

Vacuum technology UHV systems

Csonka István
Frigyes Dávid

UHV applications

Main areas:

- Surface preparation/modification [Molecular Beam Epitaxy, UHV-CVD] and analysis [UV/X-ray Photoelectron Spectroscopy UPS, XPS, Auger electron spectroscopy AES, Low Energy Electron Diffraction LEED, Scanning Tunneling Microscopy STM] (surface coating needs at least 1000 sec).
- Accelerators, particle beams (the particle should not react fast).
- FT-ICR MS [Fourier Transform Ion-Cyclotron Resonance Mass Spectrometry] (Coherent motion of the ions decays on collisions)

UHV systems

Program today:

- Set-ups, characteristics and operation of UHV systems
- Outgassing

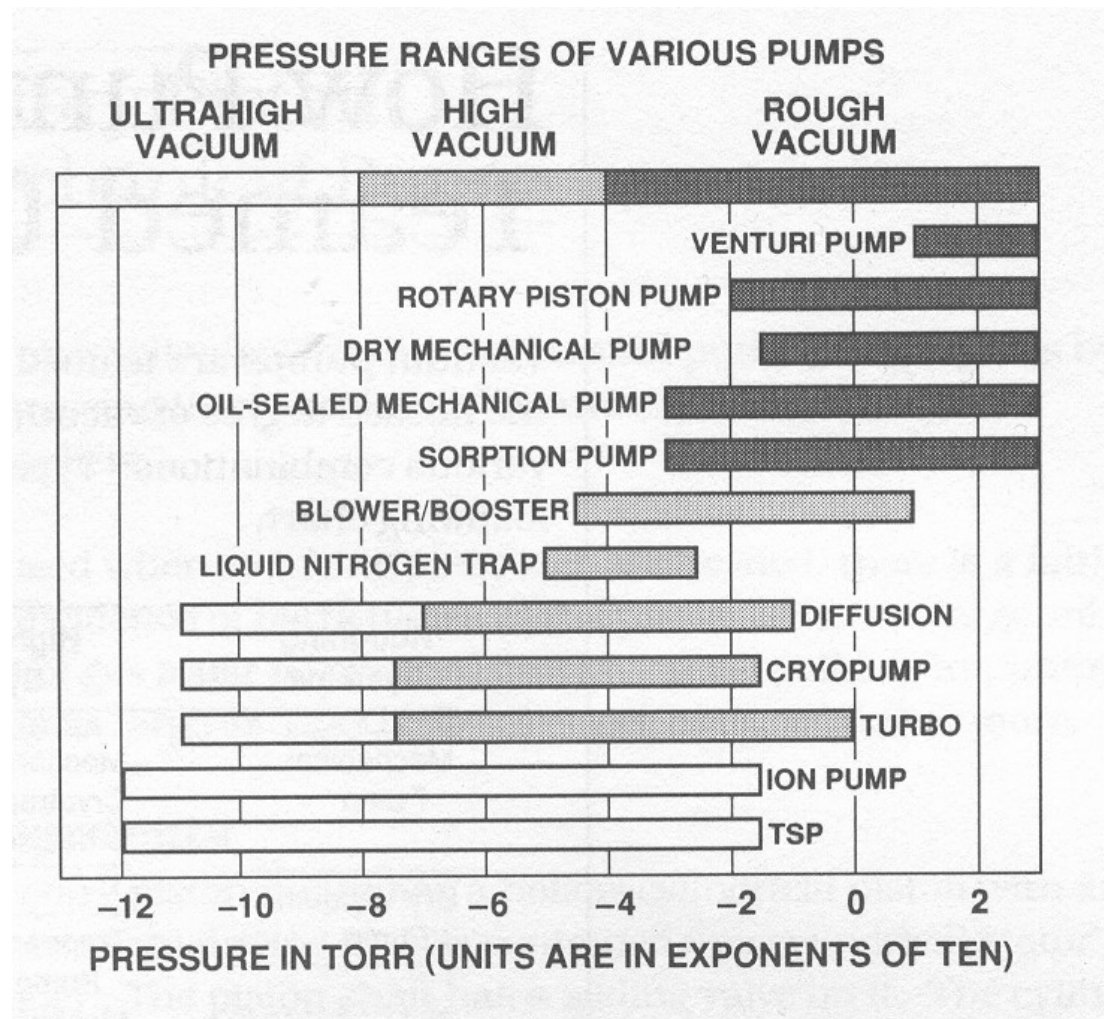
Special needs

- No oil (surfaces!)
- No vibrations (STM, AFM)

