Advanced Micro and Nanofabrication Technologies

Engineering Physics – Ingegneria Fisica - Cod. 055559

a.a. 2024/2025

Matteo Cantoni
Dipartimento di Fisica
Politecnico di Milano and Polifab
Ed. 30 - Via G. Colombo 81 – Milano
https://cantoni.faculty.polimi.it/

Lecture 2
Vacuum

prof. Matteo Cantoni

matteo.cantoni@polimi.it

POLITECNICO MILANO 1863
DIPARTIMENTO DI FISICA

polifab POLITECNICO DI MILANO

eFES

Outline of the course

- **Where** we will grow? **Vacuum** → **Vacuum technology: pressure, gas transport, vacuum systems**

- **On what** we will grow? **Surfaces**

- **Which kind** of films we will grow? **Epitaxy**

- **How** we will grow?
  - **Physical techniques**
    - **Evaporation**
    - **Sputtering**
  - **Chemical techniques**

- **How** we will check the growth? **Characterization**

Reference for this lecture: M. Ohring, Materials Science of Thin films, Academic Press, Chapter 2

«Natura abhorret a vacuo»

What is vacuum?

Pressure (P) < 1 atm (1.013 bar)

- 1 bar to 30 mbar $\Rightarrow$ Low Vacuum
- 30 mbar to 10^{-3} mbar $\Rightarrow$ Medium Vacuum
- 10^{-3} mbar to 10^{-9} mbar $\Rightarrow$ High Vacuum (HV)
- 10^{-9} mbar to 10^{-12} mbar $\Rightarrow$ Ultra-High Vacuum (UHV)
- $<10^{-12}$ mbar $\Rightarrow$ Extremely Ultra-High Vacuum

(the ranges are not unequivocally defined)

The objective of growth is to deposit layers of materials *atom-by-atom* or *molecule-by-molecule* on a solid surface, so why we need vacuum?

- **To reduce particle density of undesirable atoms and molecules (*contaminants*)**
- To reduce particle density **to increase the mean free path between collision**, thus **preserving the energy and path of the particles** to be deposited (e.g., by evaporation or sputtering)
- To provide a low-pressure environment **to sustain plasma discharges** (e.g., for sputtering)
- To provide a way to control gas and vapor composition (e.g., for deposition under reactive gases)
- To provide a way for mass flow control into the processing chamber
- To **preserve hot filaments from burning** (e.g., in incandescent light bulbs)

Vacuum system

Vacuum is basically needed for

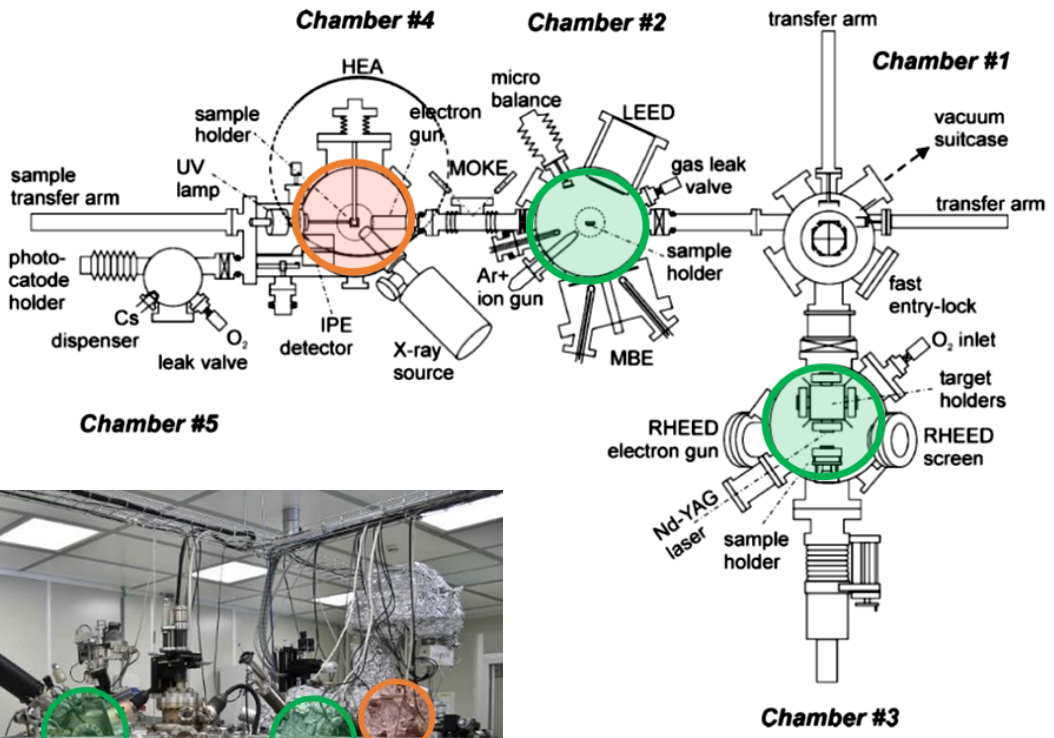
- **deposition**
- characterization

A vacuum system is a **chamber** (or a series of interconnected chambers) where **vacuum** is achieved and preserved by a **pumping** system

<u>Technological issues:</u>

- Different vacuum **levels** (UHV, HV, low vacuum,…)
- Different dynamic/static **regimes** (fast venting/pumping, vacuum preservation)

Example: deposition of epitaxial films by Molecular Beam Epitaxy → UHV (10^{-9}-10^{-10} mbar) is needed to avoid (or reduce) incorporation of contaminants (C, O_2, CO, H_2O, …) in the film under growth → *UHV level, static regime*



[Applied Surface Science 252, 1754-1764 (2005)]

Example: fast pumping of a load-lock chamber after sample insertion/extraction → *HV level, dynamic regime*

Kinetic theory of gases

Hp.:

1) gas in a **closed** system with uniform *P*, *T*, ρ ; no net gas flow

2) random motion – *f(T)*

3) elastic collisions between particles and with walls – *f(P) or f(ρ)*

4) no forces between particles (*perfect* or *ideal gas*)

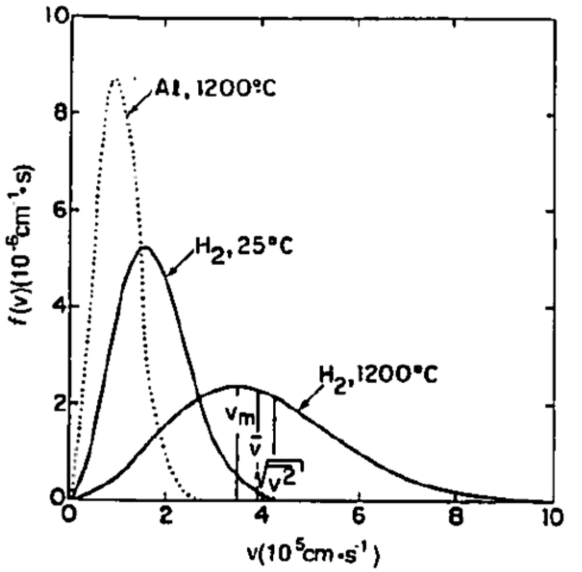
Particles can be considered as elastic spheres much smaller than their distances, that continuously exchange kinetic energy via elastic collisions ➡ there is a **steady-state distribution of molecular velocities** given by the *Maxwell-Boltzmann formula*, where $f(v)$ is the fractional number of particles per unit volume (*n*) with velocity in the range *v* to *v+dv:*

$$f(v) = \frac{1}{n}\frac{dn}{dv} = \frac{4}{\sqrt{\pi}}\left(\frac{M}{2RT}\right)^{3/2} v^2 exp\left(-\frac{Mv^2}{2RT}\right) \quad (M = \text{molecular weight, } R = \text{gas constant})$$

- $f(v)$ depends on molecular mass (*M*) and absolute temperature (*T*)
- **Average** velocity: $\bar{v} = \int_0^\infty v\, f(v)dv / \int_0^\infty f(v)dv = \sqrt{\frac{8RT}{\pi M}}$
- $\overline{v^2} = \int_0^\infty v^2\, f(v)dv / \int_0^\infty f(v)dv = \frac{3RT}{M} \Rightarrow$ **Mean square** velocity: $\left(\overline{v^2}\right)^{1/2} = \sqrt{\frac{3RT}{M}}$

$$\propto \sqrt{\frac{T}{M}}$$

- **Average kinetic energy**: $\bar{K} = \frac{1}{2}M\overline{v^2} = \frac{3}{2}RT$ depends on *T* only



Velocity distributions for Al vapor and H_2 gas. (from M. Ohring, Materials Science of Thin films, Academic Press, Chapter 2)

Pressure (1)

P is the **fundamental parameter** in vacuum technology

- What does it represent?

- Momentum transfer from the gas particles to the container walls gives rise to forces that sustain the pressure in the system

- Ideal gas law: $P = \frac{n_{mol}RT}{V} = \frac{n}{N_A}RT$
- Kinetic theory of gases: $\frac{1}{2}M\overline{v^2} = \frac{3}{2}RT$

$$\Rightarrow \quad P = \frac{M\overline{v^2}}{3}$$

n = number of particles per unit volume
N_a = Avogadro's number
n/N_A = number of moles per unit volume
$n/N_A \times V = n_{mol}$ = number of moles

Note: *P* is force per unit area $\Rightarrow$ 1 Pa = 1 N/m^2 (SI unit)

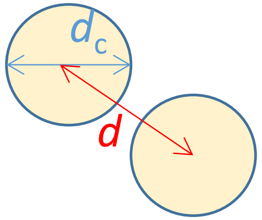
Other useful units: 1 bar = 10^5 Pa = 750 torr
1 atm = 1.013 bar = $1.013 \cdot 10^5$ Pa = 760 torr
1 torr = 1 mm Hg = 133.3 Pa = 1.33 mbar ⇒ 1 torr ~ 1 mbar (as zero-th order approximation….)

Pressure (2)

Hp. of kinetic theory of gases: 3) elastic collisions between particles and with walls – *f(P) or f(ρ)*

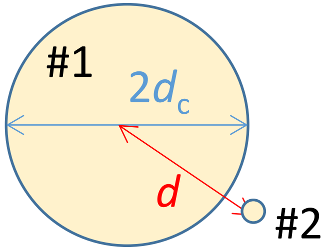
- Which is the mean distance travelled by particles between collisions (**mean-free path, λ_{mfp}**)?

- Assume that d_c is the collision diameter of the particle.
- Two particles collide each time their centers are spaced by $d<d_c$.

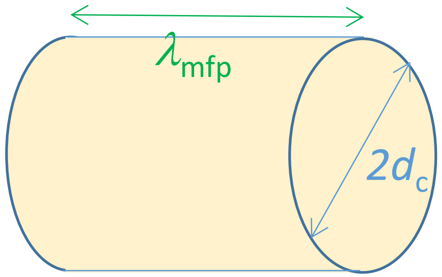


- Instead of 2 particles of diameter d_c, consider
  i. particle #1 with diameter 2 d_c
  ii. point (zero diameter) particle #2

  $\Rightarrow$ the condition above (collision for $d<d_c$) continues to be valid



- Particle #1 has a target area $\pi d_c^2 \Rightarrow$ in travelling a distance λ_{mfp} it sweeps a volume $\pi d_c^2 \lambda_{mfp}$.



- Consider all gas particles, apart from #1, to be reduced to point particles; *n* is the number of particles per unit volume

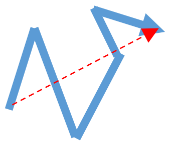
  $\Rightarrow$ one collision occurs if at least a particle is contained in the volume ➡ $n\pi d_c^2 \lambda_{mfp}=1$ ➡ $\lambda_{mfp} = \frac{1}{n\pi d_C^2}$

Pressure (3)

- $P=nRT/N_A \Rightarrow n=PN_A/RT \Rightarrow \lambda_{mfp} = \dfrac{1}{n\pi d_C^2} = \dfrac{RT}{\pi d_C^2 P N_A} = \dfrac{k_B T}{\pi d_C^2 P}$ ➡ It depends on the ratio $T\,/\,P$

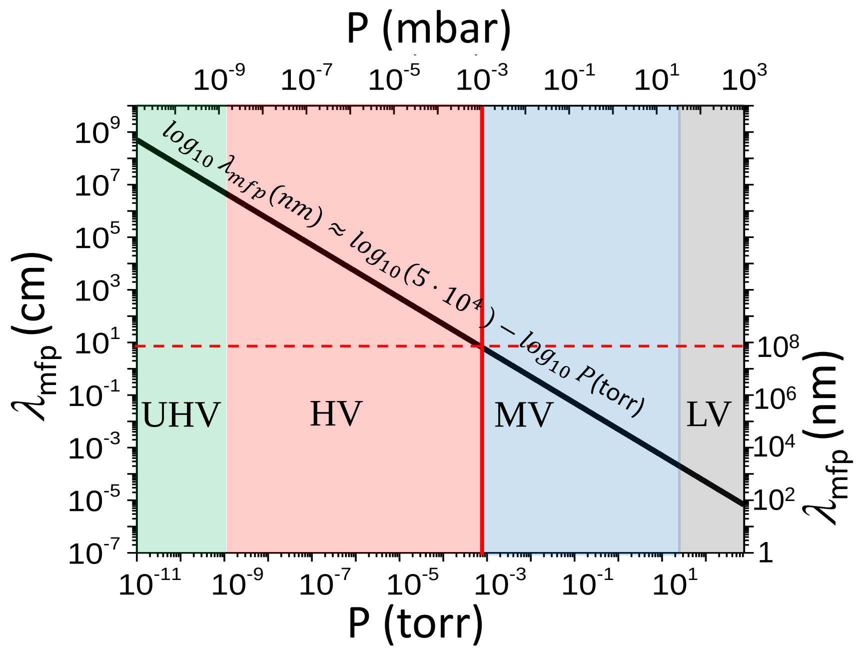
Ex.: which is λ_{mfp} at room temperature (300 K) and ambient pressure (1 atm), assuming $d_c \approx 5$ Å? [k_B=1.38·10^{-23} J/K] $\Rightarrow \lambda_{mfp} \approx 52$ nm

- *Note that*, because of collisions with other particles, the motion is not linear but zig-zag, so that the *net movement is smaller* than the ideal case above → we will consider an *effective* mean free path λ_{mfp}



- Considering zigzag trajectories, at room temperature the mean-free path λ_{mfp} remains proportional to 1/P and can be roughly estimated as:

$$\boxed{\lambda_{mfp}(\text{nm}) \approx \frac{5\cdot 10^4}{P(\text{torr})} \approx \frac{6.7\cdot 10^4}{P(\text{mbar})}}$$

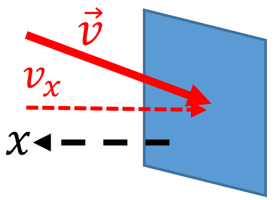


- *Note*: for $P < 10^{-3}$ torr (1.3·10^{-3} mbar, HV) we obtain λ_{mfp} >5 cm

  $\Rightarrow$ particles effectively collide with the walls of the vacuum chamber only

  $\Rightarrow$ particles trajectories are linear, not zig-zag due to collisions with other particles

Gas flux (1)



- **Gas impingement flux:** $\phi = \int_0^\infty v_x \, dn_x \Rightarrow$ it represents the number of molecules striking a surface perpendicular to *x* per unit time and unit area

- The net velocity $\vec{v}$ is the resultant of three cartesian components v_x, v_y and $v_z \Rightarrow$ we can define the steady-state distribution of molecular velocities in each of the three components; e.g., the distribution in the *x* direction (*y* and *z* are similarly defined) is:

$$f(v_x) = \frac{1}{n}\frac{dn_x}{dv_x} = \left(\frac{M}{2\pi RT}\right)^{1/2} \exp\left(-\frac{Mv_x^2}{2RT}\right) \Rightarrow \phi = \int_0^\infty v_x dn_x = \int_0^\infty v_x n f(v_x) dv_x = n\left(\frac{M}{2\pi RT}\right)^{1/2} \int_0^\infty v_x \exp\left(-\frac{Mv_x^2}{2RT}\right) dv_x = n\sqrt{\frac{RT}{2\pi M}}$$

