CAS, Small Accelerator course, Zeegse, Netherlands, 24 May 2 June 2005

# Vacuum System

Oswald Gröbner

- 1) Introduction and some basics
- 2) Vacuum pumps and gauges
- 3) Gas desorption
- 4) Components and materials

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# Literature

#### Books

The Physical Basis of Ultrahigh Vacuum, P.A. Redhead, J.P. Hobson, E.V. Kornelsen, American Vacuum Society Classics, American Institute of Physics, 1993
Foundations of Vacuum Science and Technology, Ed. J.M. Lafferty, John Wiley & Sons, 1998
Handbook of Accelerator Physics and Engineering, A. W. Chao, M. Tigner, World Scientific, 1998
CAS CERN Accelerator School : Vacuum Technology, Ed. : S. Turner. CERN 99-05, 19 August 1999
Handbuch Vakuumtechnik, M. Wutz et. al, Vieweg, Braunschweig/Wiesbaden, 2000

#### Journals:

#### VACUUM

Journal of Vacuum Science and Technology (A) Nuclear Instruments and Methods (Section A)

#### **Pressure and Molecular Density**

**Ideal gas law:** 
$$P V = \frac{N}{No} R T$$

*P* pressure, *V* volume, *T* temperature *N* number of molecules *R* gas constant = 8.31 kJ kmol<sup>-1</sup> K<sup>-1</sup>, *No* = 6.02 10<sup>26</sup> molecules kmol<sup>-1</sup>

Molecular density  $n = N_V$ 

**Pressure** : P = n k T

Boltzmann constant  $k = 1.38 \ 10^{-23} \ \text{J/K}$ 

Note : R = No k

Note: In nearly all cases, it is the gas density rather than the pressure which matters.

<u>Units :</u>

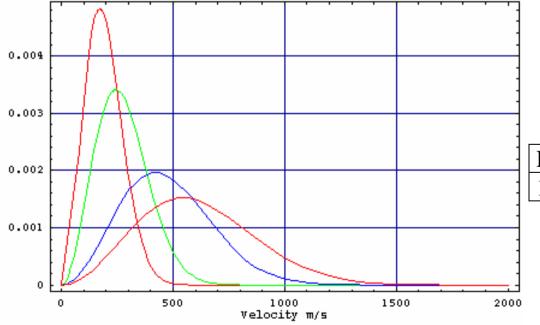
Pressure :Pa 
$$(N/m^2)$$
, mbar = 100 Pa, Torr = 133 PaGas load :Pa  $m^3 = 7.5$  Torr l, mbar l ~ 2.4  $10^{19}$  molecules at RTSpecific outgassing rate :Gas release from the wallsPa  $m^3/s/m^2 ~ 7.5 \ 10^{-4}$  Torr l/s/cm²Leak rate :Pa  $m^3/s$  or W, mbar l/s or Torr l/s

## **Distribution of Molecular Velocities**

Maxwell-Boltzmann distribution of molecular velocities at the temperature T

$$\frac{1}{N}\frac{dN}{dv} = \frac{4}{\sqrt{\pi}} \left(\frac{m}{2kT}\right)^2 v^2 e^{-\frac{mv^2}{2kT}}$$
  
The average velocity is given by (m = M m<sub>o</sub>):  $\bar{v} = \sqrt{\frac{8kT}{\pi M m_o}}$ , numerically ~146 $\sqrt{\frac{T}{M}}$  (m s<sup>-1</sup>)

Molecular velocities for  $N_2$  at 50, 100, 300 and 500K.



Mean molecular velocities at  $20^{\circ}$ C (m/s)

$H_2$	$N_2$	Air	А	Kr
1754	470	464	393	272

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#### Mean Kinetic Energy

The kinetic energy : 
$$E_{kin} = \frac{1}{2}m\bar{v}^2 = \frac{1}{2}Mm_o\left(\frac{8kT}{\pi Mm_o}\right) = \frac{4}{\pi}kT$$

*M* molecular weight,  $m_o = 1.66 \ 10^{-27} \ \text{kg}$ 

does not depend on the molecular mass, M, but only on temperature T.

In **thermal equilibrium** heavy molecules move sufficiently slowly and light molecules move sufficiently fast to carry on average the same kinetic energy.

