Vacuum Science in EM

Lecture 6

Outline

Review of general vacuum concepts

- Very brief!

Types of vacuum gauges used in TEMs

- Pirani
- Penning

Types of pumps used in TEMs

- Mechanical / Roughing pump
- Diffusion Pump
- Turbomolecular pumps - Turbo pumps
- Ion pumps
- Cold-traps

A typical vacuum system

Very brief vacuum overview

Why do we care in EM?

- Electrons scatter off of gases
- Gases contaminate samples

Pressure (P) units are a mess

- 1 Pascal (Pa) = $7.5 \bullet 10^{-3}$ Torr ($\approx 10^{-2}$ Torr) = 10^{2} mbar

Ranges (approx.)

- Low vacuum: 760 Torr > P > 1 Torr
- Medium vacuum: 1 Torr > P > 10^{-3} Torr
- High vacuum: 10^{-3} Torr > P > 10^{-8} Torr
- Ultrahigh vacuum: P < 10^{-9}
  - Surface science people will say it's not UHV until 10^{-10} Torr

Very brief vacuum overview

Kinetic theory assumptions:

- Large # of molecules
- Adjacent molecules separated by distances that are large compared to their diameter
- Molecules in constant motion
- Molecules exert no force on each other except during collision

Pressure

- Rate at which momentum is transferred to a surface

$$P = \frac{1}{3} n m v_{rms}^{2}$$

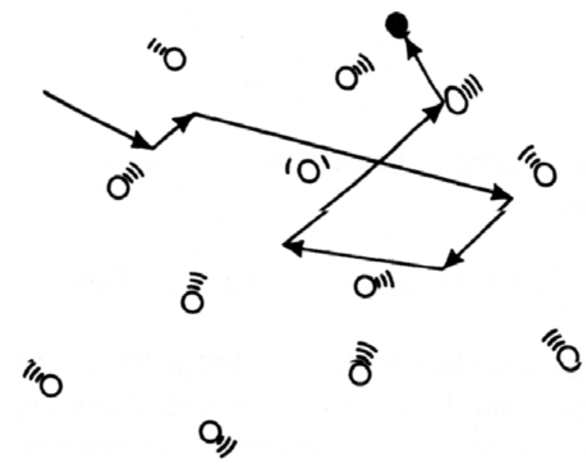
Very brief vacuum overview

Velocities follow a Maxwell-Boltzman distribution

$$v_{mean} = \left(\frac{8kT}{\pi m}\right)^{1/2} \qquad v_{rms} = \left(\frac{3kT}{m}\right)^{1/2}$$

Mean free path (λ)

$$\lambda = \left(\frac{1}{2^{1/2}\pi d_o^2 n}\right)^{1/2} = \frac{0.67}{P\ (Pa)} = \frac{0.005}{P\ (Torr)}$$



Particle flux:

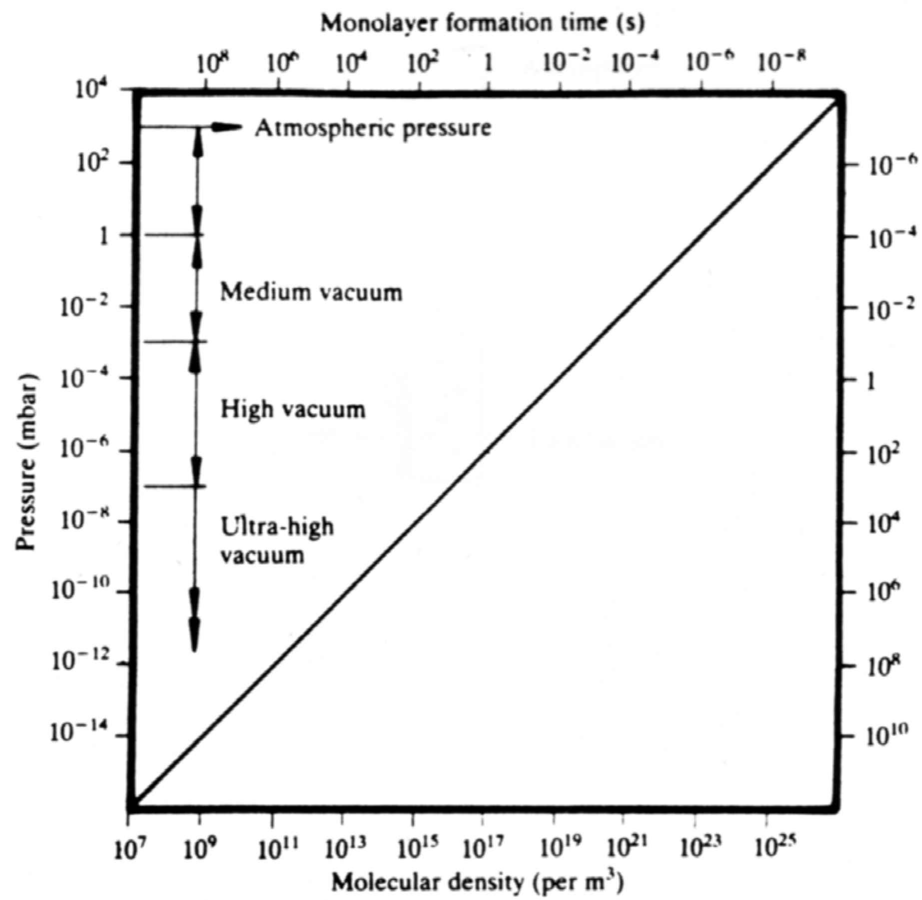
$$\Gamma = n\left(\frac{kT}{2\pi m}\right)^{1/2}$$

Very brief vacuum overview

Monolayer formation time:

$$t_{ml} = \frac{1}{\Gamma d_o^2} = \frac{4}{n v d_o^2}$$

Atmospheric	1 nanosec
10^{-3} Torr	1 millisec
10^{-6} Torr	1 sec
10^{-8} Torr	100 sec
10^{-10} Torr	3 hrs