Moreover: $\bar{v} = \sqrt{\frac{8RT}{\pi M}} \Rightarrow \phi = \frac{1}{4} n \, \bar{v}$

$$P = \frac{nRT}{N_A} \Rightarrow n = \frac{N_A P}{RT} \Rightarrow \phi = \frac{1}{4}\frac{N_A P}{RT}\sqrt{\frac{8RT}{\pi M}} = \frac{N_A P}{(2\pi MRT)^{1/2}}$$

- The **gas impingement flux** is then $\phi = \frac{N_A P}{(2\pi MRT)^{1/2}}$ (particles/cm²s)

  or $\frac{\phi}{N_A} = \frac{P}{(2\pi MRT)^{1/2}}$ (moles/cm²s)

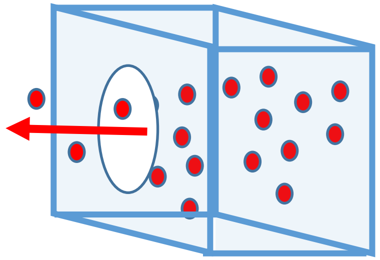
⇒ for a given gas (M) the flux depends on P and T only

$(\phi \propto P/\sqrt{T})$

- Numerically: $\phi(\text{particles/cm}^2\text{s}) = 3.513 \cdot 10^{22} \frac{P(\text{torr})}{(MT)^{1/2}} = 2.635 \cdot 10^{22} \frac{P(\text{mbar})}{(MT)^{1/2}}$

Gas flux (2)

APPLICATION #1: DETERMINATION OF GAS ESCAPING VELOCITY THROUGH A HOLE



- Consider a gas with pressure P contained in a vessel, and escaping from it through a hole of area A into a region in which the gas concentration is zero.
- *At which volume flow per second (in cm³/s) the gas does leave the vessel?*

- The rate at which particles leave is the vessel ϕA (particles/s)
- The volume flow per second is $\dot{V} = \phi A/n$ (cm³/s), with n=particles/cm³ (assumed constant over time)
- Consider $\phi = \frac{N_A P}{(2\pi MRT)^{1/2}}$ and $P = \frac{nRT}{N_A} \Rightarrow \dot{V} = \phi A/n = \frac{N_A P}{(2\pi MRT)^{1/2}} A \frac{RT}{N_A P} = \left(\frac{RT}{2\pi M}\right)^{1/2} A$ ⇨ It depends on T and M only, *not on the pressure P*
  ⇨ For a fixed gas (M) and temperature T, it is a constant

- Numerically: $\dot{V}(\text{cm}^3/\text{s}) = 3.64 \cdot 10^3 \left(\frac{T}{M}\right)^{1/2} A$, with A in cm² and M in g/mol

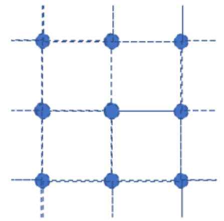
Ex.: which is $\dot{V}$ for air (M=28.9647 g/mol) at 298 K through a hole with area A = 1 cm²?

$\Rightarrow \dot{V}$= 1.17·10⁴ cm³/s = 11.7 liters/s

Application: conductance of a circular aperture

Gas flux (3)

APPLICATION #2: DETERMINATION OF TIME FOR SURFACE COATING



$\sigma = \frac{1 \text{ atom}}{(3\cdot10^{-8}\text{ cm})^2}$

$\approx 10^{15}$ atoms/cm^2

- Consider a surface exposed to a flux ϕ of a gas with molecular mass *M* and temperature *T*
- *<u>How long</u> does it take for a surface to be covered with 1 monolayer (<u>1 ML</u>) of gas particles?*

- Consider a ML containing $\sigma = 10^{15}$ atoms/cm^2 (corresponding to a square surface lattice with interatomic distance ~ 3 Å)

$\Rightarrow$ The ratio ϕ (particles/cm^{2}s) / σ (particles/cm^2 ML) represents the number of monolayers deposited per second (ML/s), *assuming all impinging atoms stick*

$\Rightarrow$ $t_C = 1/(\phi/\sigma)$ (s) represents the time for achieving a 1 ML coverage (s/ML)

$$\boxed{t_C = \frac{\sigma}{\phi} = \frac{10^{15}}{3.513\cdot10^{22}}\frac{(MT)^{1/2}}{P(\text{torr})} = \frac{2.85\cdot10^{-8}}{P(\text{torr})}(MT)^{1/2} = \frac{3.80\cdot10^{-8}}{P(\text{mbar})}(MT)^{1/2}}$$

$\Rightarrow$ The ML coating time t_C is inversely proportional to the pressure *P*

Ex. 1: which is t_C for air (*M*=28.9647 g/mol) at 298 K and ambient pressure … ?

$\Rightarrow t_C = 3.49\cdot10^{-9}$ s = 3.5 ns

The coating time changes of ~10^{13} (following the 1/*P* dependence)!

Ex. 2: … and at *P*= 10^{-10} torr (UHV)?

$\Rightarrow t_C = 2.65\cdot10^{4}$ s ~ 7h 21'

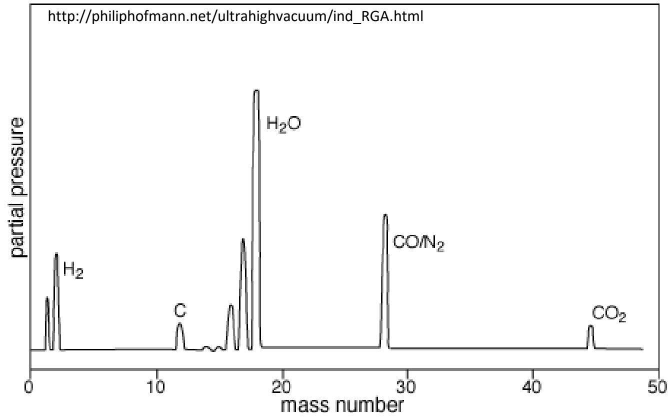
Langmuir

APPLICATION #2: DETERMINATION OF TIME FOR SURFACE COATING

- How long does it take for a surface to be covered with 1 monolayer (1 ML) of gas particles?

$$\boxed{t_C(\mathrm{s}) = \frac{2.85\cdot 10^{-8}}{P(\mathrm{torr})}(MT)^{1/2} = \frac{3.80\cdot 10^{-8}}{P(\mathrm{mbar})}(MT)^{1/2}}$$

$P\cdot t_C$ = const
(at fixed M and T)



- t_C is the **contamination time**, that is the time during which one ML of contaminants (essentially H_2, H_2O, and CO, from the vacuum chamber environments) will be accumulated on the surface.

- In order to work (during deposition and characterization) under clean conditions for a time t^*, we must have $t^* < t_C$

Ex. t_C ~ 7h21' at 10^{-10} torr (UHV), 44'10'' at 10^{-9} torr, 4'24'' at 10^{-8} torr, 26'' at 10^{-7} torr, 3'' at 10^{-6} torr, … ⇒ UHV is required when managing ultrathin films (deposition and characterization) and/or very pure materials need to be deposited!

- **Surface exposure** is measured in **Langmuir**, defined as 1 L = 10^{-6} torr·s
  - 1 L means exposure to 10^{-6} torr per 1 s, 10^{-7} torr per 10 s, … 10^{-10} torr per 10^4 s (~2h 47')
  - Because t_C = $2.65\cdot 10^4$ s at 10^{-10} torr, in $t^*=10^4$ s only the fraction t^*/t_C = 0.38 ML is deposited $\Rightarrow$ 1 L corresponds to 0.38 ML at 10^{-10} torr (UHV)

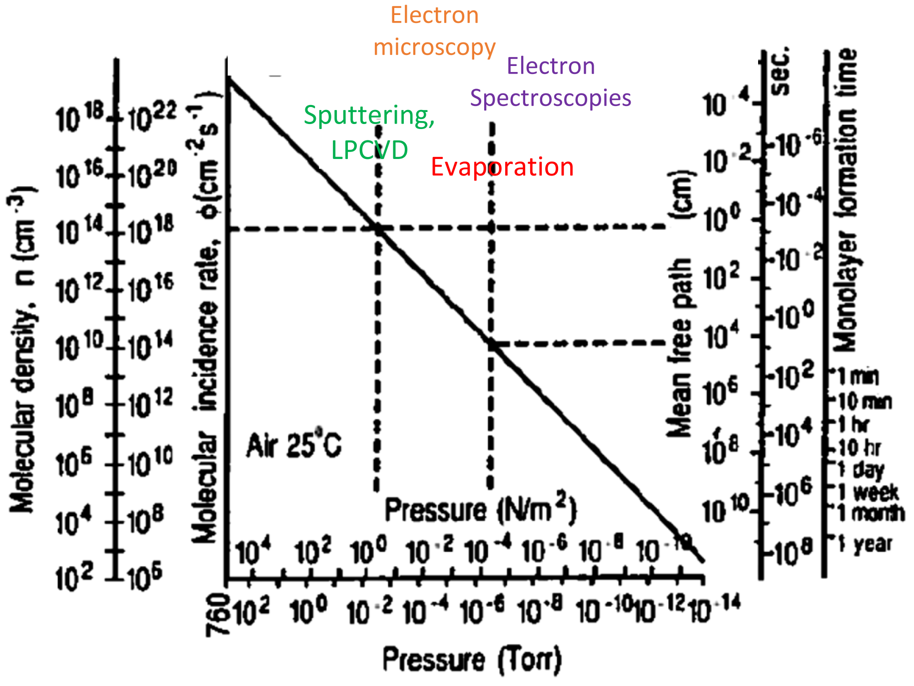
Summarizing…

$$\lambda_{mfp}(\text{nm}) \approx \frac{5\cdot 10^4}{P(\text{torr})} \approx \frac{6.7\cdot 10^4}{P(\text{mbar})} \propto 1/P$$

$$\phi(\text{particles/cm}^2\text{s}) = 3.513\cdot 10^{22}\frac{P(\text{torr})}{(MT)^{1/2}} = 2.635\cdot 10^{22}\frac{P(\text{mbar})}{(MT)^{1/2}} \propto P$$

$$\dot{V}(\text{cm}^3/\text{s}) = 3.64\cdot 10^3 (T/M)^{1/2} A$$

$$t_C(\text{s}) = \frac{2.85\cdot 10^{-8}}{P(\text{torr})}(MT)^{1/2} = \frac{3.80\cdot 10^{-8}}{P(\text{mbar})}(MT)^{1/2} \propto 1/P$$



Molecular density, incidence rate, mean free path, and monolayer formation time as a function of pressure. (from M. Ohring, Materials Science of Thin films, Academic Press, Chapter 2)

- In thin films for research & development *P* spans over **13 orders of magnitude** (from 760 torr to 10^{-10} torr):

1 bar to 30 mbar $\Rightarrow$ Low Vacuum
30 mbar to 10^{-3} mbar $\Rightarrow$ Medium Vacuum
10^{-3} mbar to 10^{-9} mbar $\Rightarrow$ High Vacuum (HV)
10^{-9} mbar to 10^{-12} mbar $\Rightarrow$ Ultra-High Vacuum (UHV)
$<10^{-12}$ mbar $\Rightarrow$ Extremely Ultra-High Vacuum

Gas flow

	Net gas flow	Pressure gradient
• *Before* we considered an isolated sealed system in thermodynamical equilibrium	✗	✗
• *Now* we consider a **net direct movement** of gas due to the presence of attached pumps	✓	✓

- There are *3 different flow regimes*, depending on local geometry of the system, pressure, temperature, type of gas.
- We define D_P as a characteristic dimension of the system, e.g. chamber or pipe diameter.
- We define the Knudsen number $Kn = \lambda_{mfp} / D_P$ to distinguish between regimes.

1. **Molecular flow:** low gas pressure, HV/UHV, $\lambda_{mfp} > D_P \Leftrightarrow Kn > 1$

   Kinetic theory provides an accurate picture of molecular motion.

   Collisions are between particles and walls.

   Ex.: HV evaporators, electron spectroscopy and microscopy techniques

2. Viscous flow: high pressure, $\lambda_{mfp} \ll D_P \Leftrightarrow Kn < 0.01$

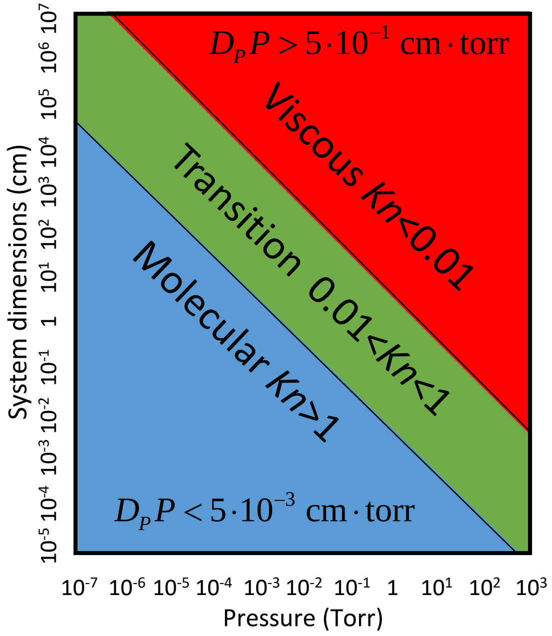
   Collisions between particles predominate.

   At low velocities the flow is laminar, with zero velocity at the walls and maximum at the center; at high velocities the flow is turbulent and influenced by obstacles.

   Ex.: APCVD processes

3. Transition flow: $\lambda_{mfp} < D_P \Leftrightarrow 0.01 < Kn < 1$

$$Kn \approx \frac{5 \cdot 10^{-3}}{D_P(\text{cm}) P(\text{torr})}$$

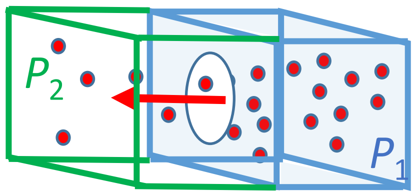


Dominant gas flow regimes in air as a function of system dimensions and pressure

Pumping (1)

- Consider a molecular flow of gas through a hole of area A that separates two chambers maintained at pressures P_1 and P_2.

  ⇨ A ***gas flow driven by the pressure difference*** is measured



- We define $Q=C(P_1-P_2)$, where Q is the gas throughput in Pressure×Volume/Time (torr × liter/s), and C is the conductance in Volume/Time (liter/s).

- The **net gas flow** at the hole is $(\phi_1-\phi_2)A$ (particles/s) or $(\phi_1-\phi_2)A/n$ (cm^3/s), where $\phi_1-\phi_2$ is the *net* flux (particles/cm^2 s) crossing the hole

$\Rightarrow$ it corresponds to the volume flow per second $\dot{V}$ previously calculated, now representing the conductance of the hole:

$$\boxed{\dot{V} = C = 3.64(T/M)^{1/2}A}$$

with C expressed in liters/s, A in cm^2 and M in g/mol.

- Note: in molecular flow, C does not depend on P_1 and P_2, but only on M (type of gas), A (geometry), and T (temperature)

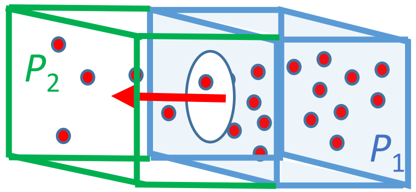
  Ex.: air at 298 K $\Rightarrow C = 11.7 \cdot A$ liters/s

Which is the physical meaning of Q?