UHV systems

- HV pumps (diffusion, turbo, cryo) can reach the UHV region. Limiting the outgassing and baking out ($>200^{\circ}\text{C}$) is needed. Advantages: high pumping speed and throughput; Disadvantages: contaminations (oil, $\text{H}_2\dots$)
- UHV pumps are effective in the lower end of HV range. They can work at even in XHV, and they cause much less contamination. Their disadvantage is the lower pumping speed and throughput. Mostly trapping pumps, thus they don't need roughing during operation.

UHV systems

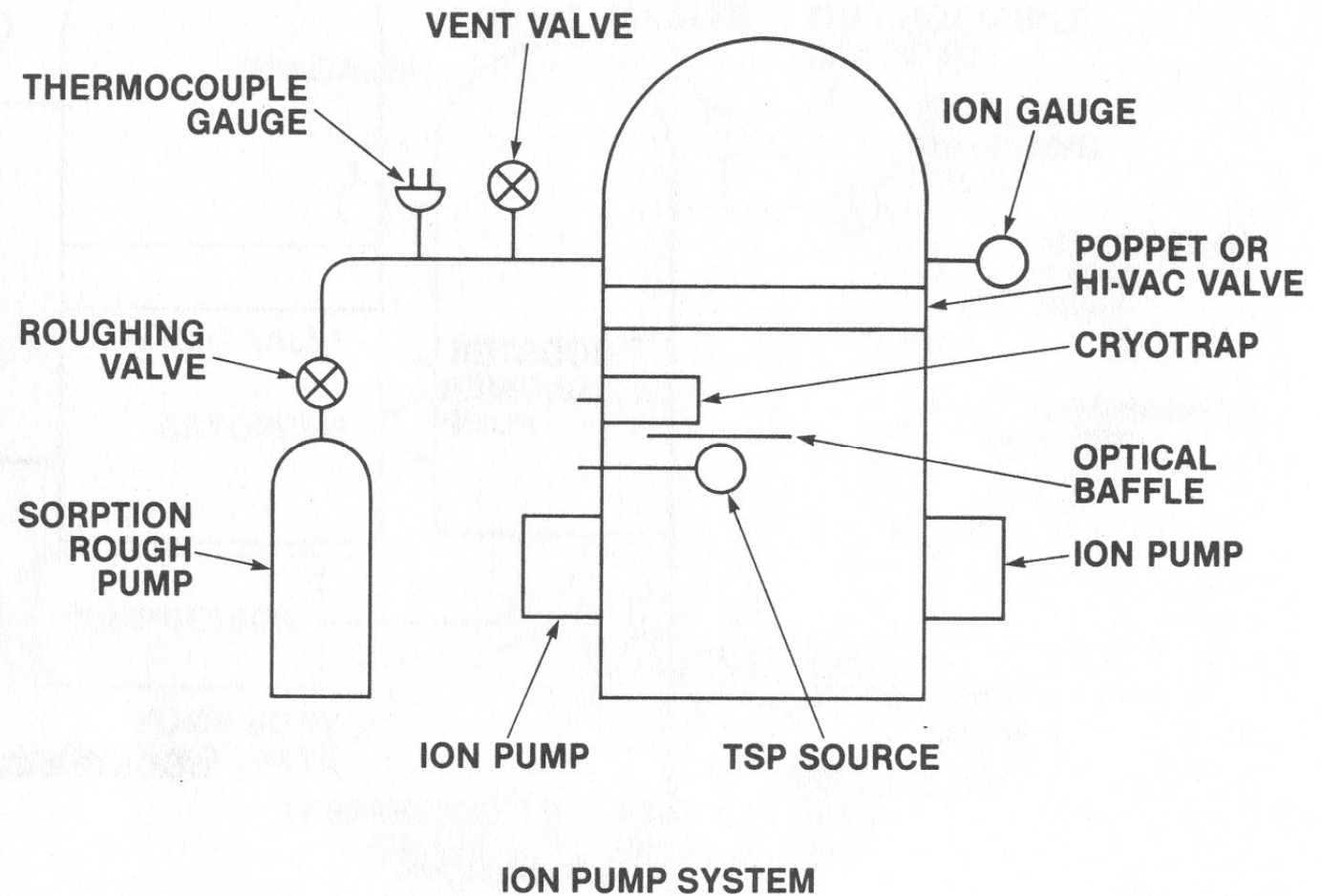


UHV systems

- Low-volume systems with low gas load, rare venting: sorption pump + UHV pump(s) can be enough.
- If the gas load is higher, roughing + HV pump can be used for reaching the HV range (diaphragm/sorption + turbo). After that the UHV pumps are started
- Different pumps can work together (pumping speeds vary for different gases): ion pump for nonreactive gases (noble gases and methane) TSP/NEG for reactive gases, cryo for 'everything' – efficacy increases with increasing molar mass