Vacuum gauges

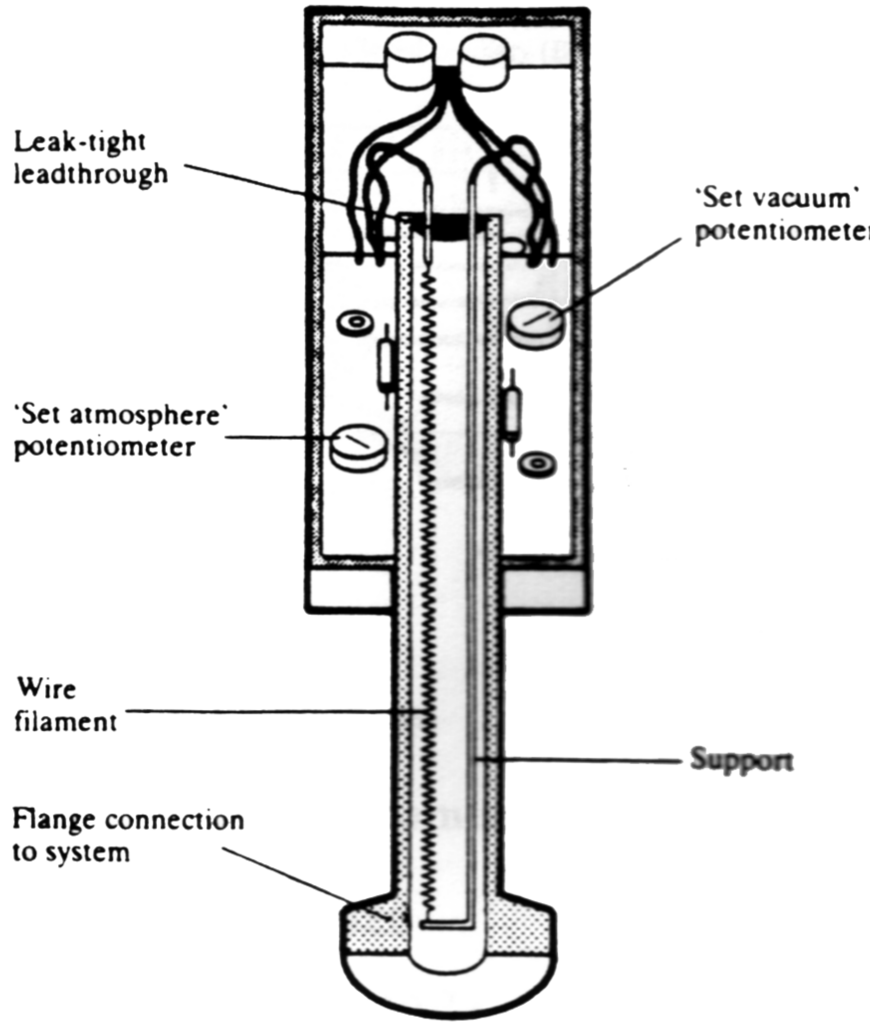
Pirani

Use a heated wire to form part of a Wheatstone bridge circuit

$\Delta P \Rightarrow \Delta T \Rightarrow \Delta R$

Range: 1000 to 10^{-4} Torr

Uncertainty in accuracy is $\pm 5\%$



Vacuum gauges
Ion gauges (hot cathode)

Force exerted by particles is too small to measure in high-vac & UHV conditions

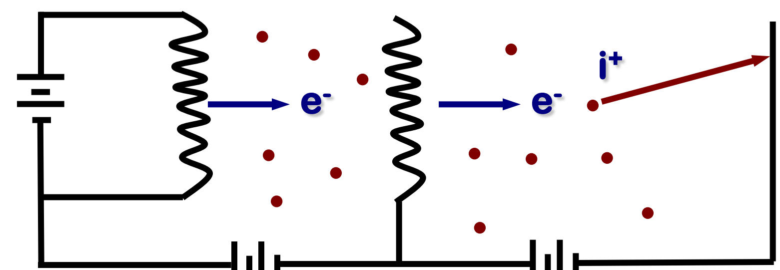
Indirect readings needed

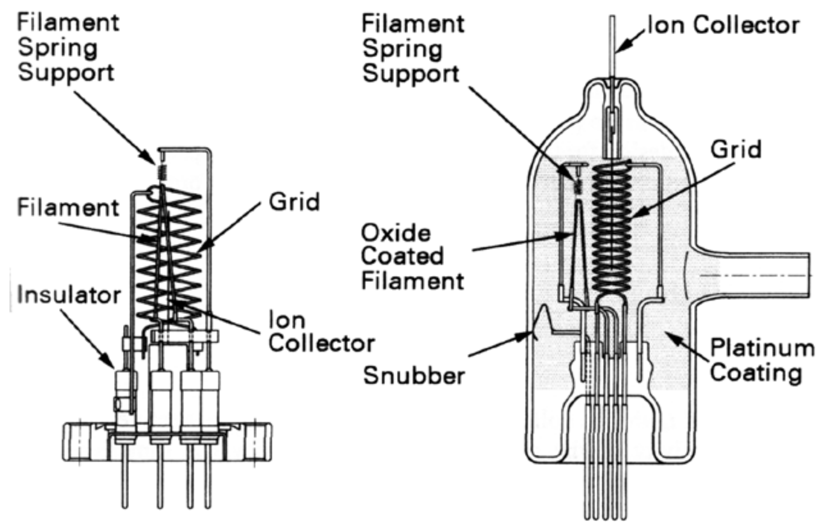
Operation

- Filament heated to produce e^{-}'s
- Accelerated to grid
- Ionize particles, which are subsequently collected