#### **Total and Partial Pressures**

For each gas component  $n_1, n_2, n_3,...$  the contribution to the total pressure :  $P_i = n_i kT$ The total pressure is the sum of all partial pressures:  $P = \sum_i P_i = kT \sum_i n_i$ 

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Gas	%	Pi (Pa)			
$N_2$	78.1	$7.9 \ 10^4$			
$O_2$	20.5	$2.8 \ 10^3$			
Ar	0.93	$1.2 \ 10^2$			
$CO_2$	0.0033	4.4			
Ne	1.8 10 <sup>-3</sup>	2.4 10-1			
Не	5.2 10 <sup>-4</sup>	7 10 <sup>-2</sup>			

Partial pressures for atmospheric air

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#### Wall collisions

Rate of molecular impacts on the walls v =

$$v = \frac{1}{4} n \,\overline{v}$$

#### **Mean Free Path**

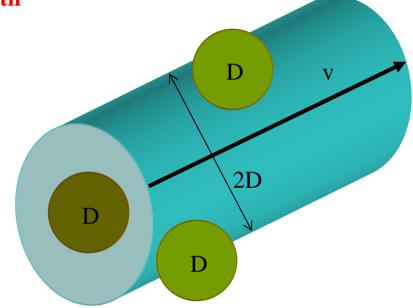
$$l = \frac{l}{\sqrt{2} \pi D^2 n}$$

D molecular diameter ( $\sim 3 \ 10^{-8} \ m$ )

Distance traversed per second  $\overline{v}$ Molecule collides with other molecules contained within a cylinder of radius *D*. Number of collisions:  $Z \approx \pi D^2 \overline{v} n$ Mean free path  $l = \frac{\overline{v}}{Z} = \frac{1}{\sqrt{2} \pi D^2 n}$ 

It also follows that  $n \ l \propto P \ l \approx const$ .

For air  $n l \approx const$  is ~ 2.5  $10^{14}$  m<sup>-2</sup> for N<sub>2</sub> at 20 °C and 1 Pa ->  $l \sim 9$  mm



#### **Molecular Flow Conditions**

**Knudsen relation**: gas flow  $Q \propto \Delta P$  applies if the mean free path >> relevant dimensions of system

Molecular flow conductance

$$c = \frac{4}{3} \frac{\overline{v}}{\int_{0}^{L} \frac{H}{A^2} dl} \qquad (\text{m}^3/\text{s})$$

L length of the element (L >> transverse dimensions). H perimeter, A cross section of the element.

The conductance is proportional to the mean molecular velocity, i.e. to  $\sqrt{\frac{T}{M}}$ .

A cylindrical duct with uniform section and radius r :

$$c = \frac{4}{3}\overline{v}\left(\frac{r^3}{L}\right) \sim 306 \cdot \left(\frac{r^3}{L}\right) \sqrt{\frac{T}{M}}.$$

 $c = \frac{1}{4} \overline{v} A \sim 36.5 \cdot A \sqrt{\frac{T}{M}}.$ 

An orifice (pumping orifice, L~0) :

Conductance of elements in series or in parallel add the same as for electric circuits Series :  $\frac{1}{c} = \frac{1}{c_1} + \frac{1}{c_2}$  and parallel:  $c = c_1 + c_2$ 

For complicated geometries it is often necessary to use Monte Carlo calculations for the molecular flow.

## **Vacuum characteristics**

# Summary expressions:

$$n = \frac{P}{kT}$$
  
kT = 4.04 10<sup>-21</sup> Joule

$$\rho = M m_0 n$$

$$M m_0 = 4.65 \ 10^{-26} \text{ kg}$$

$$m_0 = 1.66 \ 10^{-27} \text{ kg}$$

$$\nu = \frac{1}{4} n \overline{\nu} \text{ and } \overline{\nu} = 146 \sqrt{\frac{T}{M}}$$

$$l = \frac{1}{\sqrt{2}\pi D^2 n}$$

$$D (N_2) = 3.15 \ 10^{-10} \text{ m}$$

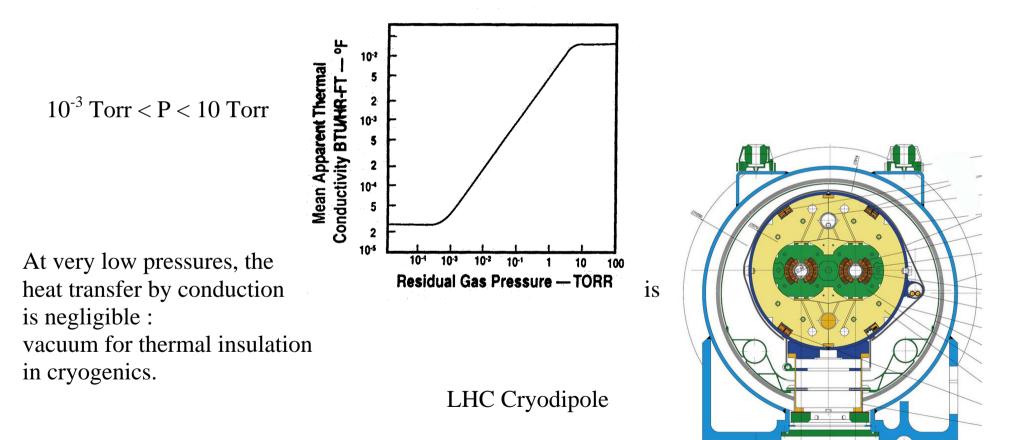
gas : Nitrogen, N<sub>2</sub>, 20°C, M = 28