UHV systems

- Usual system for low gas load



UHV systems

Starting up (from atmosphere):

- Fill up sorption pump with $1N_2$
- Pirani on
- Roughing (meanwhile regenerating the NEG, $350^\circ C$, if any)
- Fill up $1N_2$ trap
- TSP-outgassing (O_2 , H_2O desorption).
- When below 30 mbar, start ion pump
- Roughing valve close
- Start ion gauge
- TSP to standby (mild heating to minimize adsorption)
- Bake out (12 hours at $250^\circ C$, 8 hours cool down)

(Continued)

UHV systems

- Outgassing TSP again
- Fill IN2 regularly
- Start TSP regularly (5min/day can be enough; common practice to control it from gauge – pressure rises while starting)

Variants:

- If turbo is present, wait for HV pressure to isolate turbo and start UHV pump(s)
- NEG should be replaced after approx. 30 regeneration cycles (it can take years – remember, UHV systems are not to stop/start daily)

Generally: UHV can be achieved and measured relatively easily using relatively inexpensive equipments due to widespread applications

Sample handling

- Generally solid samples
- Not practical
 - To vent and stop the vacuum system at each sample (there should be space for a series of samples inside the instrument)
 - To apply differentially pumped equipment for placing in the sample
 - To place the sample directly into the instrument chamber
- Usual practice: the sample is placed into a chamber relatively far from the analyzer chamber, where it is „roughed” and cleaned (treated). Then the sample transferred to the analyzer chamber by using special manipulators. (not to forget that it takes time!)
- Usually there is more chambers (sample placing in, annealing, atom bombardment, LEED, XPS, STM...), or transfer chamber with built-in vacuum system
- There should be as many windows as possible to make the process visible

Sample manipulations

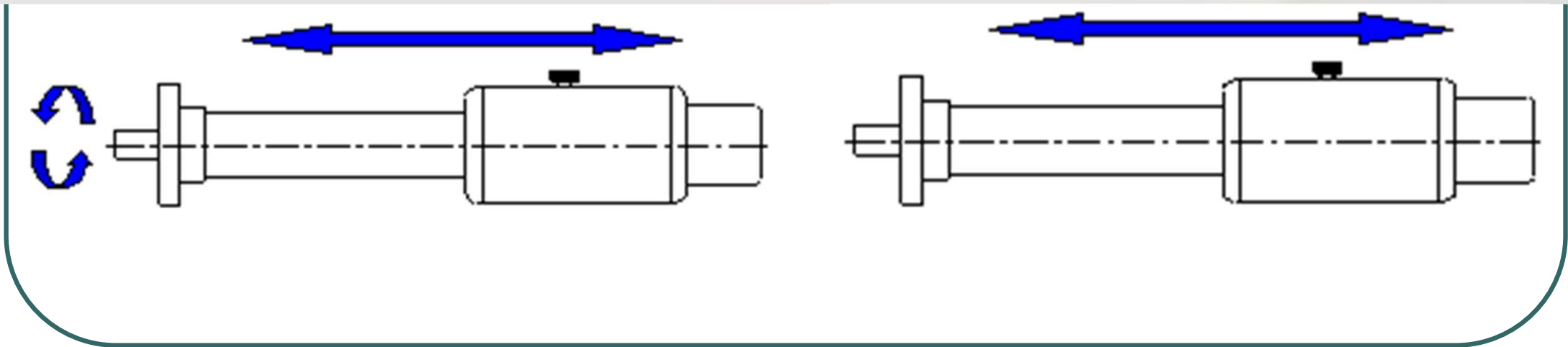
- There's no surface air layer! Metallic surfaces cannot slide easily, they can stick together
- Every movements should be considered in advance (mea culpa...)