Range: 10^{-3} to 10^{-8}

Not particularly accurate





Hot cathode

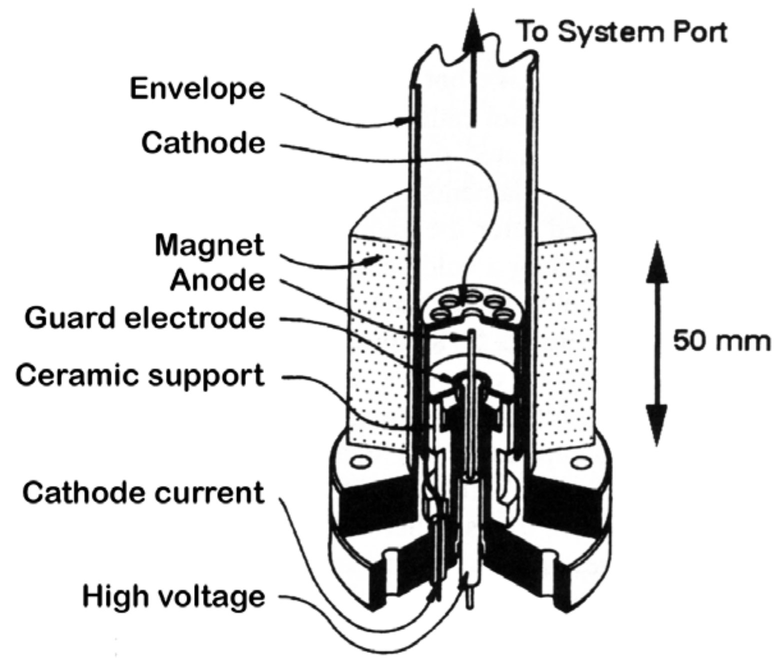
Vacuum gauges

Ion gauges (cold cathode - Penning)

Similar idea, but once e^{-}'s emitted, they are spun around in a magnetic field

Leads to increased sensitivity, longer paths

Range: 10^{-2} to 10^{-11} Torr

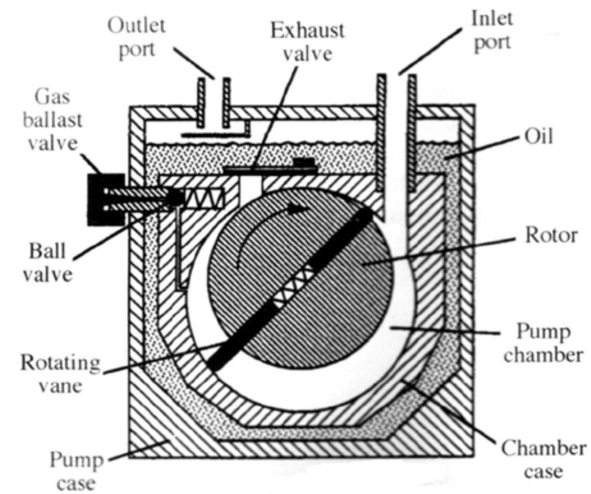


Cold cathode

“Mechanical” pumps

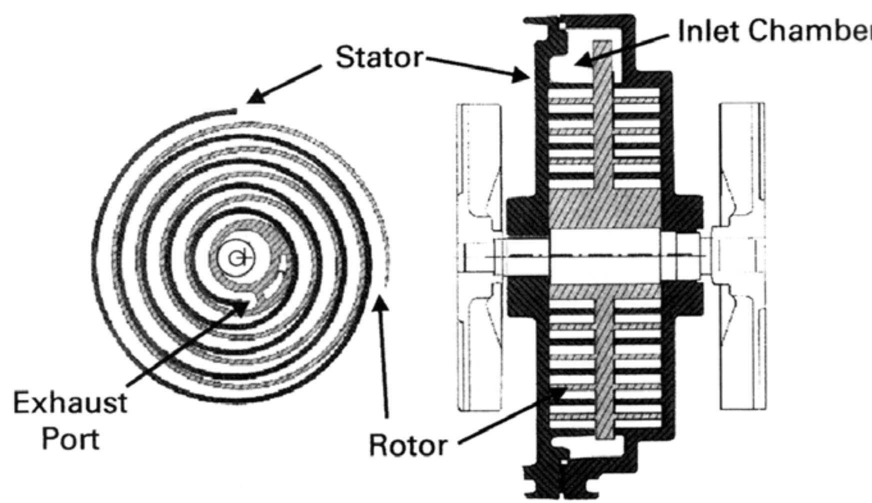
Rotary pump

- From ambient to 10^{-3} Torr
  - “Rough” or “roughing” pump
- Oil based
- Dirty, loud, lots of vibration



