Chapter 1. Ultra High Vacuum and Sample Preparation

- ***Kinetic theory of gases***
- ***Vacuum concept***
- ***Pressure measurement***
- ***Vacuum pumps***
- ***UHV hardwares***
- ***Sample preparations***

Kinetic Theory of Gases

Number density of gases: ρ

ρ = number of molecules/volume = p/kT

= 9.66 x10^{18} p (torr)/T (molecules/cm^3)

eg) p=10^{-10}torr

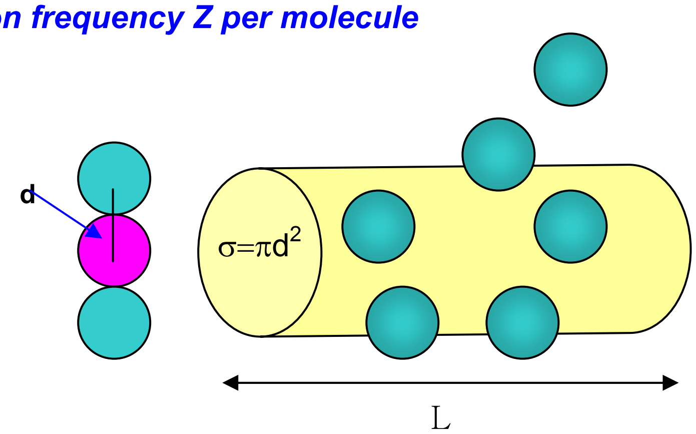
ρ = 3x10^6 molecules/cm^3

Pressure unit

1 Pa =1N/m^2 , 1bar = 10^5 Pa

1 atm = 1.013 bar = 760 mmHg = 760 torr = 101,325 Pa

Collision frequency Z per molecule



Length of cylinder L= $\langle v\rangle$t

The cross section area of the cylinder $\sigma= \pi d^2$

Total chamber volume = V

The number of collisions of a molecule in a chmaber V

= Nx(number of molecules in a cylinder)/V

= N x($\pi d^2\langle v\rangle$t)/V

= (N/V)(πd^2) $\langle v\rangle$t

= $\rho\sigma\langle v\rangle$t

The collision frequency = # of collisions per second

$Z= \sqrt{2}\rho\sigma\langle v\rangle$ $\langle v\rangle =(8kT/\pi M)^{1/2}$

Mean free path

The average distance travelled by a molecucle between collision

$\lambda = \langle v \rangle / Z = 1/\sqrt{2}\rho\sigma = kT/2p\sigma = \sim 1/p$

eg) at 10^{-10} torr $\lambda = 10^{6}$ m

Molecular flux on the surface F

The number of molecules striking the surface per unit area per unit time [molecules/m^2sec]

$F = (1/4)\,\rho \langle v \rangle = (1/4)(p/kT)(8kT/\pi M)^{1/2}$

$= p/\sqrt{2\pi mkT}$

eg) F of O^2 molecules at 298K and 10^{-6} torr

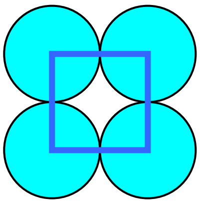
$F = 1.333\text{x}10\text{-}4\text{Pa}/(2\pi\text{x}(32\text{g/mol}/6.0\text{x}10^{23})\text{x}1.38\text{x}10^{-23}\text{J/Kx}298\text{K})$

$= 6.4\text{x}10^{18}$ molecules/m^2sec

$= 6.4\text{x}10^{14}$ molecules/cm^2 sec

$F \sim 10^{15}$ molecules/ cm^2 sec

The number of surface atoms per unit area for the cubic crystal



(a = 3.0A)

The unit cell has one atom, area = $(3\text{x}10^{-8}\text{cm})^2 = 9\text{x}10^{-16}\text{cm}^2 \sim 10^{-15}$ cm^2

Number of atoms per unit area (cm^2) = $1\text{cm}^2/10^{-15}\text{cm}^2$ $=10^{15}$atoms

1 Monolayer atoms(ML) = $1/(3\text{x}10^{-8}\text{cm})^2 \sim 1\text{x}10^{15}$ atoms/cm^2

At 10^{-6} torr, O^2 molecules will hit all the surface atoms once in 1sec

1 Langmuir (L) = 10^{-6} torr sec

How long does it take to get 1ML coveerage ?