Pressure	Р	n	ρ	V	l
	Ра	m <sup>-3</sup>	kg m <sup>-3</sup>	$m^{-2}s^{-1}$	m
atm	$10^{5}$	$2.5 \ 10^{25}$	1.16	2.9 10 <sup>27</sup>	9 10 <sup>-8</sup>
primary	1	$2.5 \ 10^{20}$	1.16 10 <sup>-5</sup>	2.9 10 <sup>22</sup>	9 10 <sup>-3</sup>
vacuum	10-1	2.5 10 <sup>19</sup>	1.16 10 <sup>-6</sup>	2.9 10 <sup>21</sup>	9 10 <sup>-2</sup>
high	10-4	$2.5 \ 10^{16}$	1.16 10 <sup>-9</sup>	2.9 10 <sup>18</sup>	9 10 <sup>1</sup>
vacuum	10-7	$2.5 \ 10^{13}$	1.16 10 <sup>-12</sup>	2.9 10 <sup>15</sup>	9 10 <sup>4</sup>
uhv	10-10	$2.5 \ 10^{10}$	1.16 10 <sup>-15</sup>	2.9 10 <sup>12</sup>	9 10 <sup>7</sup>
xhv	<10 <sup>-11</sup>				

# **Thermal Conductivity**

Thermal conductivity of a gas is independent of the pressure when the pressure is well above the molecular flow regime.

In the transition regime, the heat transfer is proportional to the pressure and to the temperature difference. Principle of pressure measurement with a Pirani gauge.



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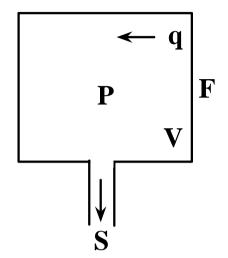
#### **Basic Vacuum System**

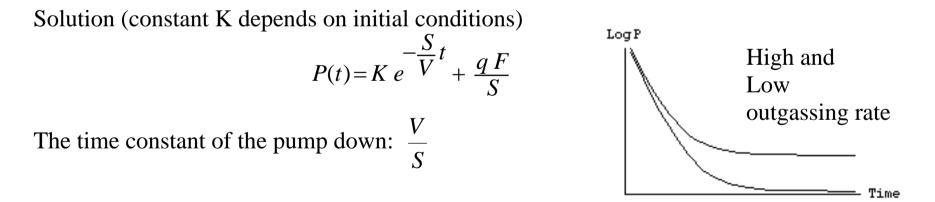
V volume  $(m^3)$ , F surface  $(m^2)$ P pressure (Pa), S pumping speed  $(m^3/s)$ q specific outgassing rate (Pa  $m^3/s/m^2$ )

Stationary conditions (P is independent of volume)  $P = \frac{q}{S}$ 

Dynamic pressure

$$V \frac{\partial P}{\partial t} = q F - S P$$





To reach low pressures: Low outgassing rate of the surface

large pumping speed and No leaks !