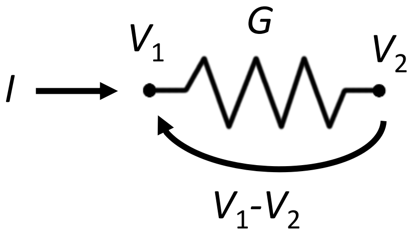
Assuming $P_1=P=$cost. and $P_2=0$, we have $Q = CP = \dot{V}P = P\frac{dV}{dt} = \frac{d(PV)}{dt} = k_B T\frac{dn}{dt}$ ⇨ Q is *proportional* to the **number of molecules** crossing the hole **per second**

Pumping (2)

- Gas flow: $Q=C(P_1-P_2)$



- Electrical current flow: $I=G(V_1-V_2)$



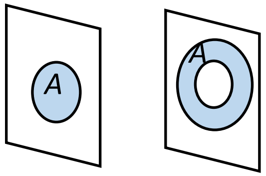
They are formally equivalent $\Rightarrow$ the same «sum» rules (series/parallel) hold

Series: $$\frac{1}{C_{series}}=\sum_i \frac{1}{C_i} \quad \left(C_{series}<C_i \ \forall\, i\right)$$

Parallel: $$C_{parallel}=\sum_i C_i \quad \left(C_{parallel}>C_i \ \forall i\right)$$

<u>Conductances (in liters/s) of various geometric shapes for molecular flow of air at 298 K:</u>

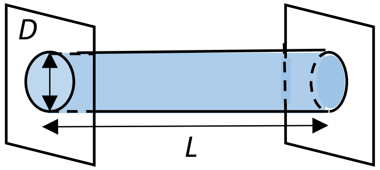
Circular or annular aperture



$$C=11.7A$$

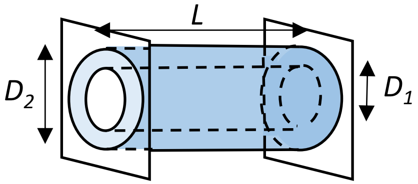
(with areas in cm², linear dimensions in cm)

Pipe



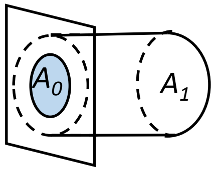
$$C=12.2\frac{D^3}{L}$$

Annular pipe



$$C=12.2\frac{\left(D_2-D_1\right)^2\left(D_2+D_1\right)}{L}$$

Aperture in end of pipe



$$C=11.7\frac{A_0}{1-A_0/A_1}$$

(see Appendix 1 for an example of conductance calculation)

Pumping speed (1)

- **Pumping** is the process of **removing gas particles** from the system through the action of pumps.
- The **pumping speed** is defined as $S=Q/P$, where
  - Q (torr·l/s) is the throughput, *proportional* to the number of particles crossing the *inlet* section per unit time, assumed constant in all sections of the system
  - P (torr) is the pressure at the pump *inlet*
- S (l/s) represents the volume of gas passing the plane at the inlet port per unit time when the pressure at the pump inlet is P

NOTE: C and S have the *same units but different physical meanings*:

- $C=Q/(P_1-P_2)$ implies a component across which a pressure *difference* (P_1-P_2) exists
- $S=Q/P$ refers to a given plane that coincides with the inlet of a pump at *pressure* P, or may be considered to be a pump for preceding portion of the system (pipe+pump).

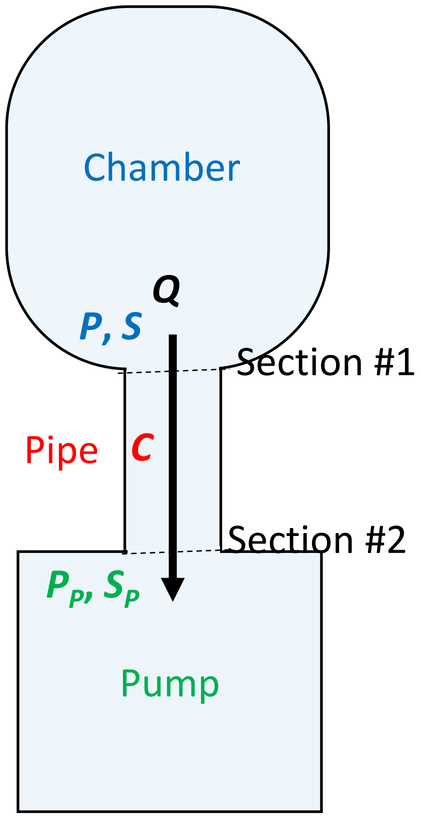
Problem: consider a pipe with conductance C connecting a chamber at pressure P to a pump at pressure P_P

$\Rightarrow$ *which is the effective pumping speed at the inlet of the chamber (section#1)?*

Q = throughput (torr·l/s) of the gas flow, constant along the pipe (and sections #1 and #2)

$$\left.\begin{array}{l} P = \text{pressure (torr) of the chamber measured at section\#1} \\ S = \text{pumping speed (l/s) measured at section\#1} \end{array}\right\} \text{chamber}$$

$$\left.\begin{array}{l} P_P = \text{pressure (torr) of the pump measured at section\#2} \\ S_P = \text{«intrinsic» pumping speed (l/s) measured at section\#2} \end{array}\right\} \text{pump}$$



Pumping speed (2)

chamber

pipe

pump

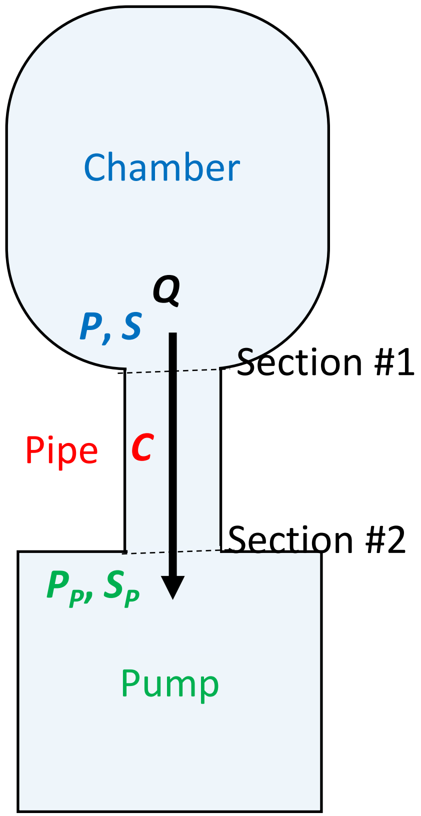
$$\begin{cases} \boxed{S = Q/P} \\ \boxed{Q = C(P - P_P)} \\ \boxed{S_P = Q/P_P} \end{cases} \Rightarrow \frac{1}{C} = \frac{P-P_P}{Q} = \frac{P}{Q} - \frac{P_P}{Q} = \frac{1}{S} - \frac{1}{S_P} = \frac{S_P - S}{S_P S} \Rightarrow \frac{S_P S}{C} = S_P - S \Rightarrow S\left(1+\frac{S_P}{C}\right) = S_P \Rightarrow S = \frac{S_P}{1+\frac{S_P}{C}}$$

$$\boxed{S = \frac{S_P}{1+S_P/C}}$$

S = effective pumping speed (l/s) at the base of the chamber (#1)

S_P = intrinsic speed (l/s) at the pump inlet (#2)

Note: S increases by increasing C (i.e. making pipes as shorter and wider as possible, because $C \propto D^3/L$)



Ex.1: $S_P=C \rightarrow S=S_P/2$

Ex.2: turbopump with pumping speed S_P=2000 l/s + nipple (pipe) with D=127mm (5 inches) and L=250.7 mm

C=997 l/s ⇒ S=665 l/s (the nipple reduces the pumping speed to 1/3 of the intrinsic value)

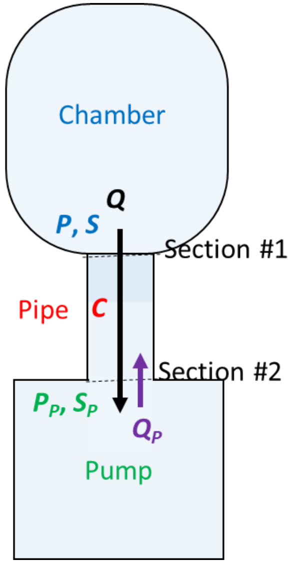
Base pressure (1)

- **Which is the time required to achieve a given pressure *P*?**

$$P(t) = P_0 + (P_i - P_0)\exp\left(-\frac{S}{V}t\right) = P_0 + (P_i - P_0)\exp\left(-\frac{t}{\tau}\right), \text{ with } \tau = \frac{V}{S} = \frac{V}{S_P}\left(1 + \frac{S_P}{C}\right) \Rightarrow t = -\frac{V}{S}\ln\frac{P(t) - P_0}{P_i - P_0}$$

  - P_0 is the ultimate pressure of the pump (>0 because of outgassing terms in the pump volume)
  - P_i is the initial pressure (at t=0)

(see Appendix 2 for the derivation of the *P*(*t*) equation)



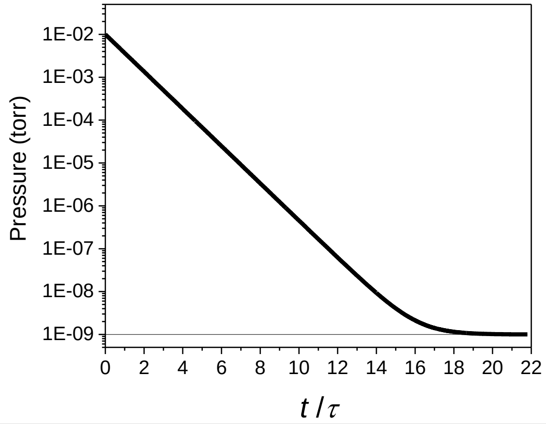
Ex. 1: **rotary pump** with S_P=8 l/s and P_0=10^{-4} torr + cylindrical chamber with ϕ=50 cm and *h*=80 cm ($\Rightarrow$ *V*=157 l), assuming no pipe between ($C \to \infty$) $\Rightarrow$ τ = 19.6 s

- It is used to evacuate the chamber from ambient pressure (P_i=760 torr) to a lower pressure P
- How log does it take to pump down to *P*=10^{-2} torr (typical forepumping time before to start a turbopump in cascade)? About 3'41s

Ex. 2: **turbopump** with pumping speed S_P=800 l/s + chamber with volume *V*=270 l (e.g., a sphere with diameter 40 cm), assuming no pipe between ($C \to \infty$) $\Rightarrow$ τ = 0.34 s

- Initial pressure (*t*=0): P_i=10^{-2} torr
- Ultimate (base) pressure ($t \to \infty$): P_0=10^{-9} torr

⇨ P_0 is «practically» (within 1%) achieved after $t \approx 18\tau \sim 6$ s



Base pressure (2)

Pumping is needed for

- **Volume** pumping (residual gases, outgas from the pump) ➡ $P(t) = P_0 + (P_i - P_0)e^{-\frac{S}{V}t}$
- Pumping of species **outgassed** from internal surfaces:
  - **permeation** from seals and metal walls penetrated by small gas molecules
  - **diffusion** (from plastic and seals with gas dissolved)

  volatile species *through system walls*

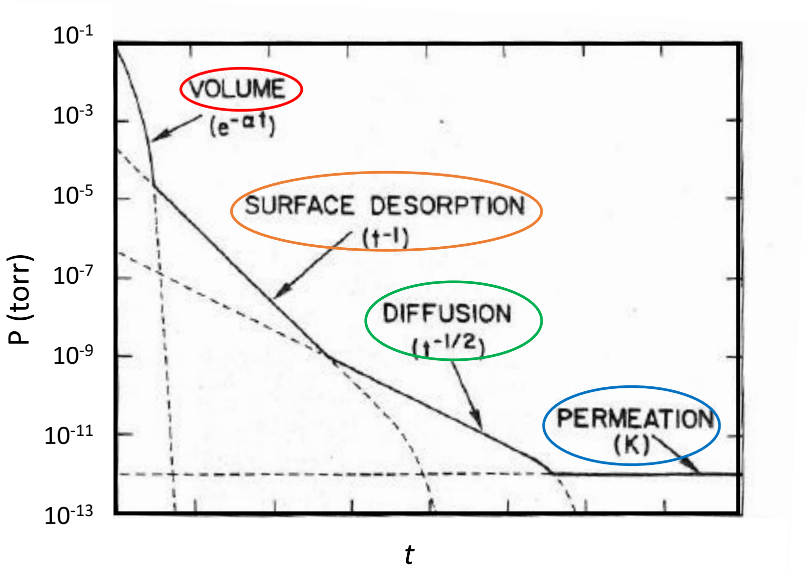
  - **desorption** of gas molecules (primarily water, previously ab/adsorbed) *from chamber surfaces and vacuum hardware*

$$P(t) = P_0 + (P_i - P_0)e^{-\frac{S}{V}t} + \frac{Q_{pe}}{S} + \frac{Q_{diff}}{S} + \frac{Q_{des}}{S}$$

Q_{pe}, Q_{diff}, and Q_{des} are the throughput (torr·l/s) associated to permeation, diffusion, and desorption

Base pressure (3)

$$P(t) = P_0 + (P_i - P_0)\, e^{-\frac{S_P}{V}t} + \frac{Q_{pe}}{S} + \frac{Q_{diff}}{S} + \frac{Q_{des}}{S}$$



Rate limiting pumping processes during evacuation of a vacuum chamber (from M. Ohring, Materials Science of Thin films, Academic Press, Chapter 2)

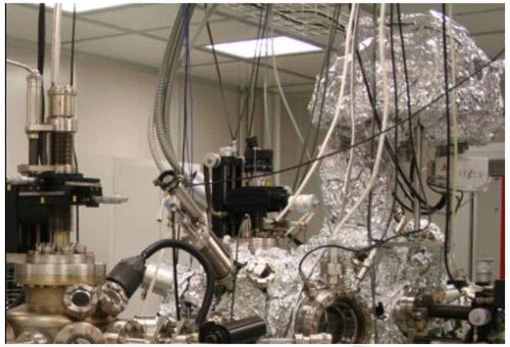
- The throughputs depend on **materials** (*Q* are lower for metals and glasses than polymers) and **surface conditions** (smooth, porous, clean, …)
- **Most pumping time to achieve UHV is spent in removing gas from surfaces.**
- Adsorbants are either
  - physically bound to surfaces (**physisorbed**) by weak Van der Waals interaction
  - chemically bonded to surface atoms (**chemisorbed**) by stronger ionic or covalent bonds

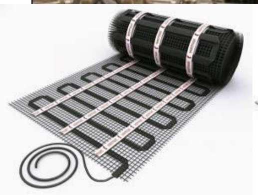
⇒ **In both cases elevated temperatures promote degassing**

Bake-out: chamber walls and components are heated to ~200°C for ~24-48 h while the system is continuously pumped

⇒ water (and other contaminants) is efficiently removed from surfaces

⇒ UHV can be obtained starting from HV conditions (achieved by pumping) in reasonable times (~1-2 days)





Heating strip + aluminum foil (like cooking in foil…)

Vacuum systems: pumps and more

Vacuum chamber(s) +

Tubing

Pumps

Valves

Gauges

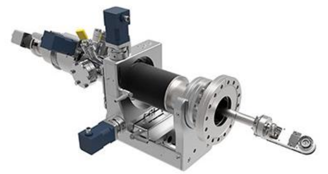
Other (windows, doors, holders, MBE, XPS, sputter gun, …)

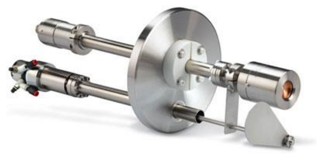
Tubing – to connect chambers, pumps, …

Valves – to separate chambers, pumps, …

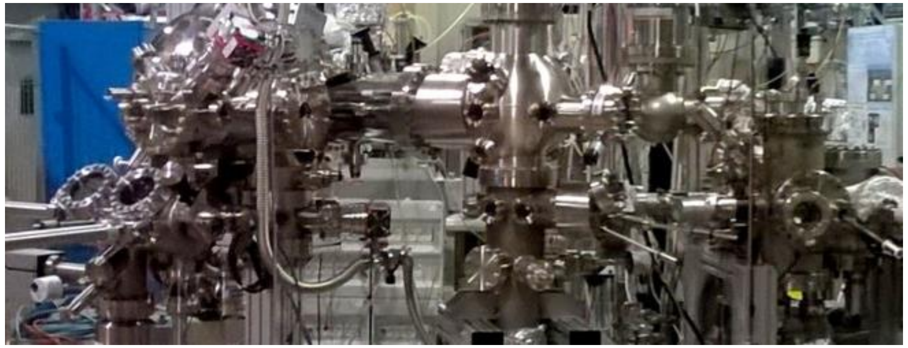
Gauges – to measure pressure, gas flow, …

Other –
- glass windows, fast entry doors, sample holders, …
- film deposition (MBE), sample characterization (XPS), substrate cleaning (sputter gun), …