Time = 10^{-6} torr sec/P(torr) if Sticking prob. =1

Eg) at $P=10^{-10}$ torr,

time to get 1ML = 10^{-6} torr sec/10^{-10}= 10^{4} sec = 2.8 hrs

Sticking coefficient (probability) S

A meaure of the fraction of incident molecules which adsorb upon surface

S = f(θ,T, crystal face, gas, substrate)

Surface coverage θ [molecules/cm²]

$$\theta = \frac{\text{Number of adsobed species per unit surface area}}{\text{Number of surface atoms per unit surface area}}$$

Pressure of Air at 25 degree *Torr*	Molecular Density *molec/cm³*	Molecular Incident Rate *molec/cm²-sec*	Mean Free Path *cm*	Time to Form a Monolayer *second* ($8x10^{14}$ *molec/cm²*)
760	$2.46x10^{19}$	$2.88x10^{23}$	$6.7x10^{-6}$	$2.9x10^{-9}$
1	$3.25x10^{16}$	$3.78x10^{20}$	$5.1x10^{-3}$	$2.2x10^{-6}$
10^{-3}	$3.25x10^{13}$	$3.78x10^{17}$	5.1	$2.2x10^{-3}$
10^{-6}	$3.25x10^{10}$	$3.78x10^{14}$	$5.1x10^{3}$	2.2
10^{-9}	$3.25x10^{7}$	$3.78x10^{11}$	$5.1x10^{6}$	$2.2x10^{3}$
10^{-12}	$3.25x10^{4}$	$3.78x10^{8}$	$5.1x10^{9}$	$2.2x10^{6}$
10^{-15}	3.25x10	$3.78x10^{5}$	$5.1x10^{12}$	$2.2x10^{9}$

Degree of Vacuum	Pressure Range (Pa) : *1 Pa = 7.5x10⁻³ Torr*
Low Vacuum (LV)	$3.3x10^{3} < P < 10^{5}$
Medium Vacuum	$10^{-1} < P < 3.3x10^{3}$
High Vacuum (HV)	$10^{-4} < P < 10^{-1}$
Very High Vacuum	$10^{-7} < P < 10^{-4}$
Ultrahigh Vacuum (UHV)	$10^{-10} < P < 10^{-7}$
Extremely High Vacuum (XHV)	$P < 10^{-10}$

Gas density is still high even in UHV conditions !!

Collision free conditions: mfp > diameter of chamber

$P < 10^{-4}$ torr

Conditions to keep clean surfaces **: time to reach 1ML contamination >> exp. Time**

$P < 10^{-9}$ torr !!

Vacuum Concept

Gas flow

Viscous flow : mfp < dim. of chamber
Molecular flow : mfp >> dim. of chamber

In the molecular flow, momentum transfer occurs *between molecules and the wall of a container,* but molecules seldom encounter one another

Pumping speed: S [m^3/sec, l/sec]

The volumetric rate at which a gas is transported across a plane

$S = Q/P$

Throughput: Q [Pa m^3/sec, J/sec, W]

The volume of gas at a known pressure and temperature that passes a plane in a known time

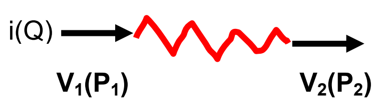
Q = Energy per unit time crossing a plane

= mass flow rate at constant T

Conductance [m3/sec]

$C = Q/\Delta P = Q/(P_1-P_2)$, $P_1 > P_2$

[compare with electrical conductivity $\sigma = 1/R = I/V$]



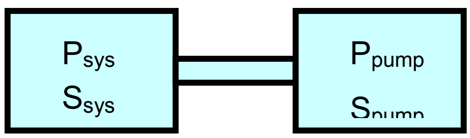
$$iR = \Delta V = (V_2-V_1)$$

$$C \leftrightarrow 1/R$$

$$i \leftrightarrow Q$$

$$\Delta V \leftrightarrow \Delta P$$

Measurement of system pressure



$Q = S_{sys}P_{sys} = S_{pump}P_{pump}$
$= C(P_{sys} - P_{pump})$

$P_{sys} = (1 + S_{pump}/C)P_{pump}$

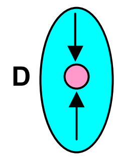
Effectve Pumping speed: S_{sys}

$1/S_{sys} = 1/S_{pump} + 1/C$

Conductance Formula

In the molecular flow region, the conductance is independent of pressure

1. For an aperture with the diameter D(cm)



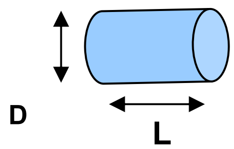
$C = 2.86\,(T/M)^{1/2}D^2$ l/sec

$= 9.33D^2$ for CO moelcules

T: temperature

M: molecular weight of gases

2. For a long pipe with the length L(cm)



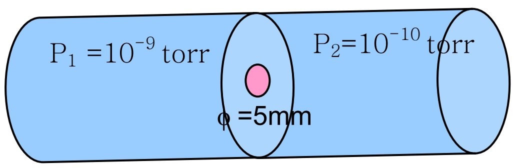
$C = 3.81(T/M)^{1/2}D^3/(L+1.33D)$ l/sec