# **Rotary Pumps**

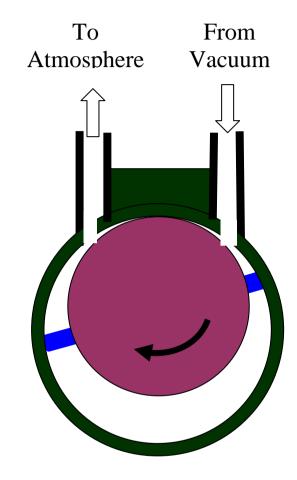
Single stage and double stage pumps

Oil sealed moving pistons

Typical end pressure :  $10^{-2}$  to  $\sim 10^{-3}$  mbar

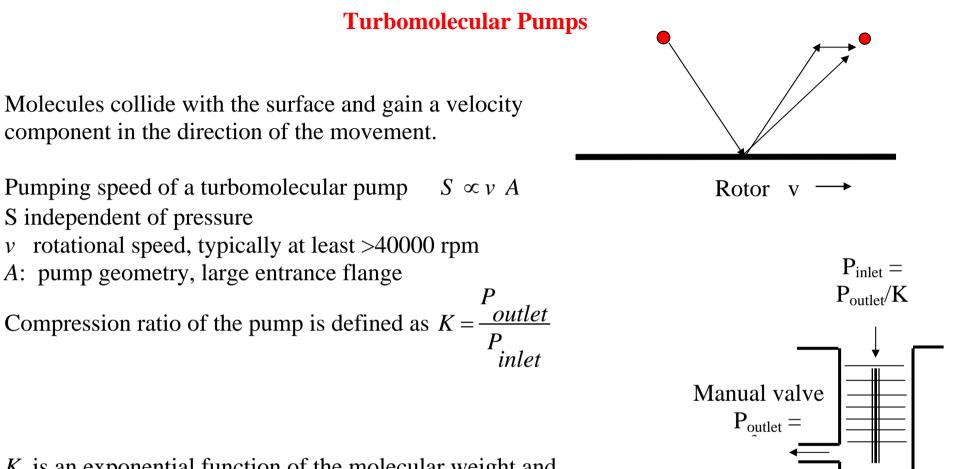
Typical pumping speed : 4 to  $\sim$  40 m<sup>3</sup>/h Adequate for systems with small volume

Filter for oil vapour is required.



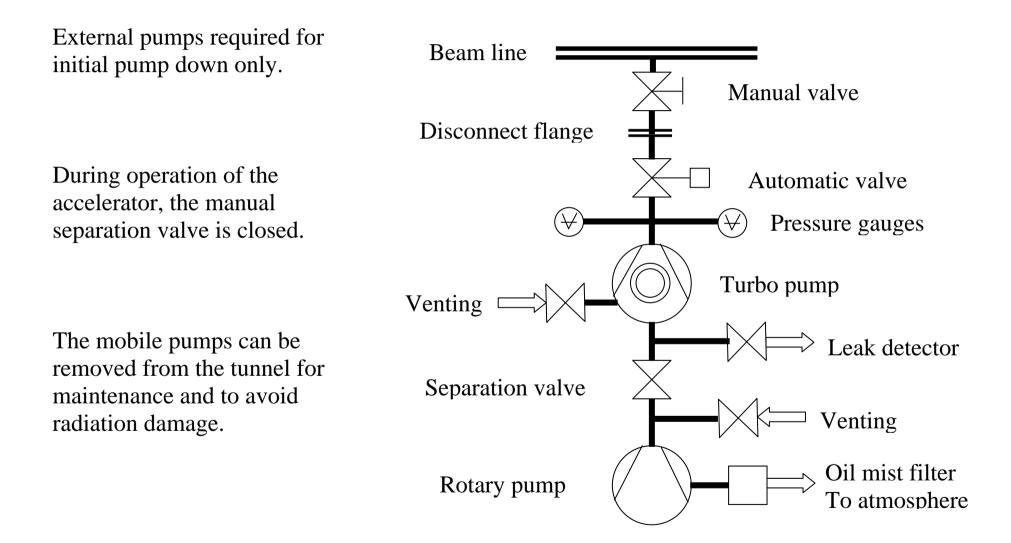
Dry pumps, without oil, are available but rather expensive!





*K* is an exponential function of the molecular weight and  $\Box$  of the rotational speed (10<sup>3</sup> for H<sub>2</sub> to 10<sup>9</sup> for N<sub>2</sub>) Hence the compression ratio is large for heavy molecules -> 'clean vacuum' without heavy hydrocarbon molecules. Oil contamination from primary pump can be avoided.

### Mobile pumping unit for LEP vacuum system



# **Sputter-Ion-Pump**

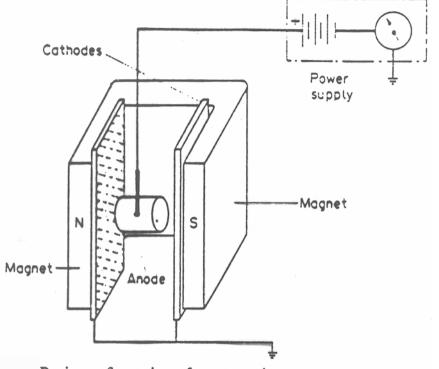
Configuration of a parallel electric and magnetic field produces a self-maintained discharge plasma. -> Penning configuration

Ionised residual gas molecules are accelerated towards the Ti cathode and 'trapped' and removed from the gas phase.

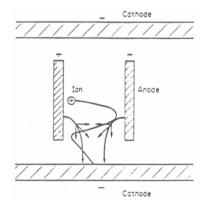
Sputtering of Ti from cathode produces a clean gettering film.

In a particle accelerator, the magnetic field is provided by bending magnets. --> <u>integrated</u>, <u>linear</u> <u>ion-pumps</u>.

To increase the pumping speed, arrays of cells are used



Basic configuration of a sputter-ion pump.



## **Pumping action**

Gettering -> chemisorption of active species  $H_2$ , CO,  $N_2$ ,  $O_2$ ,  $CO_2$ 

**Diffusion** of H<sub>2</sub> into the Ti- cathode (re-diffusion!)

Cracking of inert hydrocarbons into C, H, O which can be pumped (chemisorbed) separately

**Nobel gases**: energetic ions of He, Ne, A by implantation into the cathode: "ion burial" of energetic ions. -> Argon instability after pumping of air.