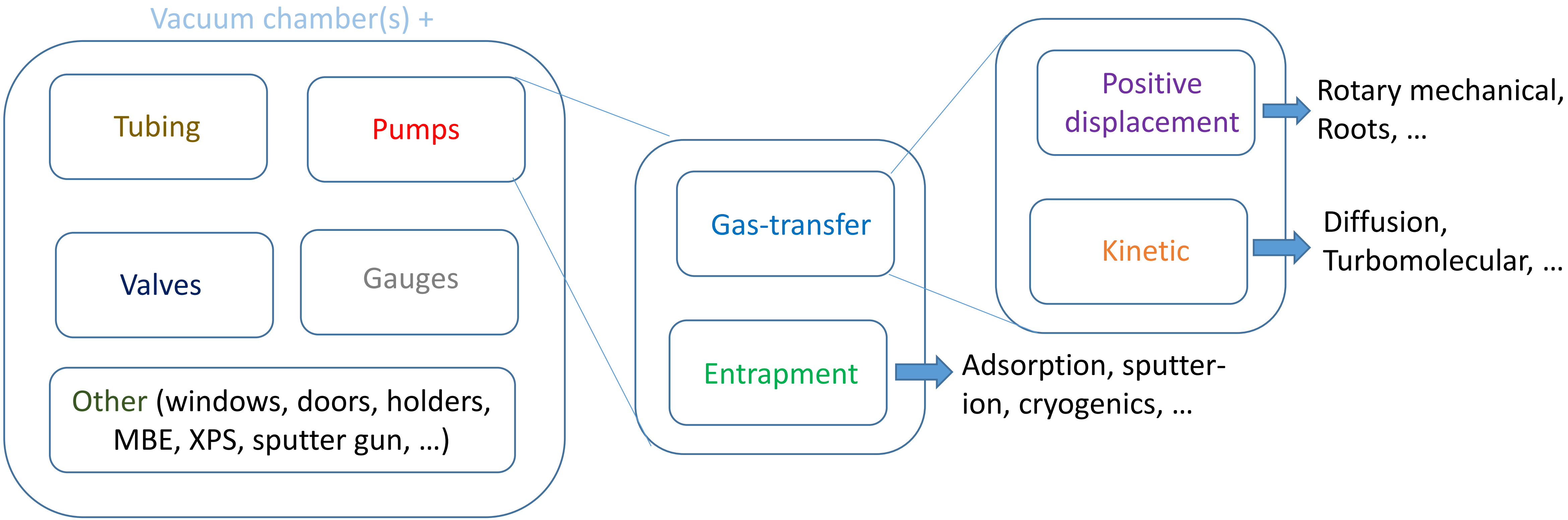
Pumps –
- to achieve vacuum (e.g. from ambient pressure)
- to preserve vacuum (e.g. HV or UHV during operation)



[APE beamline @ Elettra, Trieste]

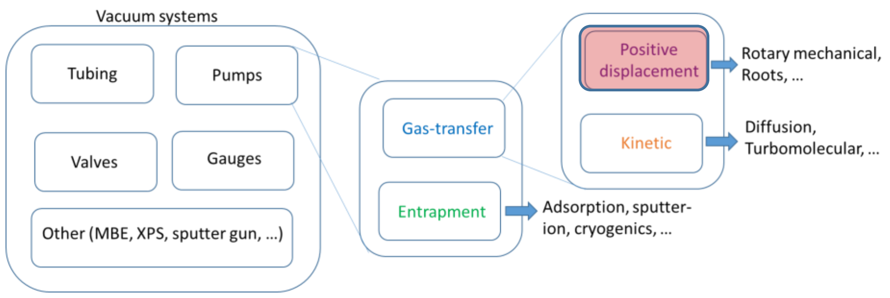
[images by courtesy of Kurt J. Lesker]

Vacuum pumps



- Pumps *do not remove molecules by exerting forces* on them, but **altering natural molecular motion**.
- **Entrapment pumps** condense or chemically bind molecules to surfaces situated within the chamber being pumped. It can be a ***reversible*** process.
- **Gas-transfer pumps** remove gas molecules from the pumped volume and convey them to the ambient. It is an ***irreversible*** process.
  - **Positive displacement** pumps displace the gas mechanically (by drawing it into a compartment at the inlet and then moving to the outlet)
  - **Kinetic pumps** impart kinetic energy to the gas (by rotating at high speed or by providing an impulse in direction of the flow).

Gas transfer - Positive Displacement Pumps (1)

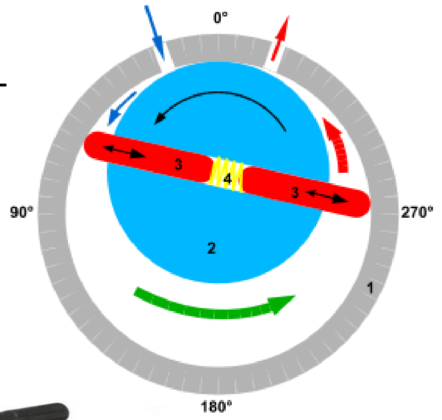


- Pumps *remove molecules by* **altering natural molecular motion**.
- **Gas-transfer pumps** remove gas molecules from the pumped volume and convey them to the ambient. It is an ***irreversible*** process.
- **Positive displacement** pumps displace the gas mechanically (by drawing it into a compartment at the *inlet* and then moving to the *outlet*).

Rotary Mechanical Pump

1) **Rotary-vane pump**: it contains an eccentrically mounted rotor (2) with spring-loaded (4) vanes (3). During rotation the vanes slide in and out within the stator (1), enabling a quantity of gas to be confined, compressed, and discharged through an exhaust valve to the atmosphere.

$$\boxed{S_P = 3-55\ \mathrm{l/s}}$$

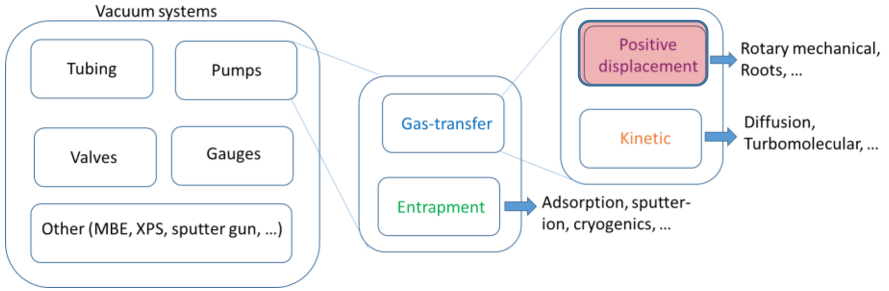


- **Oil** is employed in both pumps as a sealant as well as a lubricant between moving components. Oil evaporation by heating can cause air contamination and diffusion to the chamber, thus degrading the vacuum.

  Oil traps are usually placed between the pump and the chamber to avoid oil diffusion to the chamber

- Compression ratios **up to 10^6** can be achieved. Single-stage pumps can achieve an ultimate pressure P_0~10^{-2} torr, while two-stage can reach P_0~10^{-4} torr.
- Rotary pumps are frequently used to produce **pre-vacuum** to operate diffusion and turbomolecular pumps.

Gas transfer - Positive Displacement Pumps (2)

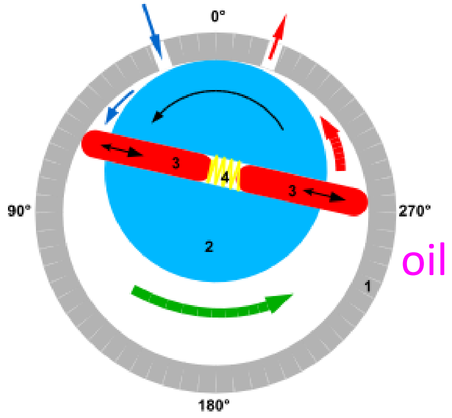


- Pumps *remove molecules by* **altering natural molecular motion**.
- **Gas-transfer pumps** remove gas molecules from the pumped volume and convey them to the ambient. It is an ***irreversible*** process.
- **Positive displacement** pumps displace the gas mechanically (by drawing it into a compartment at the inlet and then moving to the outlet).

Rotary Mechanical Pump

(See more details on Appendix 3)

1) Rotary-vane pump

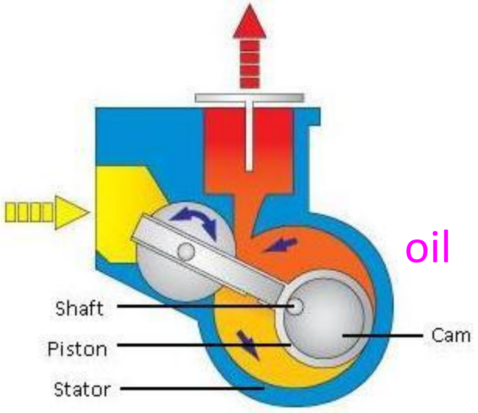


$S_P = 3 - 55$ l/s

$P_0 \sim 10^{-2}$ torr

pre-vacuum

2) Rotary-piston pump

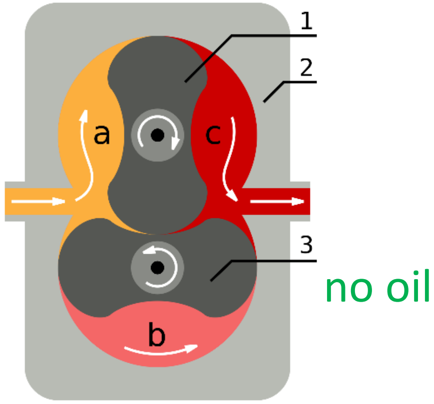


$S_P = 8 - 420$ l/s

$P_0 \sim 10^{-2}$ torr

pre-vacuum

3) Root pump

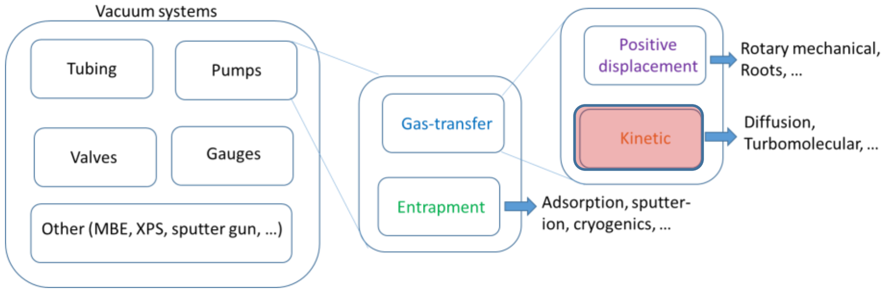


$S_P \approx 1000 - 2000$ l/s

$P_0 \sim 10^{-5}$ torr

Large pumping speed at 1 torr

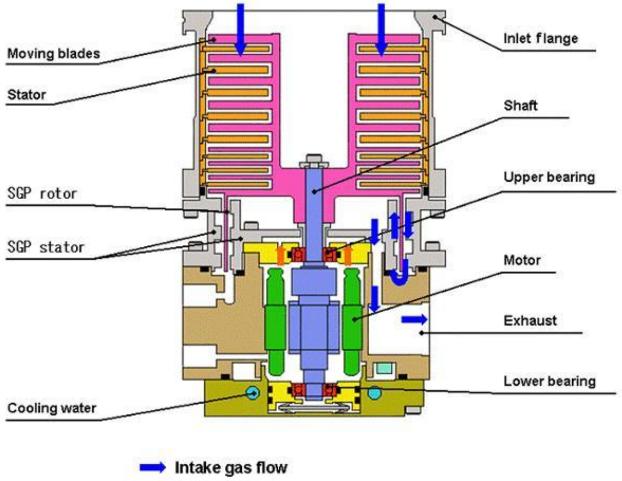
Gas transfer – Kinetic Pumps (1)



- Pumps *remove molecules by* **altering natural molecular motion**.
- **Gas-transfer pumps** remove gas molecules from the pumped volume and convey them to the ambient. It is an ***irreversible*** process.
- **Kinetic pumps** impart kinetic energy to the gas (by rotating at high speed or by providing an impulse in direction of the flow)

Turbomolecular Pump

- A preferred direction is imparted to molecular motion, the impulse being caused by impact with a rapidly whirling turbine rotor spinning at rates of $2{\cdot}10^4$-$3{\cdot}10^4$ rpm.
- The pump is generally a vertical axial-flow compressor made by many rotor-stator pairs in series: gas captured by the upper stages is transferred to the lower stages where it is successively compressed, and so on.
- Molecules hit by the rotor blades must reach the stator blades *before colliding* with other molecules on the way: distance between blades (~1 mm) < $\lambda_{mfp} \Rightarrow$ **P<5·10^-2 torr** $\Rightarrow$ **mechanical forepump** (e.g. rotary pump) is required at the outlet.



$$S_P = 10^3 \text{ l/s}$$

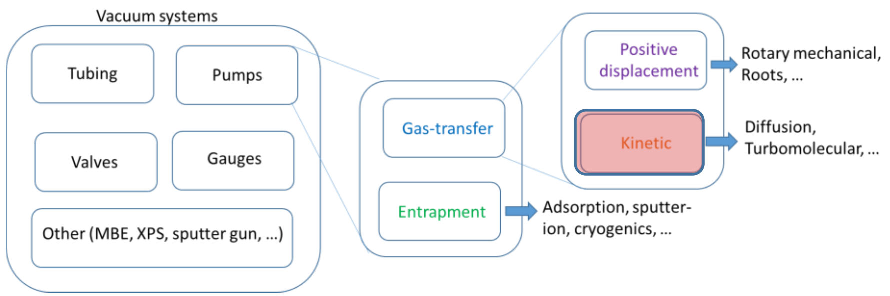
$$P_0 \sim 10^{-10} \text{ torr}$$

Used for HV, UHV

- Compression ratio *r* increases with $M \Rightarrow S_P$ larger for air than hydrogen
- Thanks to the very large r, oil backstreaming from pre-vacuum is reduced to negligible levels $\Rightarrow$ **oil-free**

(See more details on Appendix 4)

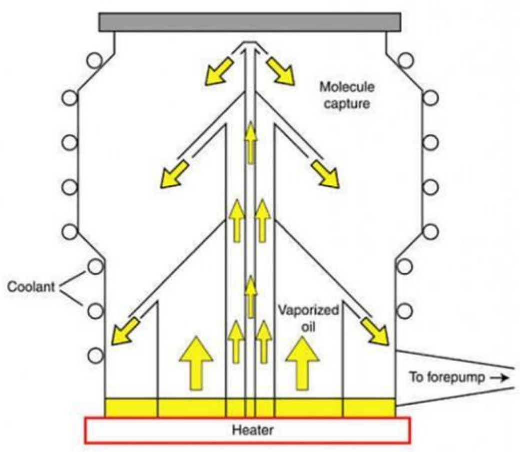
Gas transfer – Kinetic Pumps (2)



- Pumps *remove molecules by* **altering natural molecular motion**.
- **Gas-transfer pumps** remove gas molecules from the pumped volume and convey them to the ambient. It is an ***irreversible*** process.
- **Kinetic pumps** impart kinetic energy to the gas (by rotating at high speed or by providing an impulse in direction of the flow)

Diffusion Pump

- A fluid medium with low vapor pressure (typically a silicone oil) is boiled and vaporized in a multistage jet assembly.
- As the oil vapor stream emerges from top nozzles, it collides with residual gas molecules in the chamber and imparts momentum to them.
- These molecules are thus driven towards the bottom of the pump and thus compressed to the exit side, where they are exhausted.
- Diffusion pumps operate in the molecular flow regime with pressure **< 5·10^{-2} torr** ⇒ **mechanical forepump** (e.g. rotary pump) is required at the outlet



$S_P = 2 \cdot 10^4\ l/s$

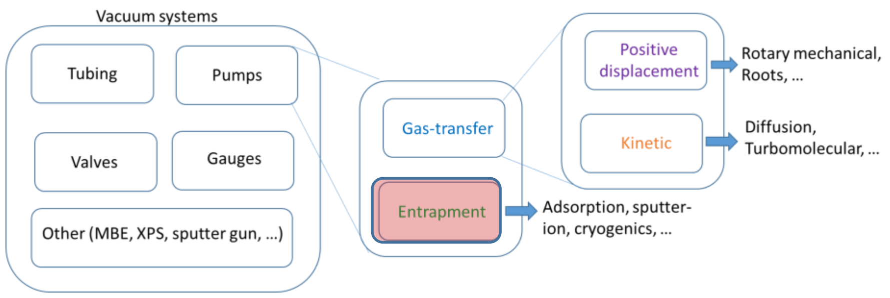
$P_0 \sim 10^{-3}$ -10^{-10} torr

Used for coating

- In contrast to mechanical pumps, diffusion pumps have no moving part.
- Backstreming of oil into the chamber leads to materials contamination ⇒ not suitable for UHV applications

(See more details on Appendix 5)

Gas Entrapment Pumps (1)

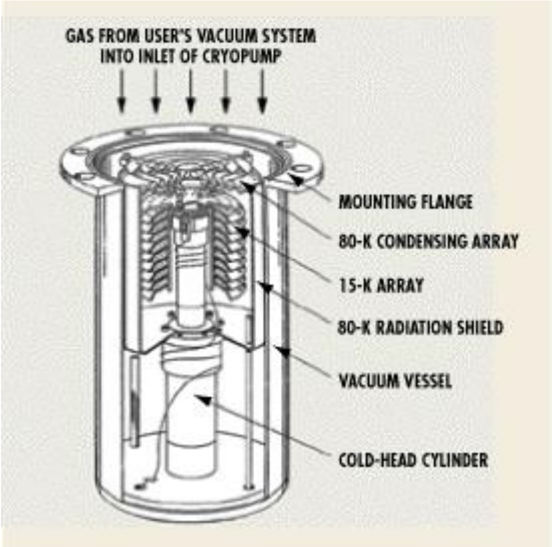


- Pumps *remove molecules by* **altering natural molecular motion**.
- **Entrapment pumps** <u>condense</u> or chemically <u>bind</u> molecules <u>to</u> surfaces situated within the chamber being pumped. It can be a ***reversible*** process.