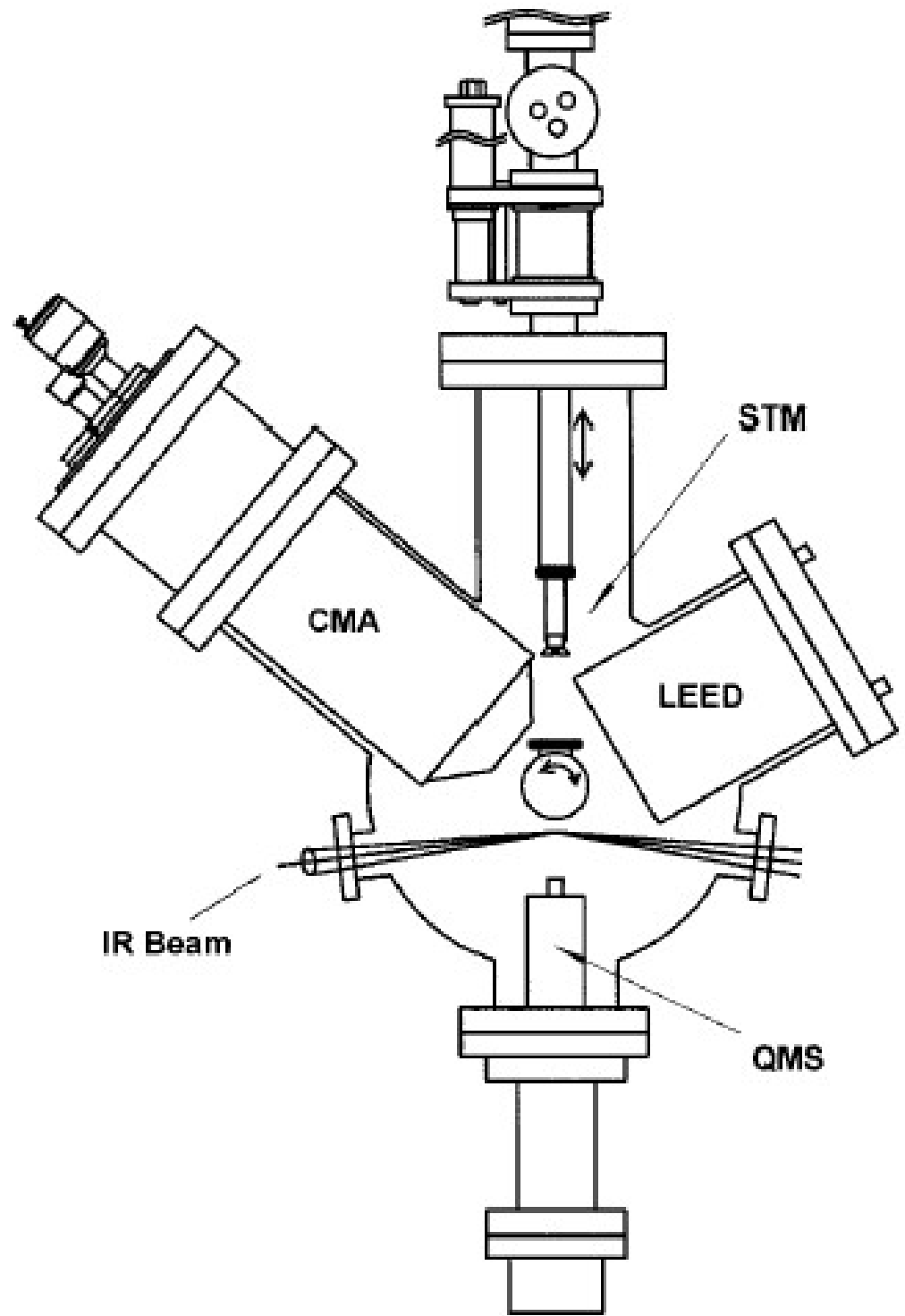














XHV systems

- $<10^{-12}$ mbar (44 days till a monolayer can set up).
- Interstellar space 10^{-19} mbar, 1.2 million years till a monolayer can set up
- Accelerators, storage rings, space technology
- It is relatively easy to reach: it was probably achieved in 30s by cooling a sealed glass bulb in liquid hydrogen bath; measurement is much less trivial – recall X-ray limit
- In principle well-trapped diffusion pump or well-prepared (rotor outgassing!) turbo pump systems can be applicable but trapping pumps are more suitable
- Special solutions: e.g. accelerator using superconducting magnet: the whole wall can be a pump (anodized aluminum + liquid He)