To increase the discharge intensity and thus the pumping speed it is desirable to increase the sputtering rate of the titanium cathode

→ Triode Sputter-Ion pump with grazing incidence of ions on a grid cathode

Note:

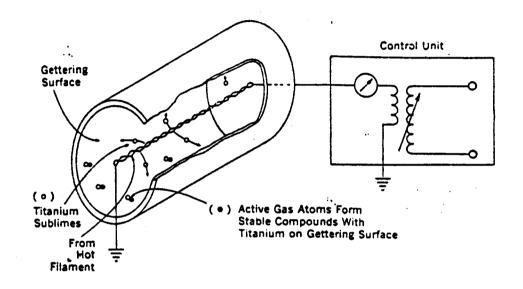
Molecules are not removed from the vacuum system. Important memory effect of previously pumped gas (Argon).

### **Evaporable getters:**

# **Titanium sublimation pump**

Deposition of a thin film of fresh Ti on the inner surface of the vacuum chamber

Filament temperature ~ 1300 deg C To increase the lifetime of the pump one uses pump holders with several filaments (3-6)



Depending on the amount of gas pumped, the film has to be regenerated - typically after 10<sup>-6</sup> Pa h The pumping speed increases with the surface of the pump and can be very substantial.

Note : only chemically active molecules can be pumped.

## Non-Evaporable Getters or Bulk getters (NEG) :

Getter material (e.g. Ti, Zr, V) produced in the form of an alloy e.g. with Al and used as a bulk material.

For LEP : metal ribbon coated with a thin layer of getter powder has been used.

Clean, active gettering surface is produced by heating under vacuum. Gas adsorbed on the surface diffuses into the bulk and a 'clean' surface can be obtained. Activation requires heating from 350 °C up to 700°C for one hour depending on the specific getter.

A new development consisting of a combination of evaporable getters and of bulk getters is under development at CERN -> sputter deposited getter films (few  $\mu$ m only) coated directly onto the inner surface of vacuum chambers. First use in insertion chambers (ESRF) and for LHC vacuum (CERN).

Note: Getters have a limited total pumping capacity and a memory effect of the gas previously pumped.Getters pump only chemically active gas i.e. nobel gases and hydrocarbons (methane, ...) are NOT pumped. Combination with ion pumps is required.

# Cryopumps

Adsorption of molecules at low temperature -> e.g. at liquid helium temperature

## A) Sorption

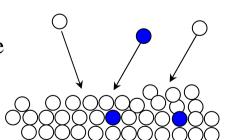
Adsorption of gas molecules with low surface coverage, to avoid the effect of the vapour pressure of the condensate. Increasing the effective surface area by a coating with a large specific surface area e.g. charcoal. -> Adsorption isotherms.

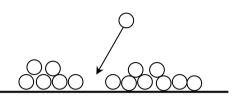
## B) Condensation

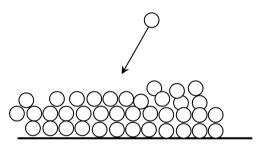
adsorption in multi-layers -> limitation due to the vapour pressure of the condensed gas.

# C) ' Cryo-trapping

Cryo-sorption of a gas e.g.  $H_2$  or He with a high vapour pressure in the presence of an easily condensable carrier gas e.g. Ar.







#### **Cryopumps in accelerators**

In combination with superconducting magnets or accelerating cavities, at little (or no) extra cost very effective integrated cryo-pumps can be obtained in an otherwise conductance limited vacuum systems.

Large freedom in the design of cryopumps : since the cold walls of the vacuum system act as pumps (LHC).

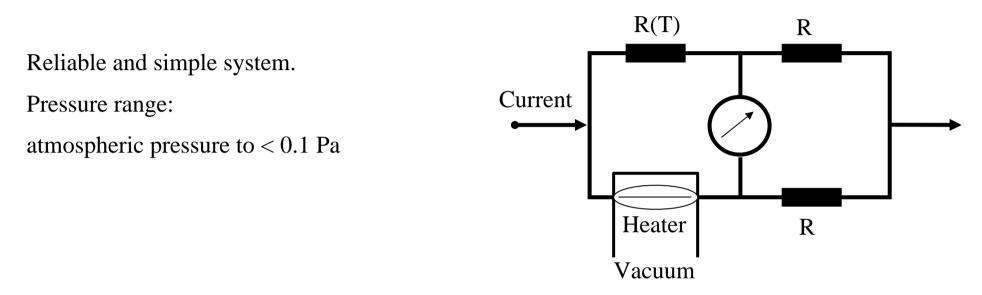
The limitations of cryopumps due to the exposure to environmental room temperature radiation and to the bombardment by beam induced energetic particles (photons, electrons, ions) must be taken into account.