Cryopump

- It relies on the condensation of vapor molecules on surfaces cooled below 120 K (bare metal surfaces or microporous surfaces, e.g. zeolite).
- Temperature-dependent van der Waals forces are responsible for physically binding or sorbing gas molecules.

- liquid ***N*$_2$ (LNT)** $\rightarrow$ P **~10^{-3} torr**
- **T<20 K** by closed-circuit refrigerators $\rightarrow$P **~10^{-10} torr (UHV)**
- An **initial forepressure ~10^{-3} torr** is needed to prevent from *excessive thermal load* and *accumulation of thick ice* condensate on the cryopanels $\Rightarrow$ **mechanical forepump** (e.g. rotary+turbopump) is required



$S_P = 3$ l/s cm^2 @ 20 K

P_0~10^{-3} -10^{-10} torr

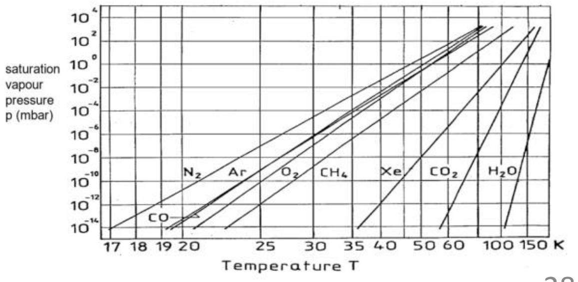
Used for HV to UHV

- <u>No moving parts, no oil</u>.
- High pumping speed (limited only by gas impingement rate)
- The ultimate pressure P_0 for a gas depends on its saturation pressure P_{S0} at T_0 :

$$P_0 = P_{S0}\sqrt{300/T_0}$$

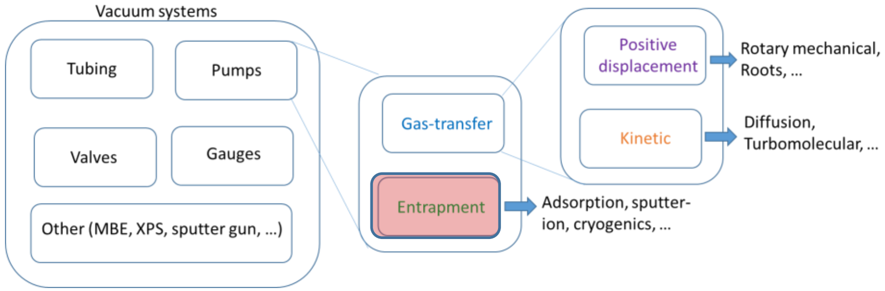
O_2, N_2 : low P_{S0}

H_2, He, Ne : high P_{S0}



[(See more details on Appendix 6)](#)

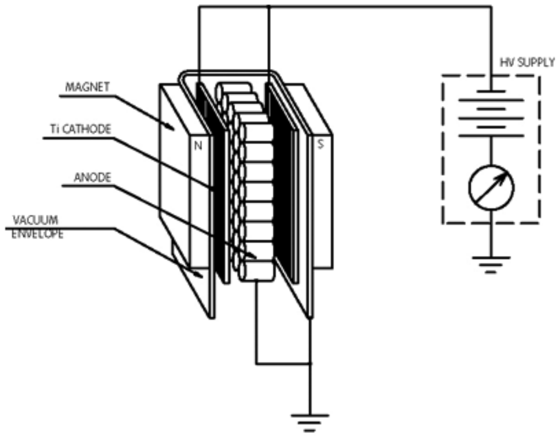
Gas Entrapment Pumps (2)



- Pumps *remove molecules by* **altering natural molecular motion**.
- **Entrapment pumps** condense or chemically bind molecules to surfaces situated within the chamber being pumped. It can be a ***reversible*** process.

Sputter ion pump

- It relies on sorption processes initiated by ionized gas to achieve pumping.
- A cold-cathode electrical discharge between titanium cathode (-) and stainless steel anode (+), with ΔV~few kV, is realized.
- Electrons emitted from the cathode are trapped by a transverse magnetic field (~ few kG) forming an high-density cloud (~ 10^{10} electrons/cm^3).
- After impact ionization of residual gas molecules, the ions travel to the cathode where they knock out or sputter Ti atoms.
- The latter deposits elsewhere in the pump forming films that getter or combine with reactive gases (N_2, O_2, H_2), and are successively buried by fresh layers of sputtered metal.



STRUCTURE OF TRIODE SPUTTER ION PUMP

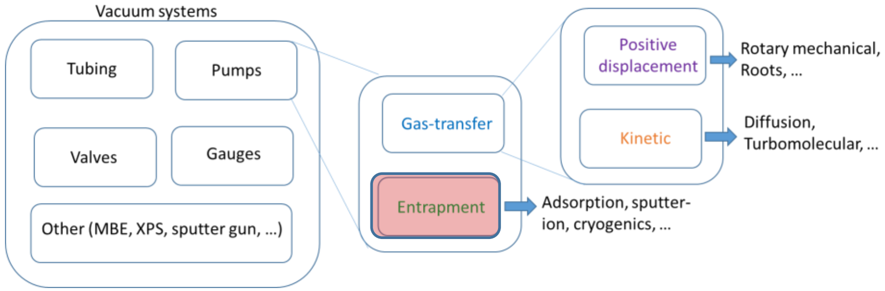
$S_P \sim 100$ l/s

$P_0 \sim 10^{-10}$ torr

Used for UHV

- No moving parts, no oil
- An **initial forepressure ~10^{-6} torr or lower** is needed to not quench the discharge $\Rightarrow$ **mechanical forepump** (e.g. rotary+turbopump) is required
- **Pumping action for H_2 is few times more efficient than N_2, O_2, H_2O** and hundred times than Ar.
- Gas are permanently removed (in cryopumps they are not) but **the lifetime is inversely proportional to the pressure**.

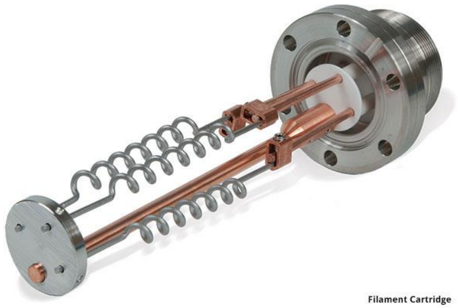
Gas Entrapment Pumps (3)



- Pumps *remove molecules by* **altering natural molecular motion**.
- **Entrapment pumps** condense or chemically bind molecules to surfaces situated within the chamber being pumped. It can be a ***reversible*** process.

Ti sublimation pump

- Ti metal is thermally evaporated (sublimed) by periodic current (~40 A), performing chemical sorption as above.
- Eventually, cryogenically cooled surfaces are used, performing a combination of cryopumping and chemical sorption.
- An **initial forepressure ~10^{-6} torr or lower** is needed to not quench the discharge $\Rightarrow$ **mechanical forepump** (e.g. rotary+turbopump) is required.



Filament Cartridge

Used for UHV maintenance (supporting other pumps)

Getter pump

- It is based on a non-evaporable (solid) getter (NEG) material (typically zirconium).
- When gas molecules strike the NEG, they combine with it chemically or by absorption, removing small amount of gases from the evacuated space.
- Flashed getters are placed in a reservoir and then evaporated in the vacuum chamber, e.g. for **pumping low power vacuum tubes** (lamps, thermionic valves…)
- Initial forepressures **~10^{-6} torr or lower** are needed for all these pumps.

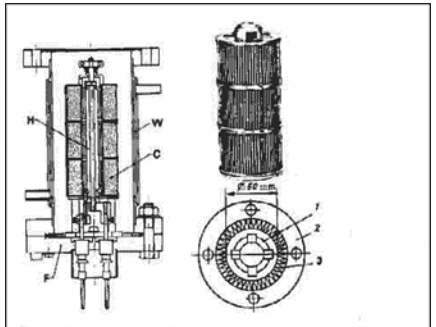
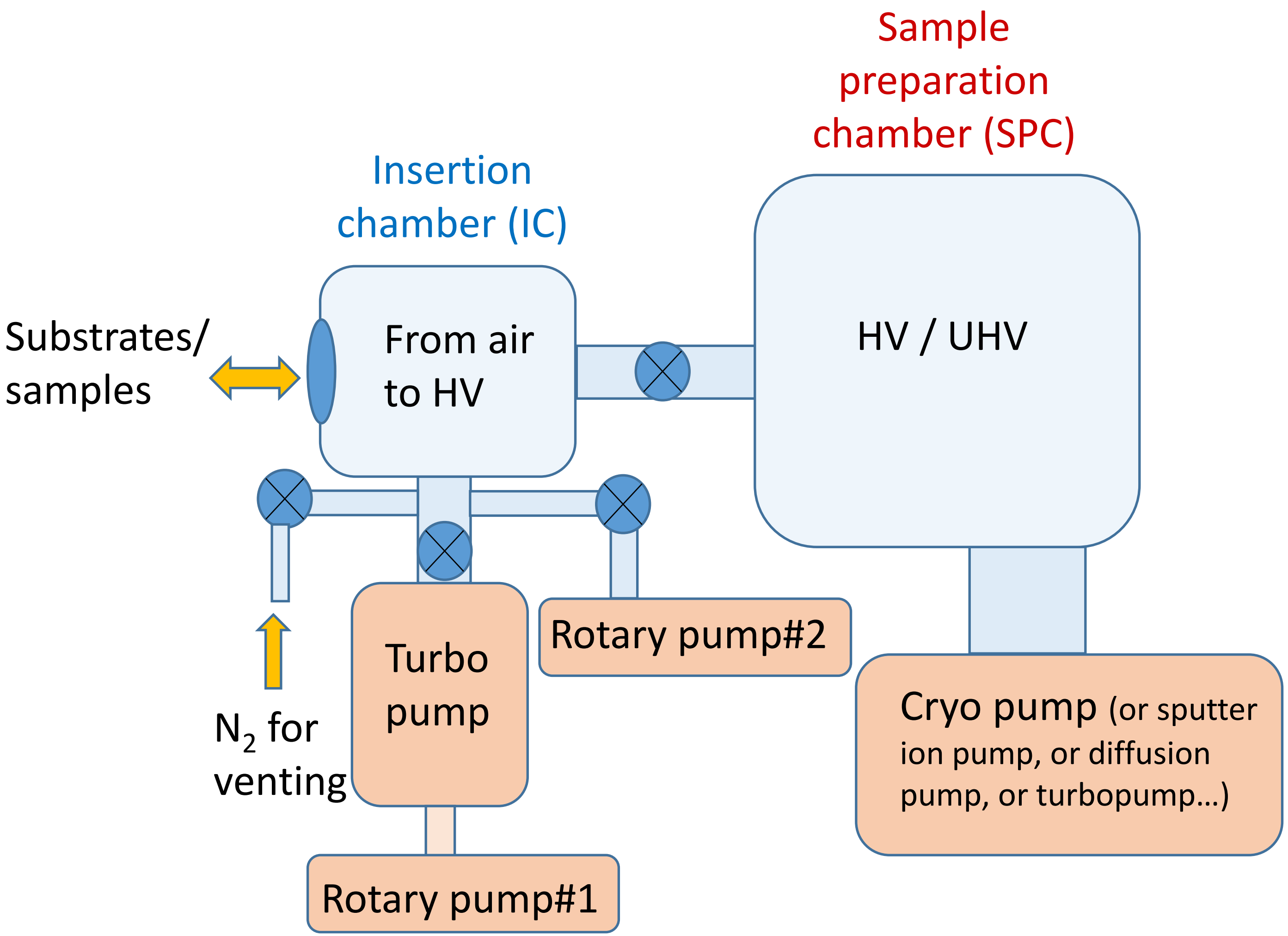


Figure 16 The view of a nonevaporated getter pump. Left, cross section schematic; right, corrugated strip design. H and 1, electric heater; W, water cooling (for large pumps); C and 3, the getter ribbon; 2, flange.

(See more details on Appendix 7)

Vacuum systems: design …

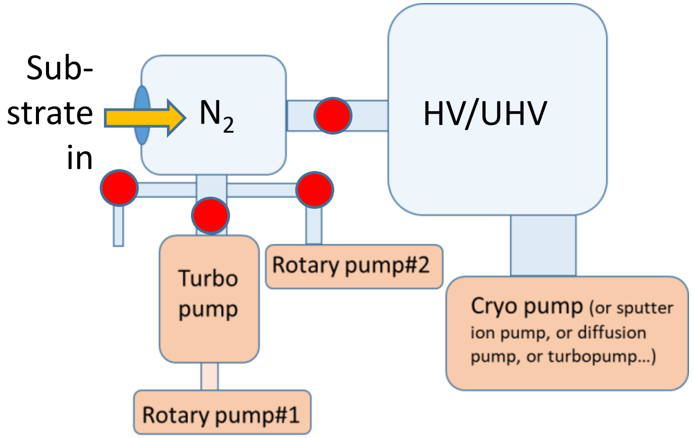


- 2 vacuum chambers
- 2 HV pumps
- 2 MV (rotary) pumps
- venting valve

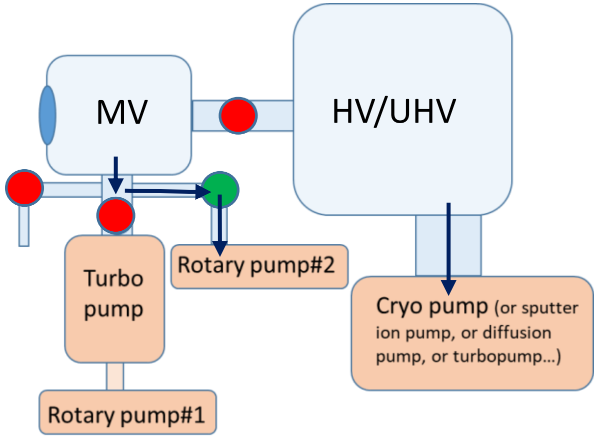
+ MBE/XPS/LEED/etc.