XHV systems

Measurement of pressure is critical. Usual solution: hot cathode gauge + elimination/correction of main errors

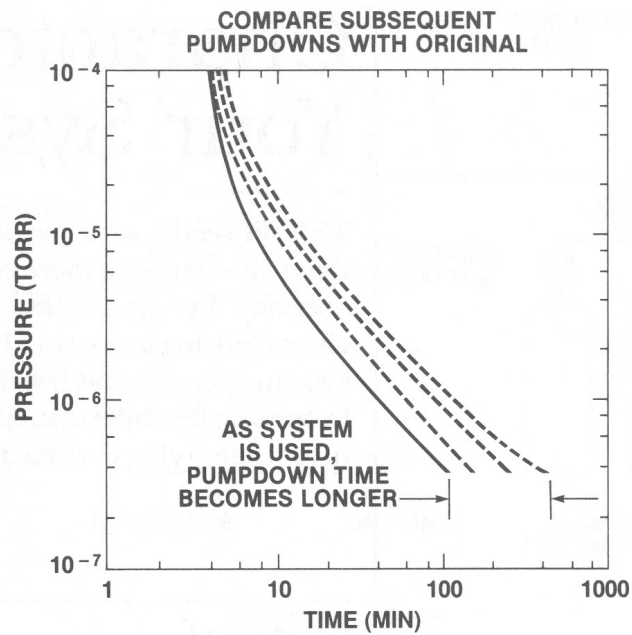
- X-ray limit (extra thin collector Bayard-Alpert gauge)
- Electron stimulated desorption (ESD): impacting electrons can result in desorption of various particles. They can be distinguished from gas-phase ions by energy.
- Hot cathode: 1) outgassing by heating 2) evaporation of cathode material 3) chemical reactions close to cathode
- Low energy X-ray stimulated desorption

The measured minimum is about one order lower than the X-ray limit. Hot cathode magnetron (Lafferty): 10^{-16} mbar.

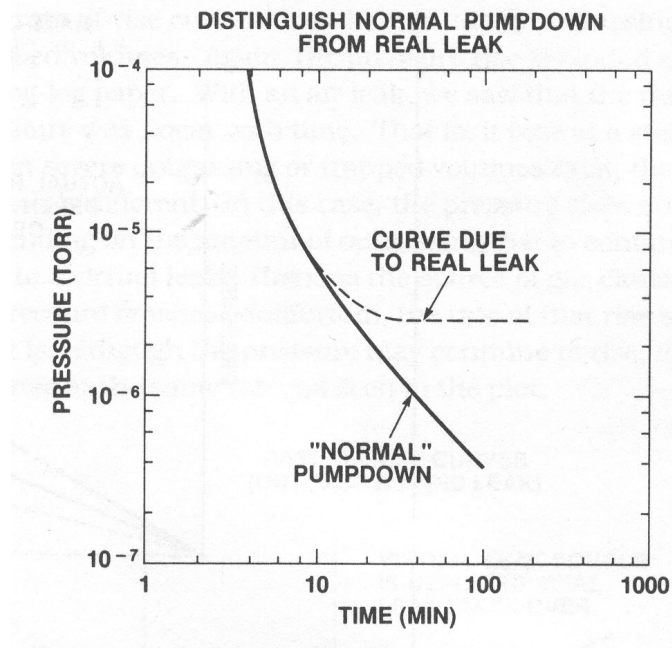
Outgassing

- In HV background gas originates from outside the system.
 - Operation (e.g. gas-phase measurements)
 - Leak, virtual leak
- In UHV (and XHV) systems, direct gas inlet is much smaller (e.g. metallic gaskets instead of Viton). Here the background comes from outgassing (it can limit the pressure reached and measured)
- Outgassing: particles in the gas phase from the surfaces or bulk of the elements of the instrument
- Background
 - Rough vacuum: N_2 , O_2 , Ar (air)
 - High vacuum: H_2O
 - UHV: H_2