The net conductance

Series $1/C_T = 1/C_1 + 1/C_2 + 1/C_3 \ldots\ldots$

Parlllel $C_T = C_1 + C_2 + C_3 + \ldots.$

(eg)



Find out the pumping speed S_p to maintain P_1 and P_2

$C\,(P_1 - P_2) = P_2S_p$

$S_p = C\,(P_1 - P_2)/P_2$

$= 2.33$ l/sec$(10^{-9} - 10^{-10})/10^{-10}$

$= 21$ l/sec

Pumping down time

- System volume: V,

- Pumping speed: S

- ***Throughput or leak rate: Q***

The pumpdown equation

PS = -Vdp/dt +Q

1. short time limit : Q~constant(small)

   $P + p_0 e^{-t/\tau}$, τ = V/S

2. long time limit: dp/dt ~ 0

   P =Q/S

Eg) Outgassing rate q=10^{-8} torr *l*/m^2sec

Chamber surface area A =$1m^2$

Q= qA =10^{-8} torr *l*/sec

P = Q/S =10^{-8} /S = 10^{-10} torr

∴ We need a pump with the pumping speed S =100 l/se

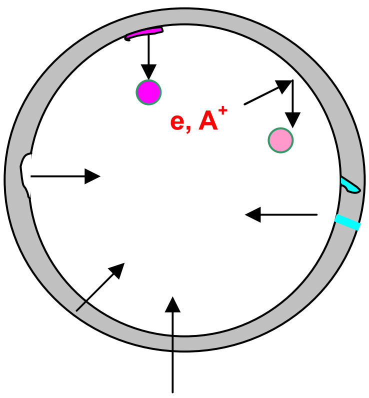
Out gassing rate : q

[1 Pa· m^3/s· m^2 ~ 10 torr L/s m^2 ~10^{-3}torrl/s cm^2]

Sources of Gas efflux from solids

1. vaporization
2. thermal desorption
3. diffusion
4. permeation
5. internal and external leak
6. electron and ion stimulated desorption

   ~10^{-2} atom per electron, ~10^{-5} ions per electron



Outgassing methods

Major gases: H_2,H_2O, CO, CO_2, N_2, O_2

vacuum firing

1. vacuum degreasing with trichloroethylene
2. electropolishing: q = 10^{-7} torrL/sm^2
3. Ar or O_2 flow discharge cleaning
4. oxidation in pure O_2 at 2700Pa at 400 C

Materials	**before baking**	**after baking q[torr L/s m^2]**
Stianless Steel	10^{-4}	10^{-8} ~10^{-10}
Viton O-ring	10^{-2}	10^{-6}

Recipe to achieve UHV (<10^{-10} torr)

1. proper pumping system: Turbo with TSP, Ion Pump with TSP
2. ***bake out*** for one-two days at 150~250C
3. ***degassing*** all filaments
4. otherwise, leak check

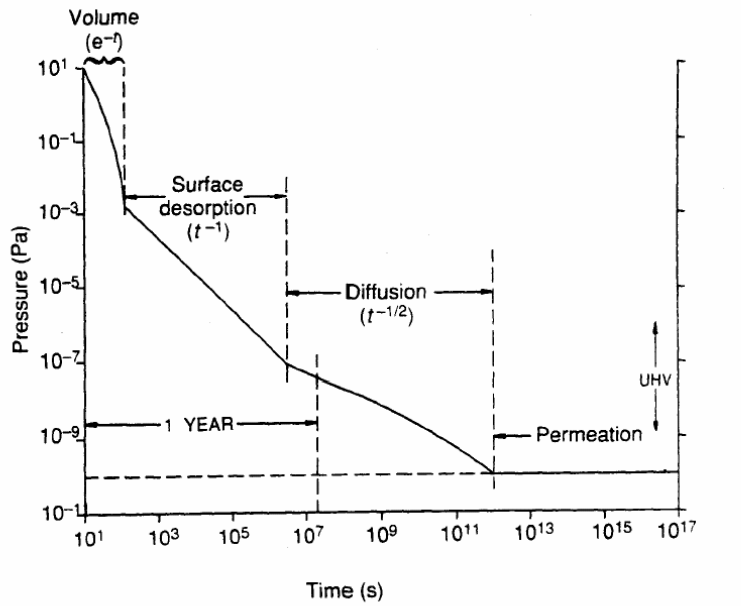
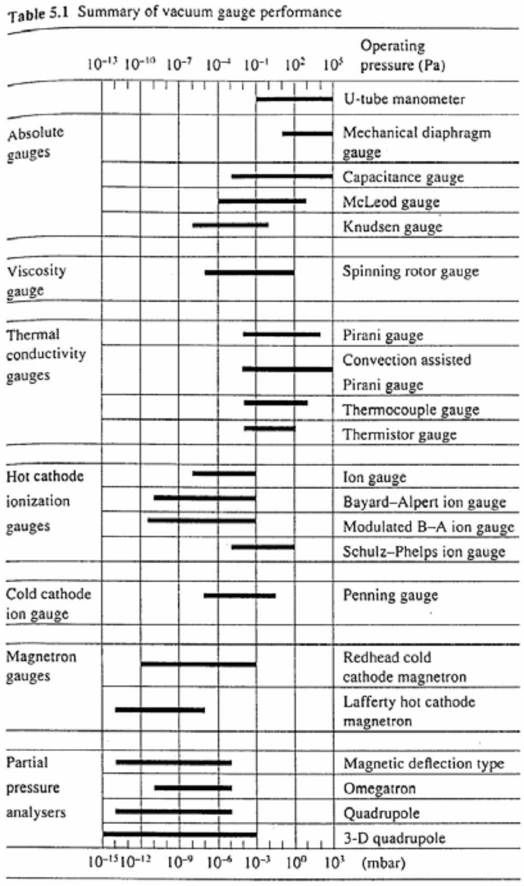