Vacuum systems: … procedures …

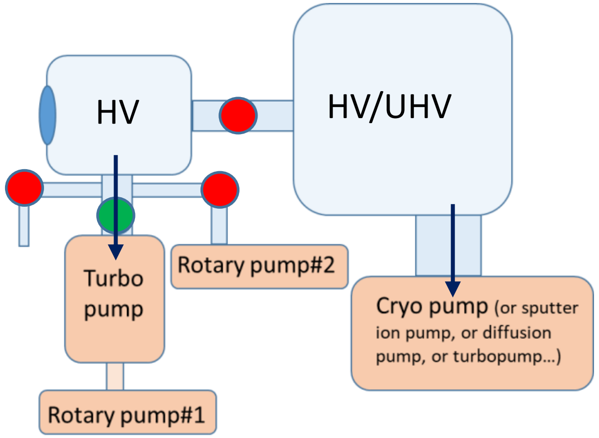
(i) Substrate insertion in IC



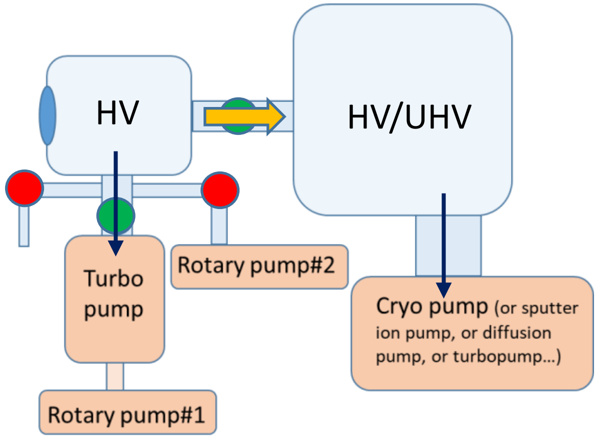
(ii) Medium vacuum in IC



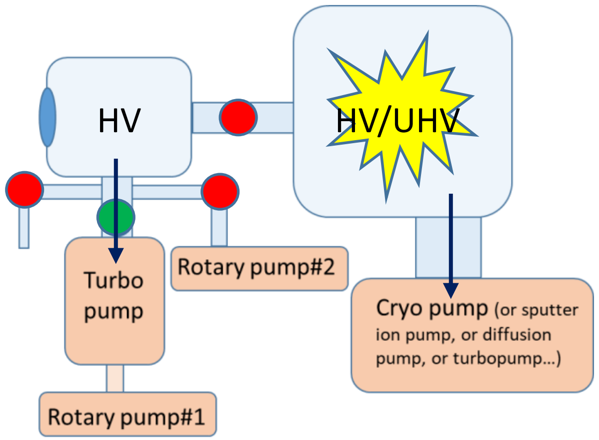
(iii) High vacuum in IC



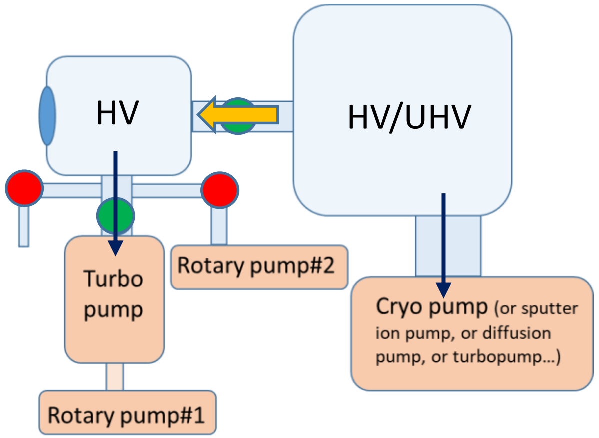
(iv) From IC to SPC



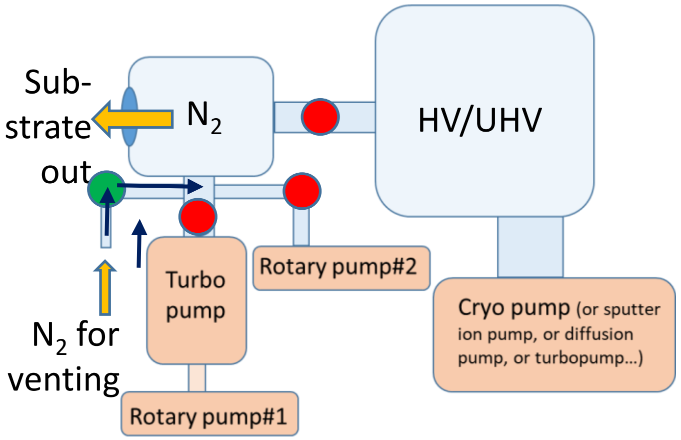
(v) Deposition in SPC



(vi) From SPC back to IC



(vii) IC venting and sample extraction

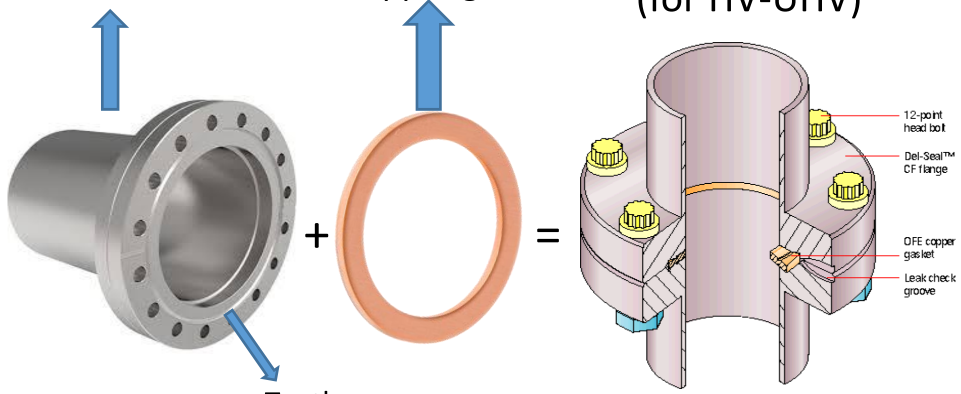


Vacuum systems: … components

Stainless steel flange

Copper gasket

(for HV-UHV)



Tooth

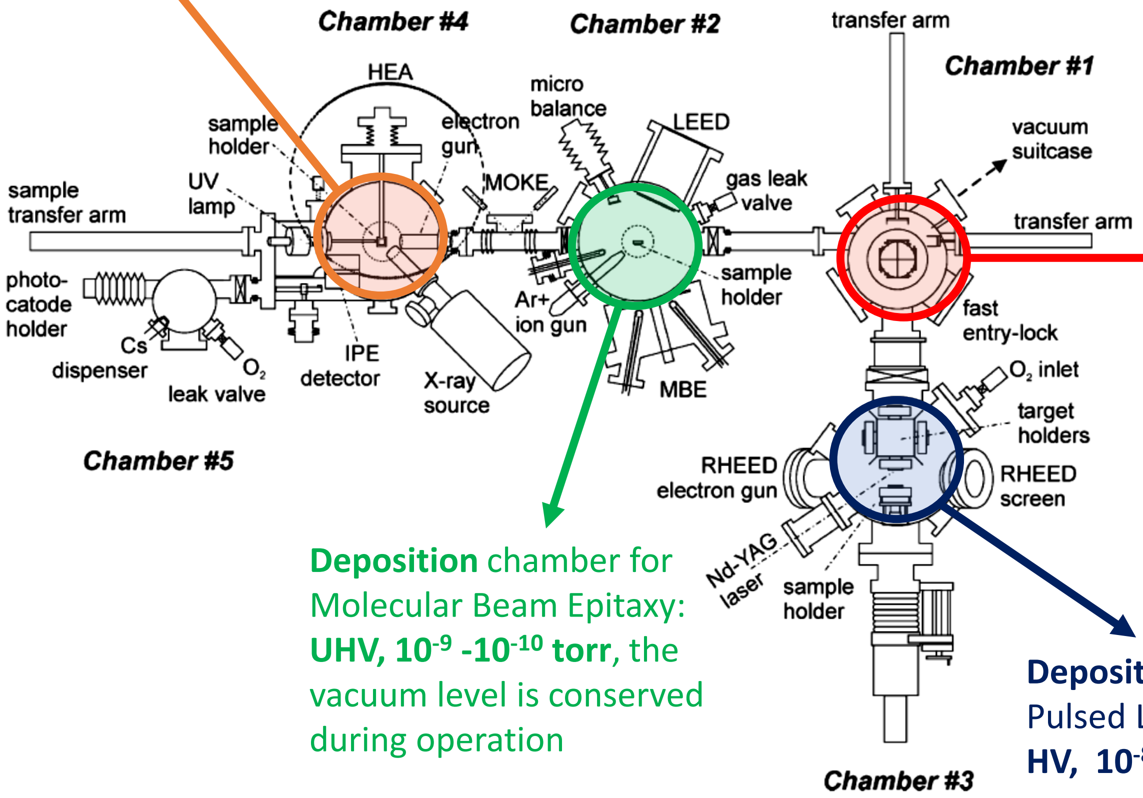
(for HV, MV …)

- Different vacuum levels (UHV, HV, low vacuum)
- Different requirements (fast venting/pumping, vacuum preservation)

Vacuum systems: an example

Vacuum is basically needed for
- **deposition**
- characterization

In-situ **characterization** chamber (XPS): **UHV, 10^{-10} torr** range, the vacuum level is conserved during operation



Fast entry lock (load lock, insertion chamber): for fast insertion/extraction of sample, from 760 to ~10^{-7} -10^{-8} torr in few minutes

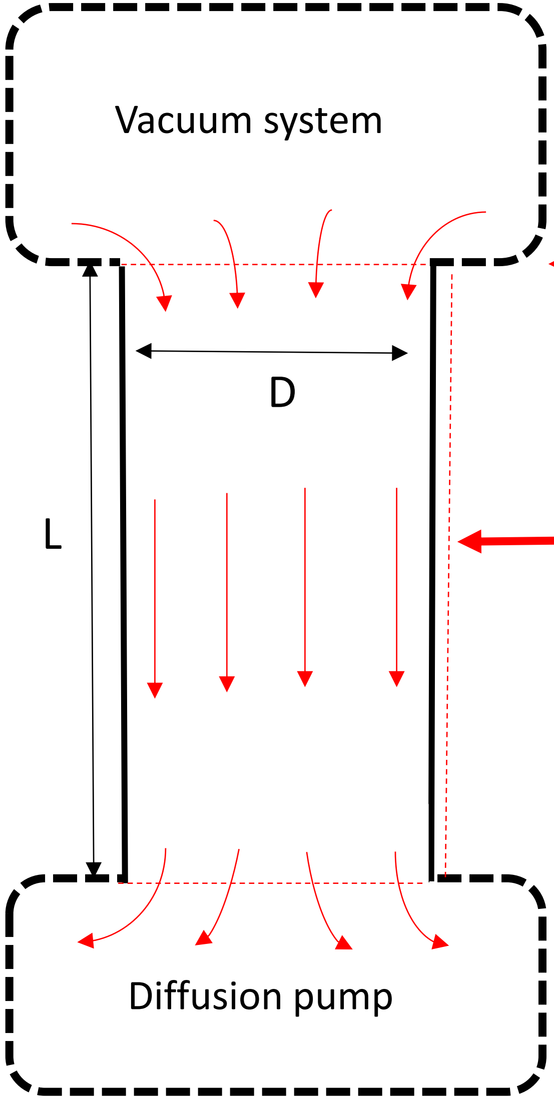
Deposition chamber for Molecular Beam Epitaxy: **UHV, 10^{-9} -10^{-10} torr**, the vacuum level is conserved during operation

Deposition chamber for Pulsed Laser Deposition: **HV, 10^{-8} torr** range base vacuum, up to torr range during operation

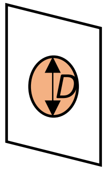
LAyered Structures for Spin Electronics (LASSE) @ Polifab

Appendix 1: example of conductance calculation (1)

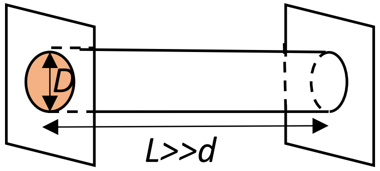
Example: cilindrical nipple connecting a vacuum system from a diffusion pump



$$C_1 = 11.7\pi \frac{D^2}{4} = 9.19 D^2$$



$$C_2 = 12.2 \frac{D^3}{L}$$



no obstacle to gas flux
(because there is no shrinking)

$$\left(C_3 \rightarrow \infty\right)$$

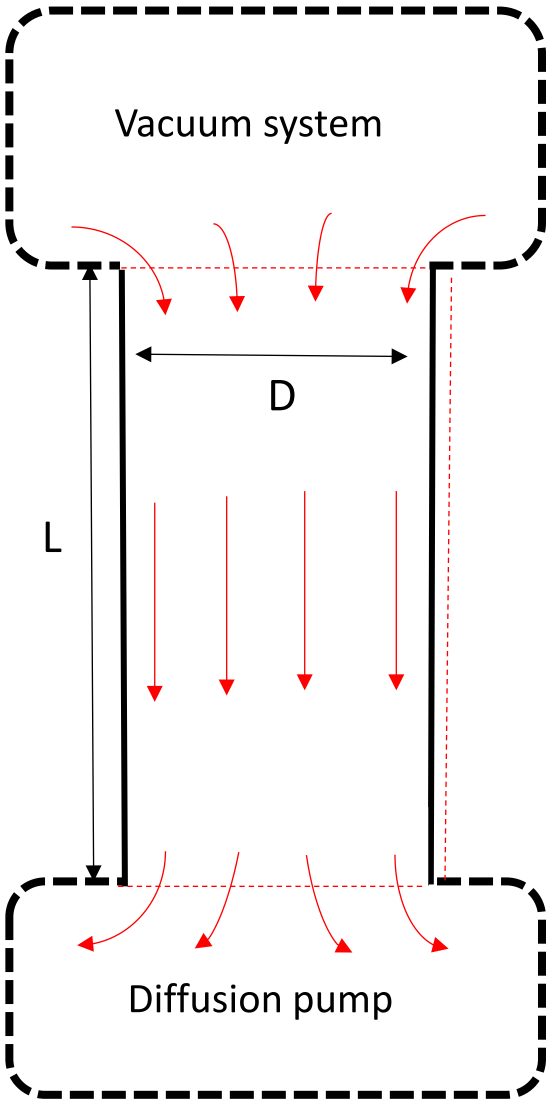
$$C = \left(\frac{1}{C_1} + \frac{1}{C_2}\right)^{-1} =$$

$$= \left(\frac{1}{9.19 D^2} + \frac{L}{12.2 D^3}\right)^{-1} =$$

$$= \frac{112.1\, D^2}{12.2 + 9.19\left(L / D\right)}$$

Appendix 1: example of conductance calculation (2)

Example: cilindrical nipple connecting a vacuum system from a diffusion pump



$$C_1 = 11.7\pi \frac{D^2}{4} = 9.19 D^2$$

$$C_2 = 12.2 \frac{D^3}{L}$$

$$\Rightarrow \quad C = \left(\frac{1}{C_1} + \frac{1}{C_2} \right)^{-1} = \frac{112.1\, D^2}{12.2 + 9.19 (L/D)}$$

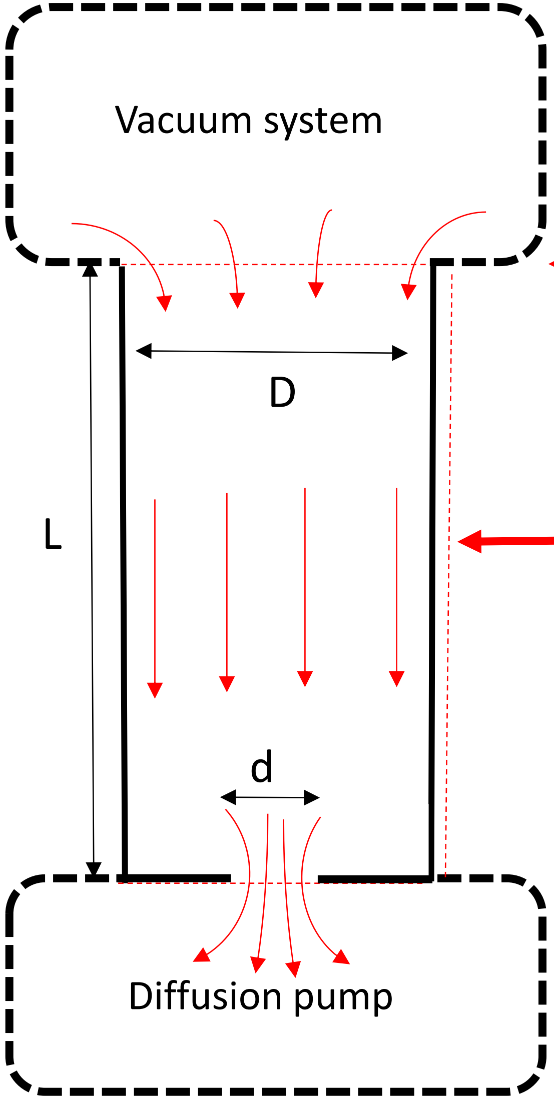
Note: if $D/L \to \infty$ we have $C_2 \to \infty$ and C is maximum (it is determined by C_1 only)

