing an exponential variation.

C Herbeaux & P Marin, J Vac Sci Technol **A17** 635 1999

Fortunately, all these effects are minimised by the same strategy – careful cleaning and processing remove contamination from surfaces and deplete the surface layer gas reservoirs.

The addition of passivation layers or active barriers also helps considerably.