Fig. 6.5 Log–log plot of pressure versus time for a typical, unbaked vacuum system, showing the rate-limiting processes which determine the ultimate vacuum attainable. The principal component of the gas phase in the surface desorption region is likely to be water: in the lower pressure regimes it is likely to be hydrogen.

Pressure Measurment

Table 5.1 Summary of vacuum gauge performance



1. ***Thermocouple gauge***

   Measure temperature of the filament cooled due to gases

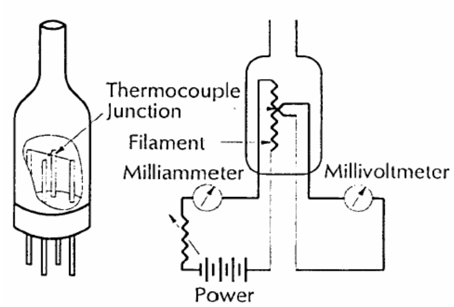
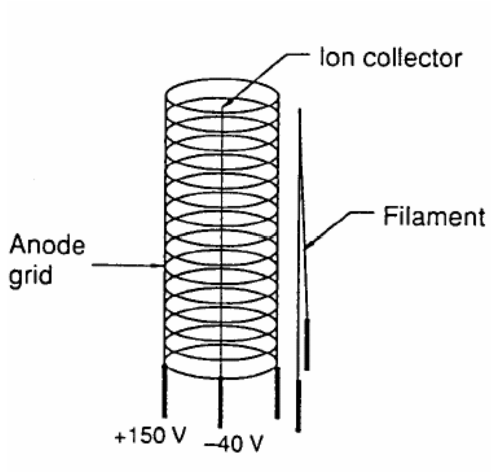


Fig. 3

Thermocouple Gauge

2. ***Ionization gauge***

   Thermionic electrons strike gases and generate ions



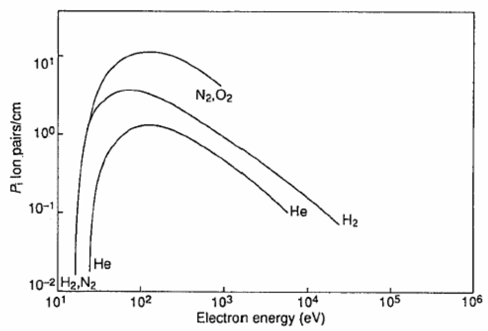


Fig. 5.13 Ionization probability versus electron energy for some common vacuum system gases.

3. Capacitance gauge

- Variation of capacitance
- sensitive, stable, accurate

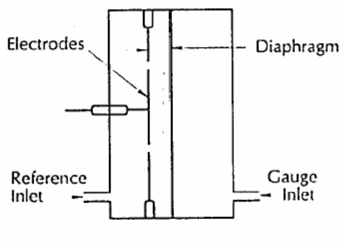


Fig. 5
Capacitance Manometer

4. Quadrupole mass spectrometer(QMA) or resdual gas analyzer (RQA)

- 10^{-4} ~10^{-14} torr
- partial pressure : residual gas
- very accurate, expensive