Ex.: D=10.16 cm (4 inches)
L=26.98 cm

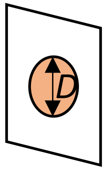
C_1=948.6 l/s
C_2=474.2 l/s $\Rightarrow$ C=316.1 l/s

Appendix 1: example of conductance calculation (3)

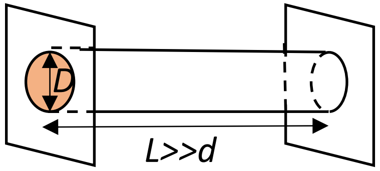
Example: cilindrical nipple ending with a shrinking ring



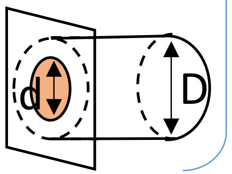
$$C_1 = 11.7\pi\frac{D^2}{4} = 9.19D^2$$



$$C_2 = 12.2\frac{D^3}{L}$$



$$C_3 = 11.7\frac{\pi d^2/4}{1-\dfrac{\pi d^2/4}{\pi D^2/4}} = 9.19\frac{D^2 d^2}{D^2 - d^2}$$



$$C = \left(\frac{1}{C_1} + \frac{1}{C_2} + \frac{1}{C_3}\right)^{-1}$$

Ex.: D=10.16 cm (4 inches)
d=5.08 cm (2 inches)
L=26.98 cm

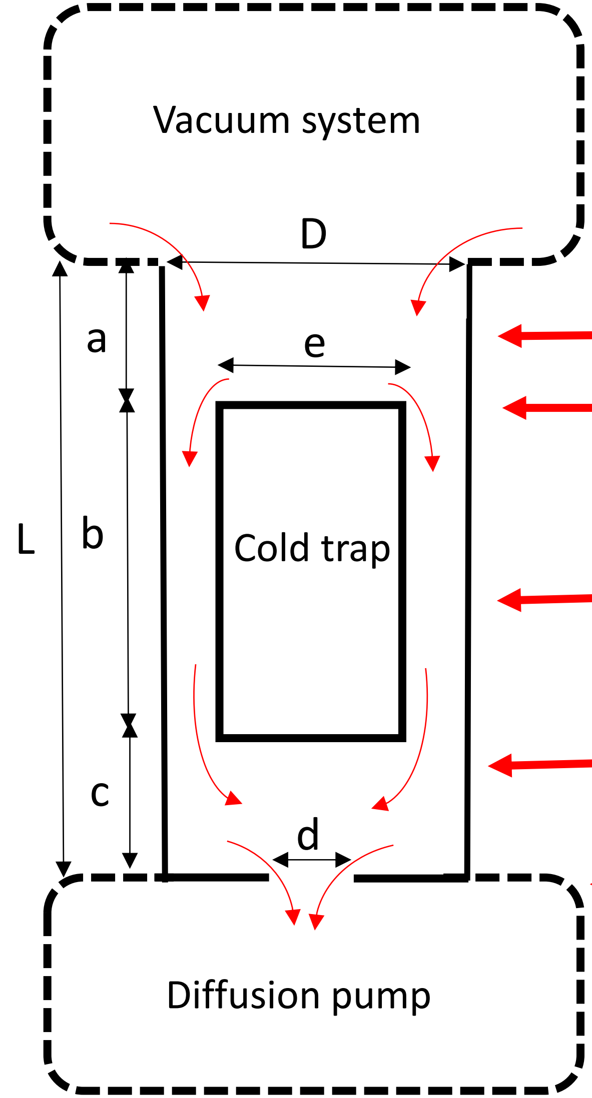
C_1=948.6 l/s
C_2=474.2 l/s
C_3=316.2 l/s

C=158.1 l/s

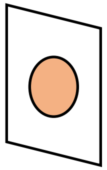
Note: if $d=D$ (that is there is no shrinking) we have $C_3 \to \infty$, as in the case before

Appendix 1: example of conductance calculation (4)

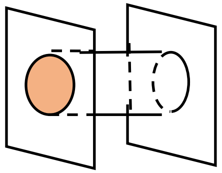
Example: cilindrical cold trap assembly which isolates a vacuum system from a diffusion pump



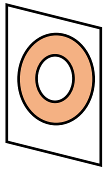
$$C_1 = 11.7\pi \frac{D^2}{4}$$



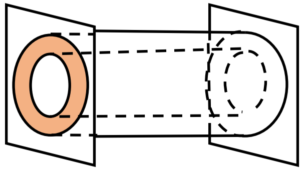
$$C_2 = 12.2 \frac{D^3}{a}$$



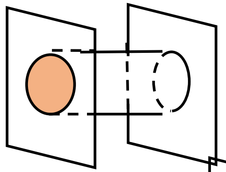
$$C_3 = 11.7\pi \frac{D^2 - e^2}{4}$$



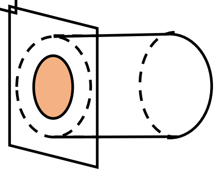
$$C_4 = 12.2 \frac{(D-e)^2 (D+e)}{b}$$



$$C_5 = 12.2 \frac{D^3}{c}$$



$$C_6 = 9.19 \frac{D^2 d^2}{D^2 - d^2}$$



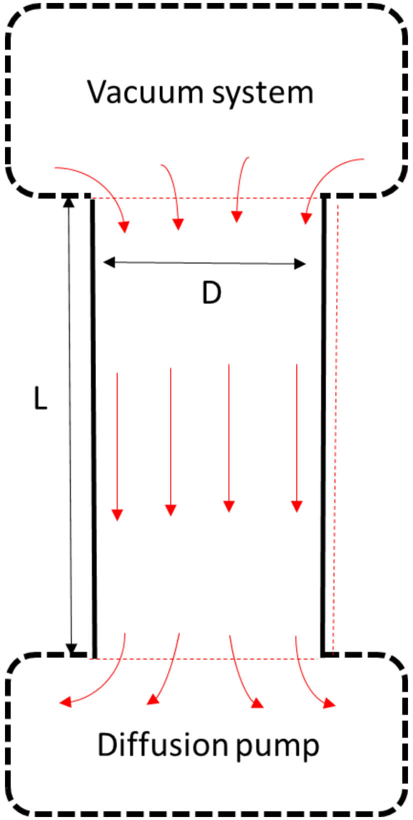
$$C = \left(\frac{1}{C_1} + \frac{1}{C_2} + \frac{1}{C_3} + \frac{1}{C_4} + \frac{1}{C_5} + \frac{1}{C_6} \right)^{-1}$$

Ex.: D=10.16 cm (4 inches)
d=5.08 cm (2 inches)
L=26.98 cm
a=c=5 cm
b=16.98 cm
e=8 cm

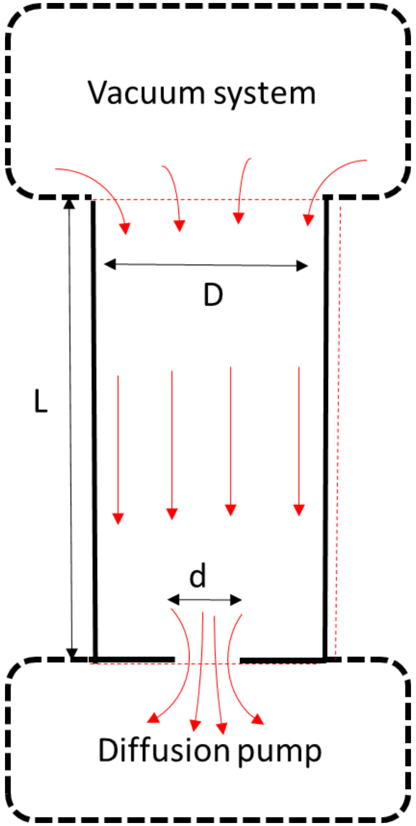
C_1=948.6 l/s
C_2=2559.0 l/s
C_3=360.4 l/s
C_4=60.88 l/s
C_5=2559.0 l/s
C_6=316.2 l/s

$\Rightarrow$ C=41.3 l/s

Appendix 1: example of conductance calculation (5)

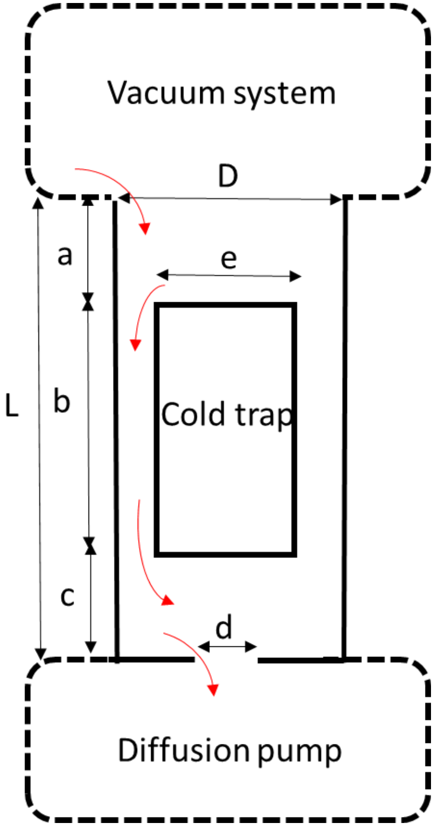


C=316.1 l/s



C=158.1 l/s

(the final shrinking ring, with C=316.2 l/s, reduces the overall conductance to 50%)



C=41.3 l/s

(the cold trap, with C=60.9 l/s, further reduces the overall conductance to about 26% of the previous case)

Appendix 2: pressure dynamics with pump outgassing (1)

- Real pumps **outgas** or release gas into the system. This can be accounted by introducing a second throughput term with opposite direction (***Q_P***).

- **Consider a pipe with $C\rightarrow\infty$**, so that $S=S_P \Rightarrow$ The equation for the throughput ($Q=SP=S_PP$ without outgas) becomes

$$Q = S_P P - Q_P = S_P P(1 - Q_P/S_P P)$$

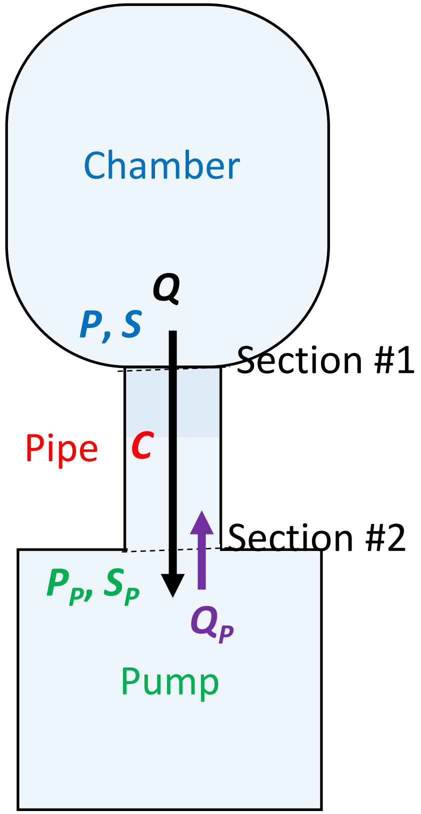
- When Q=0 (no net flow) the **ultimate pressure** of the pump (P_0) is reached: $1 - Q_P/S_P P_0 = 0$

$$\Rightarrow P_0 = Q_P/S_P$$

- The **effective pumping speed** is $S = \frac{Q}{P} = S_P(1 - Q_P/S_P P) = S_P\left(1 - \frac{P_0}{P}\right)$

$\Rightarrow$ S falls to zero when the ultimate pressure is reached ($P=P_0$)

$S_P = Q_P/P_0 \Rightarrow$ the pump intrinsic speed is used for pumping the outgas term



Appendix 2: pressure dynamics with pump outgassing (2)

- **Which is the time required to achieve a given pressure *P*?**

Dimensionally the throughput is $Q = PS = P\dfrac{V}{t} \Rightarrow$ it may be defined with differential notation as: $Q = \dfrac{-d(PV)}{dt} = -V\dfrac{dP}{dt}$

Note#1: *V* is the chamber volume and it is constant

Note#2: the – sign in the differential expression for *Q* can be justified as follows: a positive *Q* corresponds to flux of particles *from the chamber to the pump* $\Rightarrow$ in this case the density of particles in the chamber will *decrease* $\Rightarrow$ correspondingly the pressure *P* in the chamber will *decrease* too $\Rightarrow$ a positive *Q* will produce a *negative dP* $\Rightarrow$ $Q \propto -dP$

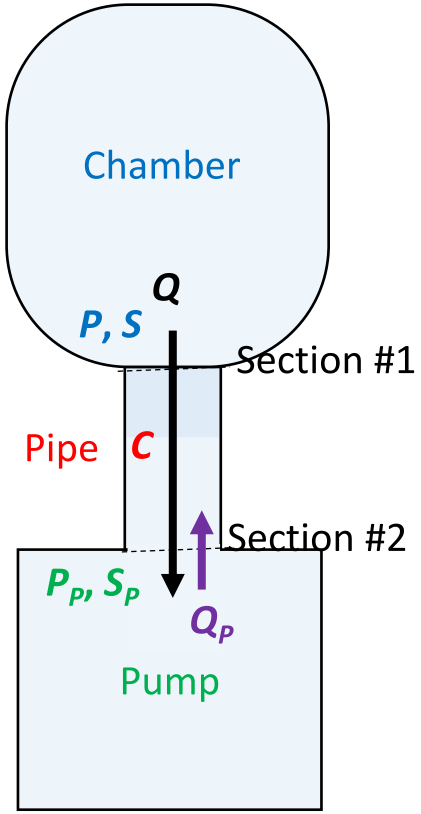
- The relation between the throughput and the pump characteristics leads to the differential equation:

$$Q = -V\frac{dP}{dt} = S_P P - Q_P$$ (assuming, as before, $C \rightarrow \infty$, so that $S = S_P$)

Solving it we obtain

$$-V\frac{dP}{dt} = S_P P - Q_P \Rightarrow \frac{dP}{dt} = -\frac{S_P}{V}P + \frac{Q_P}{V} = -\frac{S_P}{V}\left(P - \frac{Q_P}{S_P}\right) \Rightarrow \frac{dP}{P - P_0} = -\frac{S_P}{V}dt$$

$$\Rightarrow \int_{P_i}^{P_0}\frac{dP}{P - P_0} = \ln\frac{P - P_0}{P_i - P_0} = \int_0^t -\frac{S_P}{V}dt = -\frac{S_P}{V}t \Rightarrow \boxed{P(t) = P_0 + (P_i - P_0)e^{-\frac{S_P}{V}t}}$$



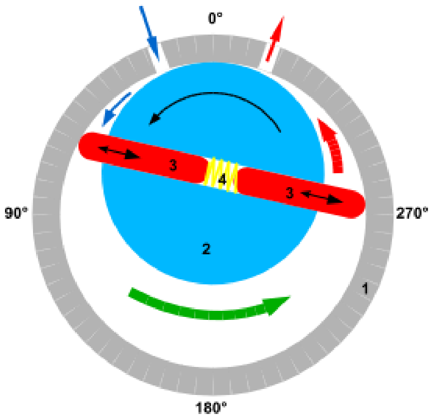
Appendix 3: Rotary Mechanical Pumps

- Rotary mechanical pumps are **gas-transfer** – **positive displacement** pumps.

$S_P = V_0 f_S$ where V_0 is the volume of gas enclosed between the rotor and stator, and swept into atmosphere after each revolution of the rotor, and f_S is the rotor rotation frequency.