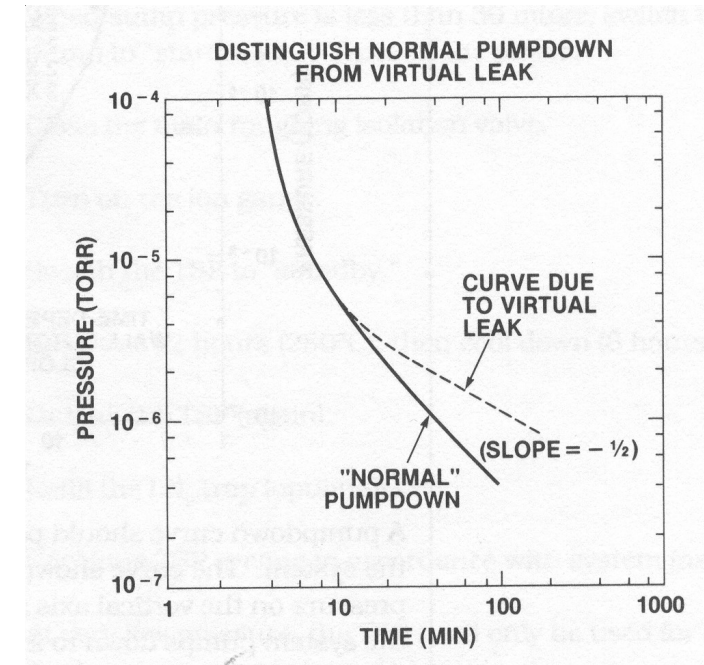
Outgassing



Normal pumpdown



Leak



Virtual leak

Outgassing

Outgassing is more than simple evaporation from walls:

- Desorption
- ESD (electron stimulated desorption)
- Other stimulated desorptions
- Diffusion and desorption of gases dissolved in the structural material
- Gas diffusion through walls
- Evaporation of structural material

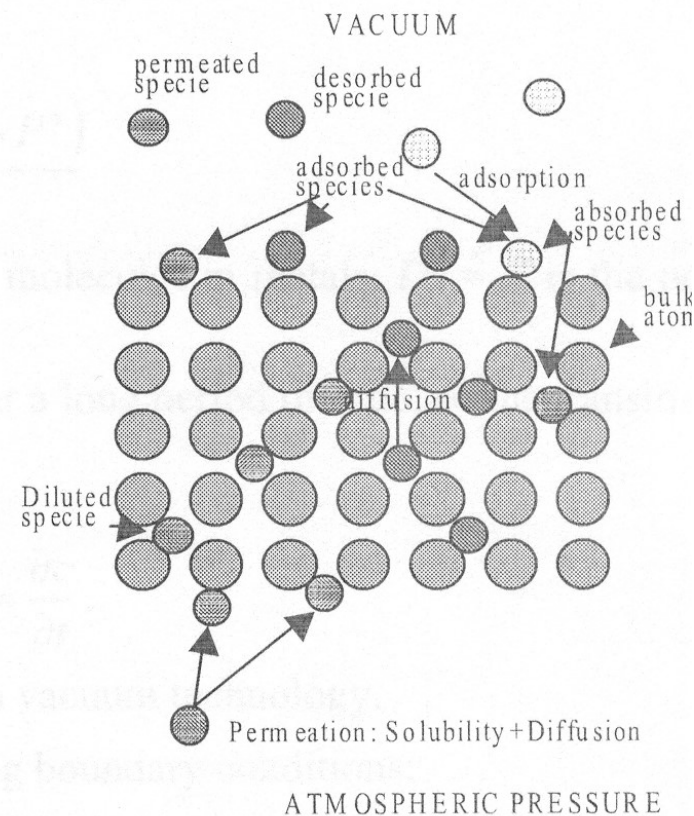


Fig. 1 Surface and bulk phenomena in vacuum

Outgassing

- Unit: $\left[\frac{pV}{At}\right] = \frac{Pa \cdot m^3}{m^2 s} = \frac{Pa \cdot m}{s} = \frac{1000 \text{ mbar} \cdot l}{s \cdot cm^2}$
- Outgassing can be decreased by suitable material selection, pre-treatment and operation

Approximate outgassing rate K_1 for several vacuum materials, after one hour in vacuum at room temperature.