Imposes ->  $LN_2$  cooled baffles and the LHC beam screen. This requirement arises not only for heat load reasons but mainly to avoid re-desorption of molecules.



## Pirani gauge, thermal conductivity gauge

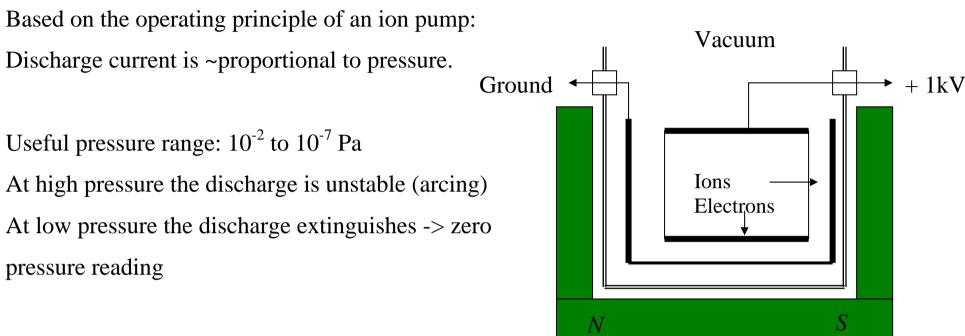
Uses the variation of the thermal conductivity with pressure



A resistor with a large temperature coefficient is mounted inside the vacuum and is heated to a constant temperature. The required heating current to maintain the bridge balanced is a measure of the pressure.

The electronic circuitry provides temperature compensation (R(T) and linearization of the pressure reading.

# **Cold Cathode Ionisation Gauge, Penning Gauge**



Leakage current in the cables and in the gauge can simulate a higher pressure.

Contamination of the gauge may change the calibration.

Extended operation at high pressure will 'contaminate' the gauge -> required demounting and cleaning of the gauge.

Improved version for low pressures on the market: Inverted magnetron gauge

### **Hot Filament ionization Gauge**

Operating principle :

Residual gas molecules are ionized by the electrons emitted from a hot filament.

Ions are collected by a "collector electrode".

This ion current is proportional to the gas density, n, and hence to the pressure, P.

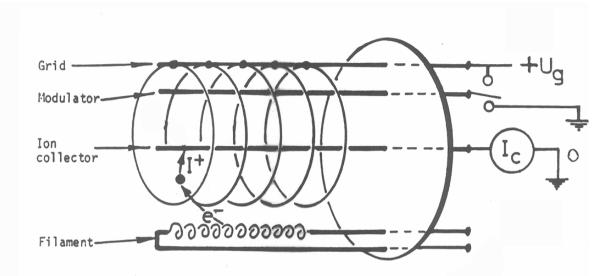
The ionization probability Pi

(number of ion–electron pairs produced per m and per Pa) depends on the type of molecule and on the kinetic energy of the electrons.

Ion collector current :

$$I^+ = Ie Pi L P$$

- Where : Ie emission current of the filament
  - L path length of the electrons
  - P pressure





#### **Gauge Sensitivity**

 $S = P_i L \quad [Pa^{-1}]$ 

Obtained by calibration with a known pressure  $(N_2)$ Nitrogen equivalent pressure  $N_2$ .

- ➔ To measure a pressure for another gas, the relative gauge sensitivity for this particular gas with respect to nitrogen must be known.
- →  $S_i/S_{N2}$  must be known for different gas species. For H<sub>2</sub>, one finds typically  $S_{H2}/S_{N2} \sim 0.38$



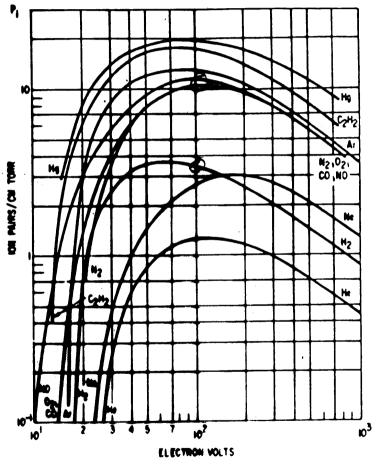
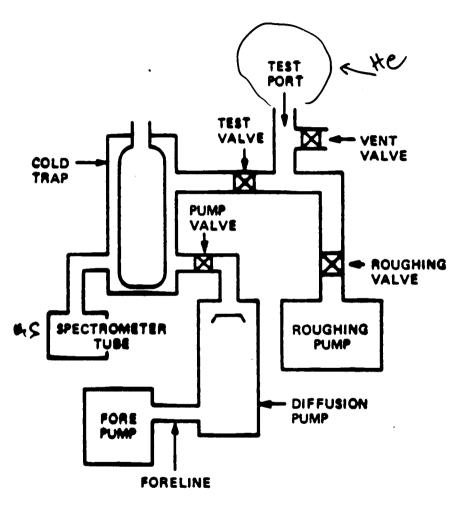


FIGURE 2. Probability of ionization as a function of electron energy for various gases at 1 Torr and  $0^{\circ}C^{\circ}$ .