- low snesitivity for high mass

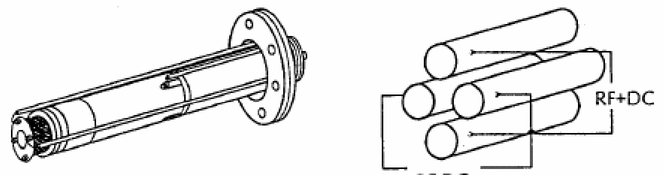
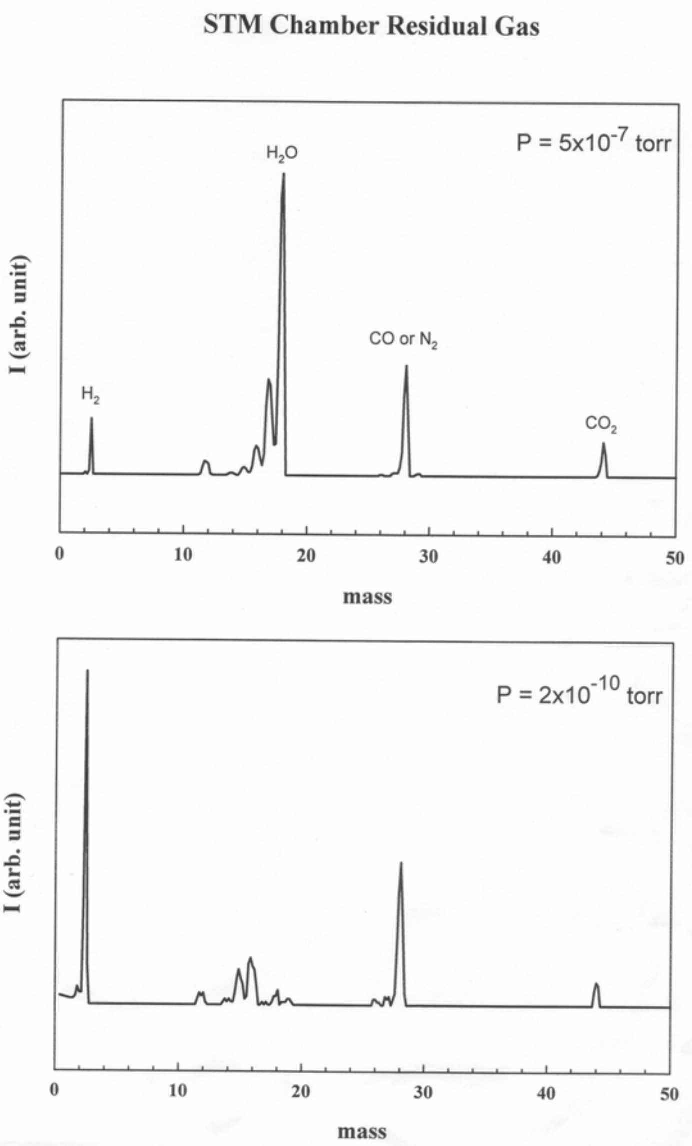
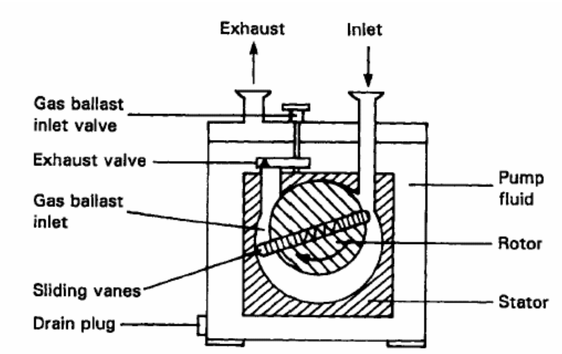


Fig. 8 Fig. 9



Vacuum pumps

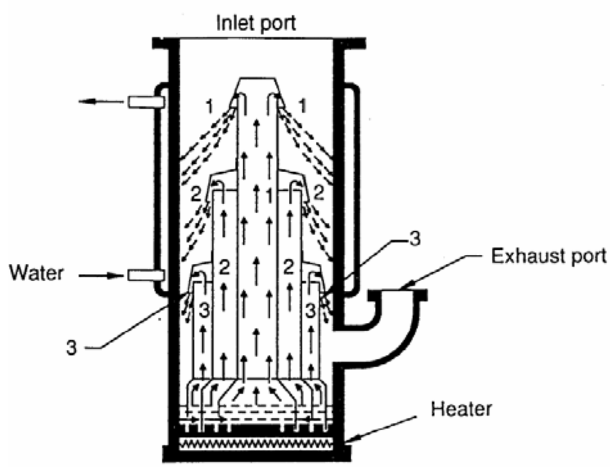
1. Rotary pump



- pumping speed: 1~500L/sec
- pressure 1atm ~10^{-3}torr
- belt-dirve or direct-drive
- vibration, not UHV compatible,oil contamination
- backing pump for ion, turbo, diffusion pumps