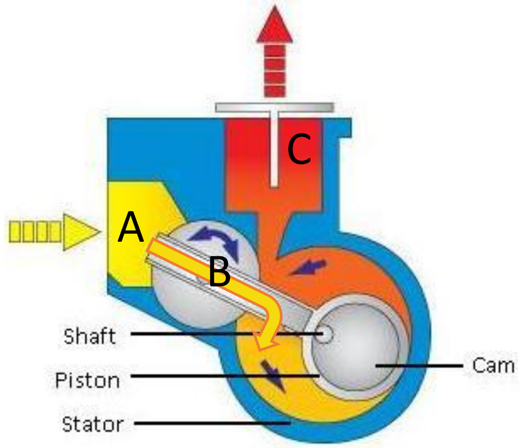
1) **Rotary-vane pump**: it contains an eccentrically mounted rotor (2) with spring-loaded (4) vanes (3). During rotation the vanes slide in and out within the stator (1), enabling a quantity of gas to be confined, compressed, and discharged through an exhaust valve to the atmosphere.

$$\boxed{S_P = 3 - 55\ \mathrm{l/s}}$$



2) **Rotary-piston pump**: gas is drawn into space B as the keyed shaft rotates the eccentric and the piston. There the gas is isolated from the inlet (A) after one revolution, then compressed and exhausted (C) during the next cycle.

$$\boxed{S_P = 8 - 420\ \mathrm{l/s}}$$



- **Oil** is employed in both pumps as a sealant as well as a lubricant between moving components.

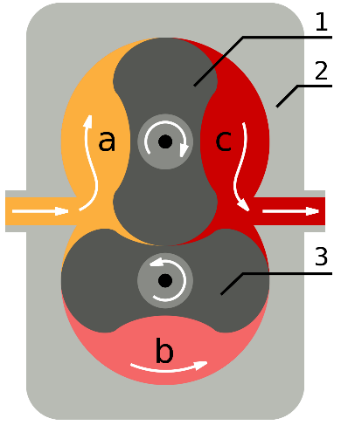
Oil traps are usually placed between the pump and the chamber to avoid oil diffusion to the chamber.

- Compression ratios **up to 10^6** can be achieved. Single-stage pumps can achieve an ultimate pressure P_0~10^{-2} torr, while two-stage can reach P_0~10^{-4} torr.
- Rotary pumps are frequently used to produce **pre-vacuum** to operate diffusion and turbomolecular pumps.

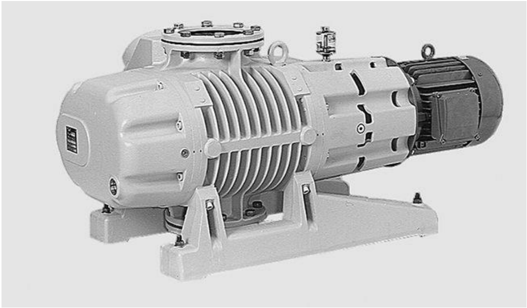
Appendix 3: Roots pumps

- Roots pumps are **gas-transfer** – **positive displacement** pumps.

Roots pump: two eight-shaped lobes (1 and 3) contained in the pump body (2) rotate in opposite directions relative to each other. Gas is entrapped in pockets (b) surrounding the lobes and carried from the intake (a) to the exhaust (c).



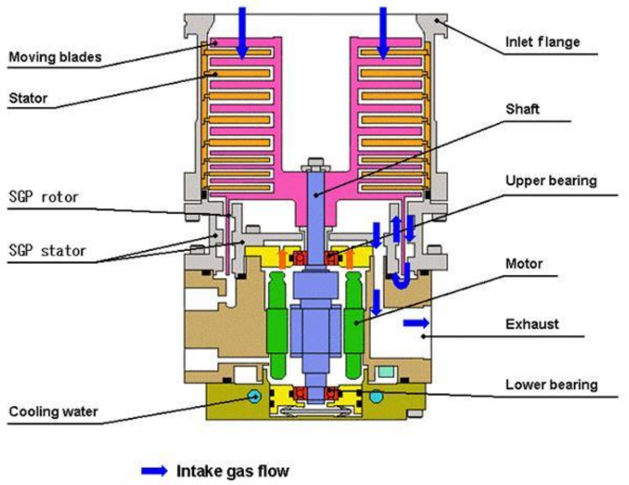
- **Oil** is not needed because of the extremely close tolerances.
- Roots pumps can achieve an ultimate pressure $\boldsymbol{P_0}$**~10^{-5} torr**.
- S_P is up to several **thousands of l/s** in the pressure range 10^{-3} – 20 torr.
- Roots pumps are frequently used in **sputtering** and Low-Pressure Chemical Vapour Deposition (**LPCVD**) because of large gas volumes moving at ~1 torr.



Appendix 4: Turbomolecular pumps

- Turbomolecular pumps are **gas-transfer** – **kinetic** pumps.

Turbomolecular pump: like in diffusion pumps, a preferred direction is imparted to molecular motion, the impulse being caused by impact with a rapidly whirling turbine rotor spinning at rates of 20 000-30 000 rpm. The pump is generally a vertical axial-flow compressor made by many rotor-stator pairs in series: gas captured by the upper stages is transferred to the lower stages where it is successively compressed until the level of the fore-vacuum pressure.

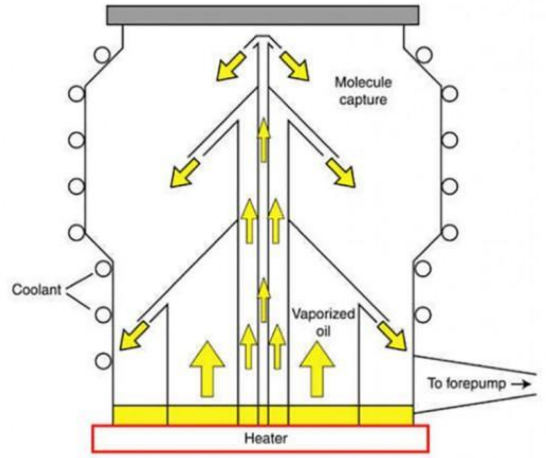


- Molecules hit by the rotor blades must reach the stator blades *before colliding* with other molecules on the way: the distance between the blades (~1 mm) must be less than the mean free path $\Rightarrow$ **pressure must be lower than ~ 5·10^{-2} torr**, thus **a mechanical forepump is required at the outlet**.
- The compression ratio r is proportional to $\exp\left(v/\bar{v}\right) \propto \exp\left(\omega R\sqrt{M}\right)$, where $v=\omega R$ is the circumferential rotor speed and M is the molecular weight of the gas $\Rightarrow$ r ~ 10^{10}, 10^{9}, 10^{3} for hydrocarbons, N_2 and H_2, respectively.
- **S_P is around 10^3 l/s** with ultimate pressure *below~10^{-10} torr*.
- Thanks to the very high compression ratio, oil backstreming is reduced to negligible levels $\Rightarrow$ turbomolecular pumps can be considered **oil-free**. They are used in **thin-film deposition** and **characterization** equipments.

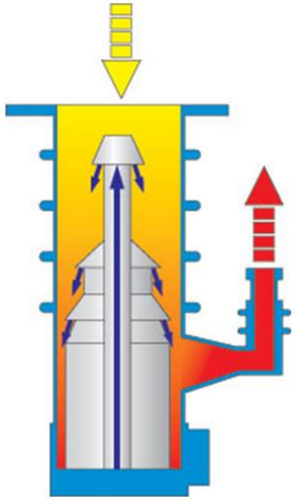
Appendix 5: Diffusion pumps

- Diffusion pumps are **gas-transfer** – **kinetic** pumps.

Diffusion pump: a fluid medium with low vapor pressure (typically a silicone oil) is boiled and vaporized in a multistage jet assembly. As the oil vapor stream emerges from top nozzles, it collides with residual gas molecules in the chamber and imparts momentum to them. These molecules are thus driven towards the bottom of the pump and thus compressed to the exit side, where they are exhausted. A region of reduced gas pressure in the vicinity of the jet is produced and molecules from the high-vacuum side of the pump move in this zone, where the process is repeated.



- In contrast to mechanical pumps, diffusion pumps have no moving part.
- Diffusion pumps operate in the molecular flow regime with pressure **below ~ $5 \cdot 10^{-2}$ torr**.
- Because they cannot discharge directly in the air ($P \leq 5 \cdot 10^{-2}$ torr), a **mechanical forepump is required** at the outlet.
- ***S_P* is up to $2 \cdot 10^4$ l/s** in the pressure range **$5 \cdot 10^{-2}$ to ~10^{-10} torr**.
- Backstreming of oil into the chamber is a serious problem (in particular at low pressure, where it depends on the vapor pressure of the oil), leading to substrate and materials contamination. Diffusion pumps are not suitable for UHV applications (deposition, spectroscopy, …) and in presence of hot filaments, whereas they are used for **nonelectronic coating deposition**.
- A cold cap on the uppermost jet, refrigerated traps and/or optically dense baffles help in condensing oil before entering the vacuum chamber, thus reducing backstreaming.



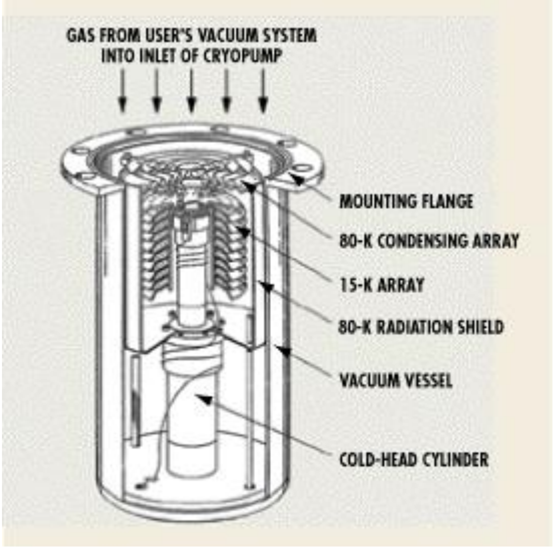
Principle of Operation

External View

Appendix 6: Cryopumps

- Cryopumps are **gas-entrapment** pumps.

Cryopump: it relies on the condensation of vapor molecules on surfaces cooled below 120 K (typically untreated bare metal surfaces or microporous surfaces, e.g. zoelite, with very large area). Temperature-dependent van der Waals forces are responsible for physically binding or sorbing gas molecules.

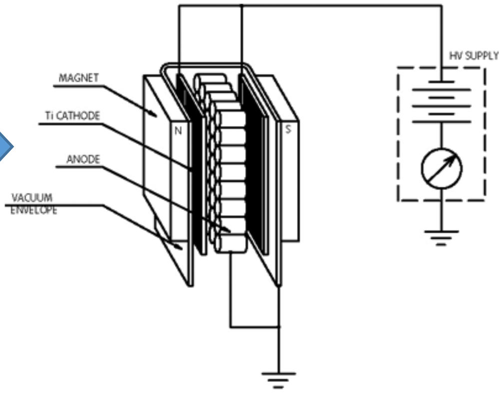


- Pressure **~10^{-3} torr** can be achieved cooling microporous surfaces by surrounding with a dewar of liquid ***N_2* (LNT)**.
- Pressure **~10^{-10} torr (UHV)** requires pumps with panels cooled to **20 K** (or below) by closed-circuit refrigerators.
  - These cryosurfaces cannot be directly exposed to RT because of the radiant heat load, so that they are surrounded by a LNT shroud.
  - An **initial forepressure ~10^{-3} torr** is needed to prevent from *excessive thermal load* on the refrigerant and the *accumulation of thick ice* condensate on the cryopanels.
- The ultimate pressure for a given gas is achieved when the impingement rate on the cryosurface at T_0 equals that on the vacuum chamber at 300 K: $P = P_{S0}\sqrt{300/T_0}$, where P_{S0} is the saturation pressure at T_0. Low P_{S0} gases (O_2, N_2) are efficiently pumped, high P_{S0} gases (H_2, He, Ne) are not.
- **S_P for air is ~ 3 l/s for each cm² of cooled surface (20K)**. Being limited only by gas impingement rate, cryopumps have the **highest pumping speed**, given by $S_P/A = 3.64\cdot 10^3\left(T_0/M\right)^{1/2}$

Appendix 7: Sputter ion, sublimation, getter pumps

- Sputter ion pumps, T sublimation pumps and getter pumps are **gas-entrapment** pumps.

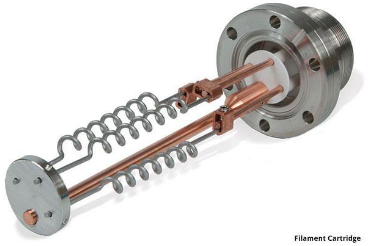
- **Sputter ion pump**: it relies on sorption processes initiated by ionized gas to achieve pumping.



STRUCTURE OF TRIODE SPUTTER ION PUMP

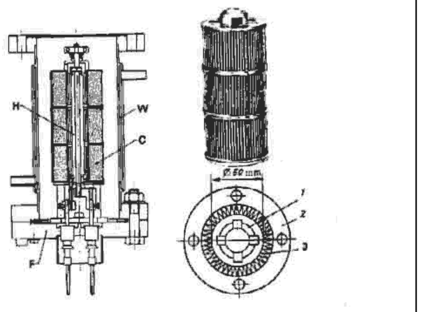
- Gas ions are generated in a cold-cathode electrical discharge between titanium cathode (negative or ground potential) and stainless steel anode (positive, ΔV~few kV). Electrons emitted from the cathode are trapped by a transverse magnetic field (~few kG) forming an high-density cloud (~10^{10} electrons/cm^3).
- After impact ionization of residual gas molecules, the ions travel to the cathode where they knock out or sputter Ti atoms. The latter deposit elsewhere in the pump forming films that getter or combine with reactive gases (N_2, O_2, H_2), and are successively buried by fresh layers of sputtered metal.
- **Pumping action for H_2 is few times more efficient than N_2, O_2, H_2O** and hundred times than Ar.
- Gas are permanently removed (in cryopumps they are not) but **the lifetime is inversely proportional to the pressure**.
- They are used in **oilless UHV applications**.

- **Ti sublimation pump**: Ti metal is thermally evaporated (sublimed) by periodic current (~40 A), performing chemical sorption as above. Eventually, cryogenically cooled surfaces are used, performing a combination of cryopumping and chemical sorption.


Filament Cartridge

- **Getter pump**: it is based on a non-evaporable (solid) getter (NEG) material (typically zirconium). When gas molecules strike the NEG, they combine with it chemically or by absorption, removing small amount of gases from the evacuated space.

- Flashed getters are placed in a reservoir and then evaporated in the vacuum chamber, e.g. for **pumping low power vacuum tubes** (lamps, thermionic valves…)
- Initial forepressures **~10^{-6} torr or lower** are needed for all these pumps.


The view of a nonevaporated getter pump. Left, cross section schematic; right, corrugated strip design. H and 1, electric heater; W, water cooling (for large pumps); C and 3, the getter ribbon; 2, flange.

Appendix 8: Monitoring the vacuum environment

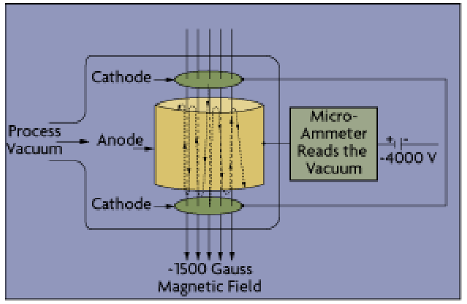
Direct pressure gauges

- They are **<u>absolute</u>** gauges, because any gas at the <u>same pressure</u> yields the <u>same signal</u>
- Working principle: deformation of a diaphragm between a reference and the chamber under test
- Reading mode (to convert the deformation into an electric signal): capacitance, piezoelectricity, resonance …
- Typical pressure range 1 atm - ~10^{-5} mbar

Indirect pressure gauges

- They measure properties (thermal conductivity, ionization, …) related to the pressure but also <u>dependent on the gas</u> $\Rightarrow$ ***not absolute***
- **Pirani** (1 atm-10^{-4} mbar): a metal filament is heated by a current and reaches a steady state temperature depending on the heat transport by the enviroment molecules $\Rightarrow$ filament resistance $\propto$ filament temperature $\propto$ gas pressure
- **Penning** (~ 10^{-2} mbar - 10^{-8} mbar): the gas is ionized by an high potential field and the ions are accelerated towards a collector anode, that measures the ion current $\Rightarrow$ ion current $\propto$ gas pressure
- **Baird-Alpert** (~ 10^{-4} mbar - 10^{-10} mbar): the gas is ionized by an electron current produced by a filament and the ions are accelerated towards a collector anode, that measures the ion current $\Rightarrow$ ion current $\propto$ gas pressure

Penning (cold cathode)



Baird-Alpert (hot cathode)