#### Helium leak detector

Mass spectrometer tuned to Helium, which is commonly used as the 'tracer gas'.

Purpose of the  $LN_2$  filled cold trap is to remove oil vapours of the diffusion pump as well as water vapour from the spectrometer cell



#### Leaks and leak detection

Common leaks to atmospheric pressure:

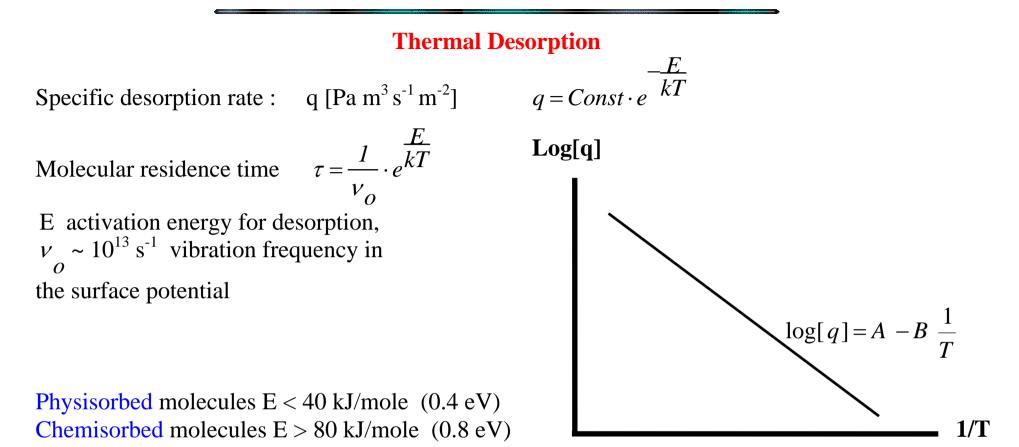
Gaskets Porosities in the materials Cracks and porosities in welds

Virtual leaks: are not found by a conventional leak check Porosities, a dead volume enclosed inside the system

Example of a virtual leak: The volume enclosed by a bolt in a threaded hole.

Solution: bolts have to be drilled with a central hole or a separate hole must be drilled to pump the dead volume.

In a large vacuum system, leak checks of all sub-components are mandatory. A global leak check after complete assembly should only concern those joints, which have been made during the final installation phase in the accelerator.



Bakeout between  $150 - 300^{\circ}$ C : reduced residence time. Reduction for H<sub>2</sub>O, CO, CO<sub>2</sub> (by factors of  $10^{-2}$  to  $10^{-4}$ ) At higher temperature > 400-500°C -> cracking of hydrocarbon molecules (C-H) Note: Strongly reduced thermal desorption at cryogenic temperatures

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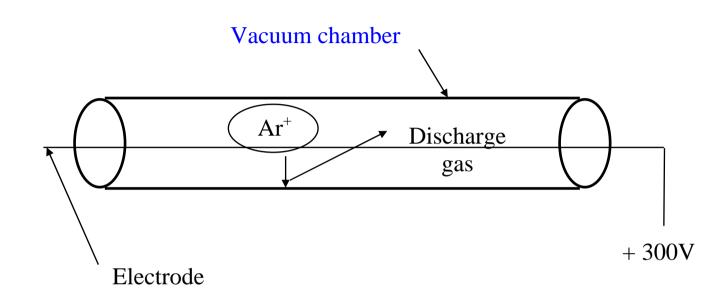
### **Chemical solvent pre-cleaning procedure**

Removal of gross contamination and machining oils using the appropriate solvents Perchloroethylene ( $C_2Cl_4$ ) vapour degreasing at 121°C (no longer applicable) Ultrasonic cleaning in alkaline detergent (pH = 11) Rinsing in cold demineralised water  $< 5 \ \mu S \ cm^{-1}$ Drying in a hot air oven at 150°C Wrapping in clean Al-foil or paper 6 6 Large number of methods exist: Cleaning method will depend on the material i.e. stainless steel, aluminium, copper 5 All subsequent handling with **clean gloves**. Contamination by any residues in the air must be $^3$ avoided. No car exhaust gases, No smoking!! 2

3

Legend: 1) hot detergent, 2) ultrasonic generator, 3) heaters, 4) hot solvent bath, 5) solvent vapour zone, 6) cooling zone.