2. Diffusion pump

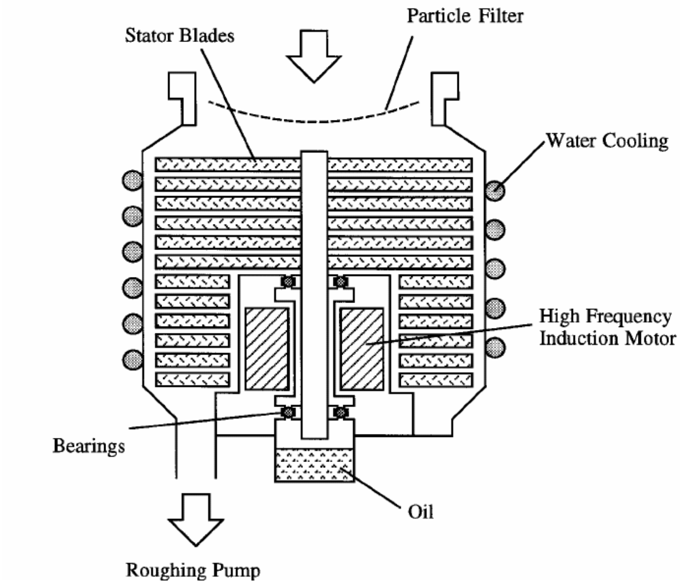
- oil spray changes the momentum of gases
- high pumping speed for light gases
- inexpensive, oil contamination
- 10^{-3}~10^{-9} torr, <10^{-10} torr with LN2 trap



3. Turbomolecular pump

- blades changes the direction of travel of gases
- high throughput

- oil, grease, magnetic bearing
- pressure 1atm ~10^{-10} torr
- expensive, poor pumping of light gases



4. Sputter-ion pump

- high E field ionizes gas molecules and B field causes spirled ions into walls and imbed themselves
- oil free, reliable, good for static vacuum
- pressure up to 10^{-11} torr
- poor pumping speed for noble gases

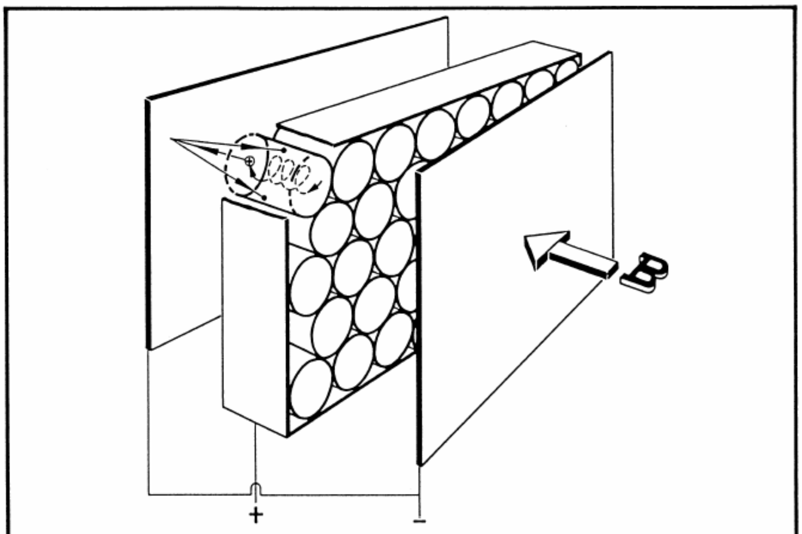


Fig. 29.1 Mode of action of the sputter-ion pump (diode type)

5. Ti-sublimation pump (TSP) or getter pump

- not primary pump
- Inexpensive, reliable

6. cryogenic pump

- **vacsorb pump**
  - molecular sieve, oil free
  - pressure 1atm~10^{-3} torr
- **cryo pump**
  - He cryostat
  - pressure ~10^{-13} torr
  -

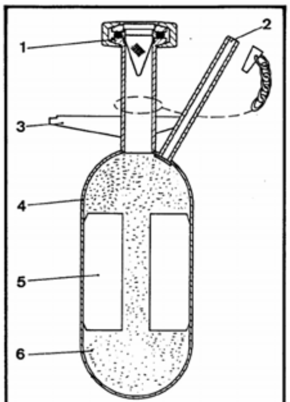


Fig. 28.1 Section through an adsorption pump
1 Inlet port; 2 Degassing port (safety outlet); 3 Support; 4 Pump body; 5 Thermal conducting vanes; 6 Adsorption material (e.g. Zeolite)