Material	K_1 (mbar l s ⁻¹ cm ⁻²)
Aluminium (fresh)	9×10^{-9}
Aluminium (20 h at 100 °C)	5×10^{-14}
Stainless steel (304)	2×10^{-8}
Stainless steel (304, electropolished)	6×10^{-9}
Stainless steel (304, mechanically polished)	2×10^{-9}
Stainless steel (304, electropolished, 30 h at 250 °C)	4×10^{-12}
Perbunan	5×10^{-6}
Pyrex	1×10^{-8}
Teflon	8×10^{-8}
Viton A (fresh)	2×10^{-6}

Outgassing

Table 5
Some measured outgassing rates at room temperature suitable for XHV

Material	Surface treatment	Outgassing rate (Pa m s ⁻¹)	Reference
Stainless steel	Glass bead blasted + vacuum fired at 550°C /3 days + baked at 250°C/24 h.	1.6×10^{-13} [H ₂]	[59]
Stainless steel (304L)	Air fired at 400°C/38h + baked at 150°C/7 days.	1×10^{-12} [H ₂]	[54]
	Air fired 390°C/100 h + baked at 150°C.	5×10^{-12} [H ₂]	[53]
Stainless steel	Vacuum fired at 960°C/25 h +baked at 180°C /6 h.	1×10^{-12} [H ₂]	[60]
Aluminium	Vacuum fired at 960°C/25 h +baked at 180°C /6 .	9×10^{-13} [N ₂]	[58]
Copper (OFHC)	Vacuum fired at 550°C/3 days + baked 250°C/24 h	6×10^{-14} [H ₂]	[59]
Copper (OFHC)	Baked at 525°C	2×10^{-13} [N ₂]	[58]
TiN on stainless steel.	Vacuum fired at 430°C/100 h and 500°C/ 100 h.	1×10^{-13} [H ₂]	[61]
Aluminosilicate glass	Baked at 500°C/18 h + 600°C/2 h + 700°C/2 h/500°C/10 h.	2.5×10^{-13} [N ₂]	[62]

Outgassing

Adsorption, desorption

- Physical (<30 kJ/mol), desorb fast in vacuum, no trouble
- Chemical (>100 kJ/mol), there is no desorption, usually no trouble (except for high temperature, stimulated desorptions)
- Water is between the two extremes
- H₂: supplied from bulk

Adsorbed gases and partly dissolved hydrogen can be removed by heating

- Usually >200°C while pumping (bake out) but in special cases (equipment) 450°C - 850 °C (vacuum firing)
- Water and dissolved hydrogen can be removed almost completely
- Firing can be harmful for the structural material (structural stability, hole forming, magnetizing)
- All the materials should be compatible with high temperature (Viton 140°C!)
- There should be no cold spot, temperature should be uniform
- Surface area should be decreased (polishing, coating, TiN, metal oxide)