## **Glow-Discharge Cleaning**



Cleaning of the surface by energetic ion bombardment (Usually Argon or some other inert gas) Dose approx.  $10^{18} - 10^{19}$  ions/cm<sup>2</sup>

Argon pressure between  $10^{-1} - 10^{-2}$  Pa for optimum conditions

Desorption of chemisorbed, strongly bound molecules corresponding to a high activation energy.

Effective cleaning by removing the top layer of the surface by sputtering. -> Tokamak vacuum systems

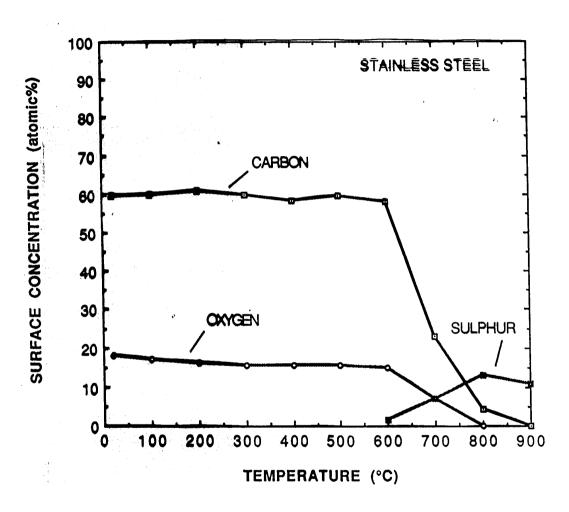
## Vacuum firing at high temperature

High temperature baking in a vacuum oven at ~950 deg C

Cracking of hydrocarbons and organic compounds.

Reduction of the surface oxide layer.

After the high temperature treatment, cool down in a clean gas to generate a controlled oxide layer



## **Thermal outgassing rates of some materials**

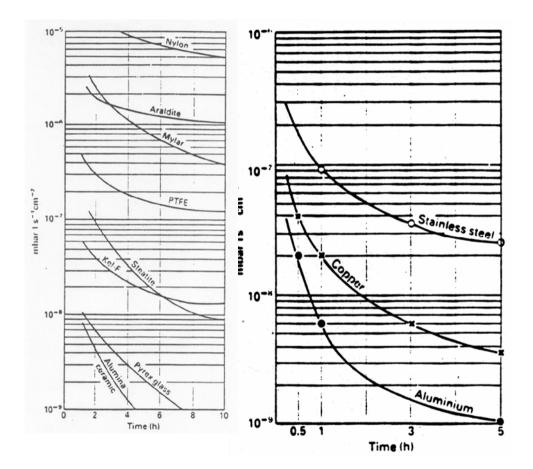
#### **Comparison of organic materials and of metals**

Unbaked samples (usually H<sub>2</sub>O dominates)

Baked samples (24 hours at 150°C to 300 °C)

Typical values after 50 hours of pumping : (units : Torr  $1 \text{ s}^{-1} \text{ cm}^{-2}$ )

Gas	Al, Stainless steel
$H_2$	5 10 <sup>-13</sup>
$CH_4$	5 10 <sup>-15</sup>
CO	1 10 <sup>-14</sup>
$CO_2$	1 10 <sup>-14</sup>



# Criteria influencing the choice of materials

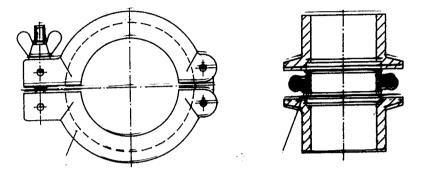
Low outgassing rate Low vapour pressure Temperature resistant -> bakeout Thermal and electrical conductivity -> beam interaction Corrosion resistance -> leaks Low induced radioactivity -> handling High mechanical strength -> 1dN/cm<sup>2</sup> external pressure! Machining, welding, mounting/demounting requirement Low cost

## **Common choices:**

Stainless steel Aluminium Copper Ceramics for electric insulation Low porosity -> leaks Brazing to metal -> leaks For particular applications Organic materials (e.g. as composite materials (carbon-fiber & epoxy), polymers to be used in small quantities

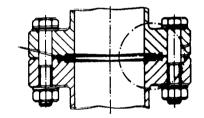
# Flanges and gaskets for primary vacuum and for high vacuum applications

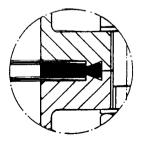
Flange with clamp and elastomer seal for high vacuum systems

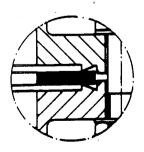


'ConFlat' flange for uhv systems Copper gasket for 'all metal' vacuum system









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# Reminder

# Special CAS for Vacuum Technology is on the program

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