UHV Hardwares

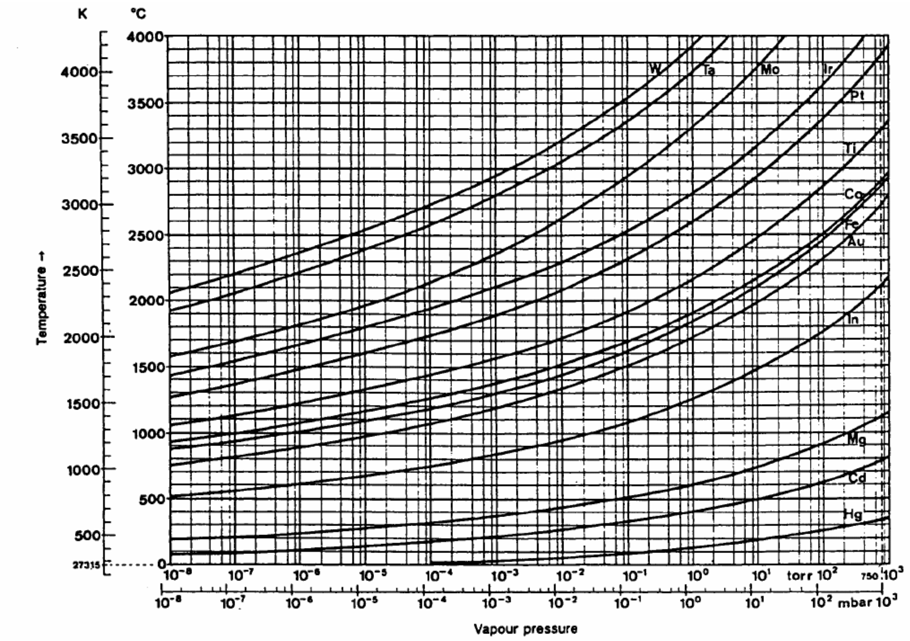
Materials

Conditions: low outgassing rate, weldability, chemical inertness, mechanical stability, thermal stability, conductivity

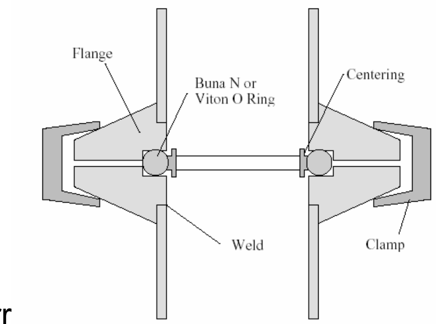
-metals: OFHC copper, SS316, SS304, Al alloy

-ceramics: quartz, alumina, mica

-Polymers: Viton, Teflon, High T plastics

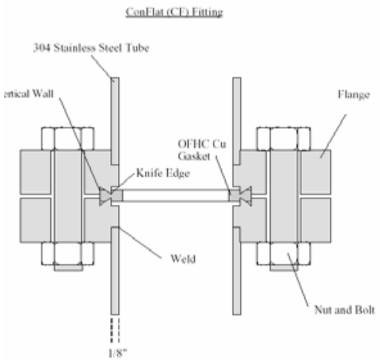


Vacuum connections



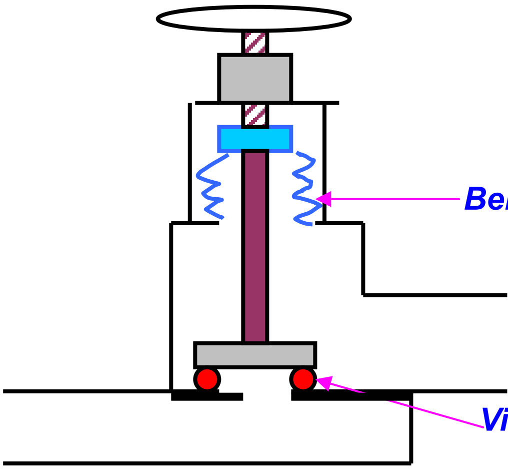
O-ring: up to 10^{-7} torr

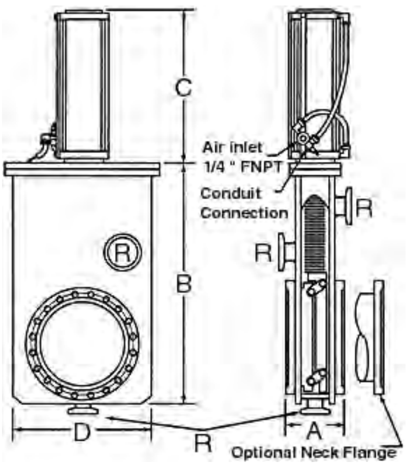
Metal sealed: Cu gasket, CF flange : ~10^{-13} torr