Bakeout

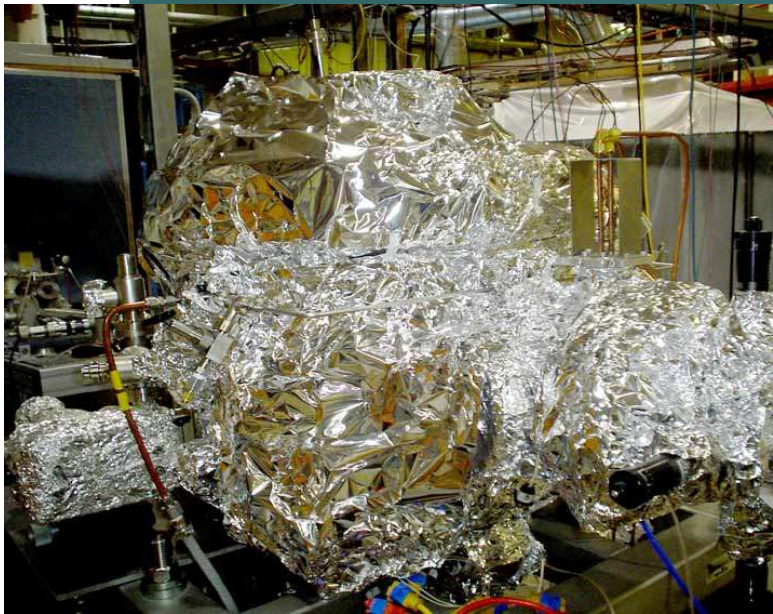


Fig 1: MBE mini bake



MBE bake-out



Bakeout



Outgassing

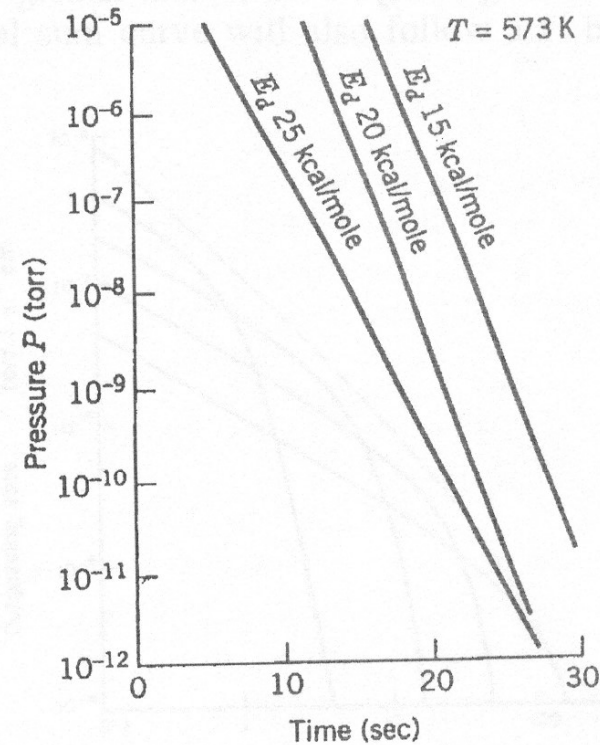
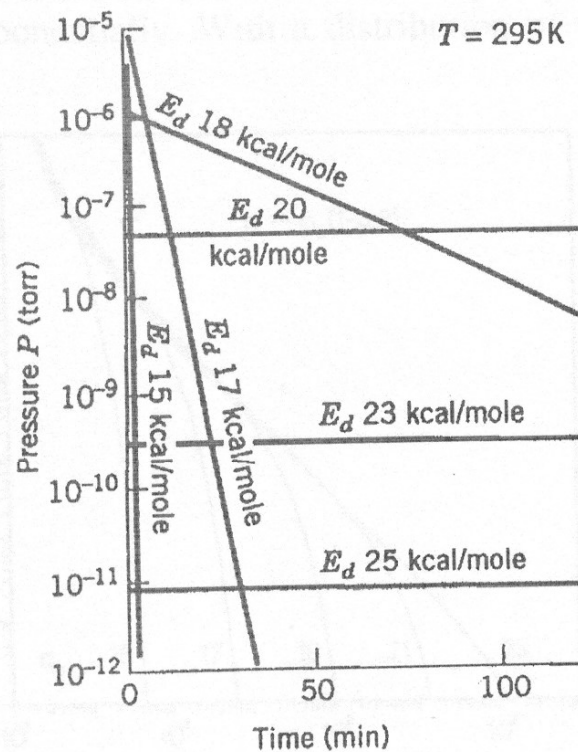


Fig. 3 Calculated pressure vs. time curves for the pumpdown of vacuum chambers having adsorbed layers with the adsorption energies indicated. From Ref. [3]. Copyright 1961 American Vacuum Society. Reprinted by permission of John Wiley & Sons, Inc

Outgassing

After bakeout

- Stainless steel always contains H_2 .
 - It is absorbed during molding (even in vacuum molding)
 - Raw metal is usually treated by acid
- Presence of H_2 can result in presence of H_2O and CH_4

Diffusion, permeation

- Metals.
 - Noble gases do not permeate.
 - H₂ through Pd, always present in iron; OFHC (oxygen-free high thermal conductivity) – not important
 - O₂ through Ag (ezüst, illetve ezüstözött réz tömítés) Permeation is proportional to square root of the pressure
- Polymers – Much more important than metals: all gases permeate all polymers; proportional to square root of the pressure
 - Water can go through silicon,
 - He through Viton
- Glasses: H₂, He, Ne, Ar, O₂.
- Standard inlet can be fabricated from thin-wall glass (gauge calibration)

Induced desorption

- At elevated temperature: e.g. the rotor of a turbo pump, surroundings of a hot-cathode gauge
- Electron bombardment
- X-ray radiation
- Other particles
- The pressure reached did not improve in the last 3 decades. Is it measurement problem?

Evaporation, miscellaneous

- One should always remember that all material has vapour pressure (Zn, Cd) – temperature, pressure
- Some materials should be avoided:
 - Porous material (e.g. pyrolytic graphite instead of sintered carbon)
 - Oil, esp. Silicon oil
 - (even) vacuum grease
 - Fingerprint (UHV: cotton gloves, HV: thorough hand wash with cold water)