Valves

- gate valves: metal or O-ring sealed
- right angle valves

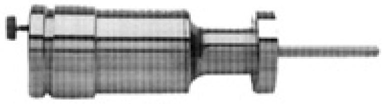


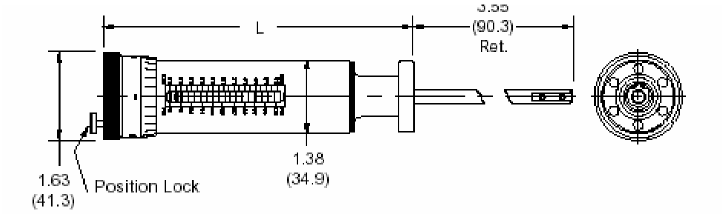


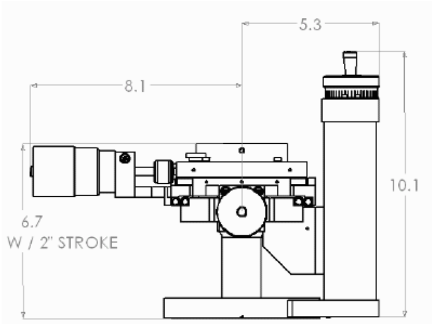
Mechanical motions in vacuum

- linear or rotary motion feed through

- Y, Z manipulator

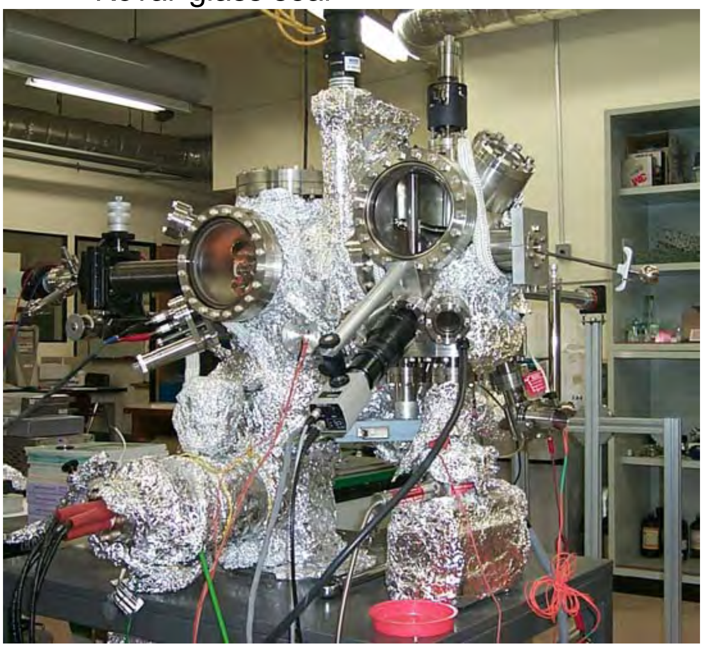


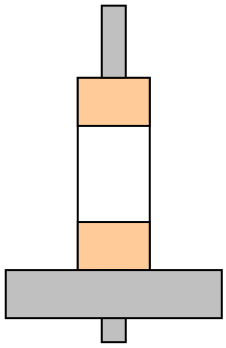




Electrical connection in vacuum

- ceramic to metal,
- Kovar-glass seal





Sample Preparations

Cleaning of UHV parts

- dirt removal
- degreasing
- ultrasonic cleaning with acetone and alcohol
- rinsing with alcohol

"More haste less speed"

How to get clean sample surfces

1. *cleaving*

- samples with natural cleavage planes: graphite, alkali halides, layered materials
- meta stable surfaces

2. *Heating*

- desorb wealkely bound surface species: water, CO, oxides, carbons
- temperature up to 50~80% of melting point
- cause surface contamination, segregation
- irreversible surface reconstructions

3. *ion sputtering*

- ions (Ar+, Ne+) with 1~5keV energy and 1~100 μA ion current
- sputtering yield varies for different samples

4. *chemical treatment*

- Carbide: heating in O_2
- Oxides: heating in H_2

5. ***Evaporated sample***

prepared by evaporation or sputtering of sample materials on the substrate

Metallic samples

- orientation with Laue diffractometer

ref: E.A. Wood, "*Crystal Orientation Manual*"

- cut the sample: wire saw, diamond saw, arc cutter

-polishing

-degreasing

-flashing: high mp metals, W, Mo,V

-Ar ion sputtering and annealing; soft metals, Cu, Ag, Au

Semiconductor sample

- Si

-Flashng to 1000C

-Sputter and annealing

- Ge

-light Ar ion sputtering and annealing

- Other samples: GaAs, CdTe …

-cleaving

-evaporation: molecular beam expitxay (**MBE**), chemical

-vapor deposition (**CVD**)

-sputtering and annealing: differential sputtering

80% of equipment failure is related to contamination !!