Scroll pump

- Oil free
- Like a ‘turbo pump’ (more later) but designed to cover the low vacuum range
- Cleaner, still has some vibration
- Becoming the standard



Diffusion pumps

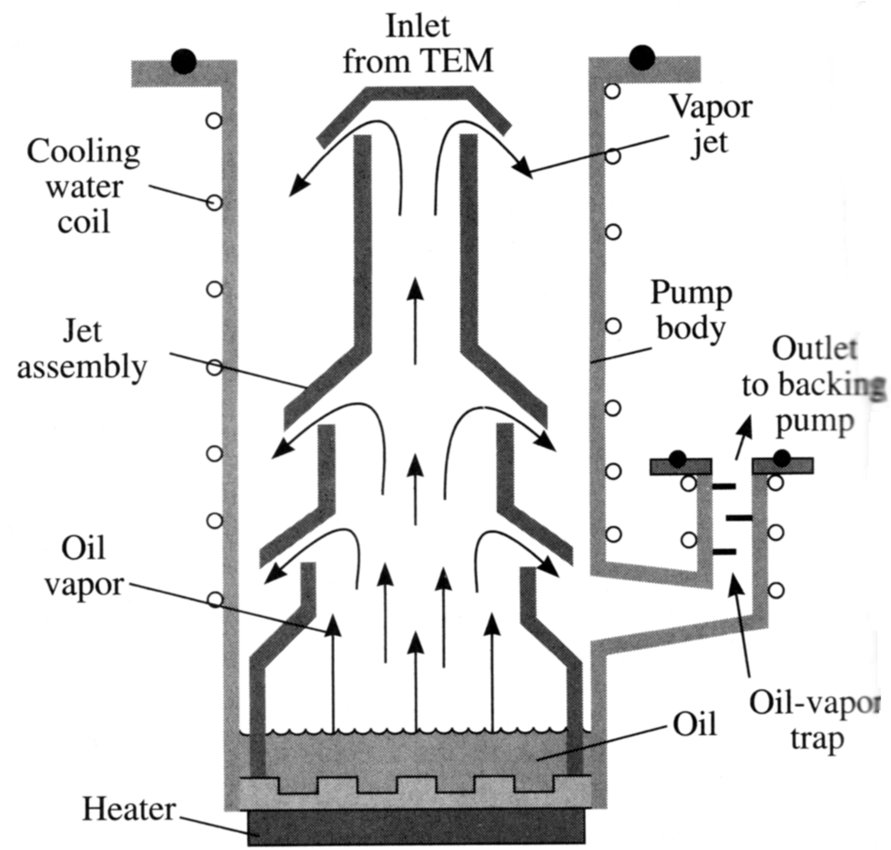
Hot oil used to trap gases

Very efficient

Vibration free

Range: capable of 10^{-3} to 10^{-11} Torr

- Remember, that is only if system can handle 10^{-11} Torr!
- Not used in UHV, though, for fear of back-streaming



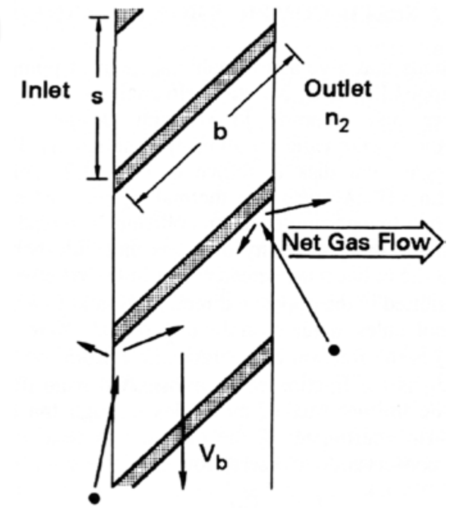
Turbomolecular pumps

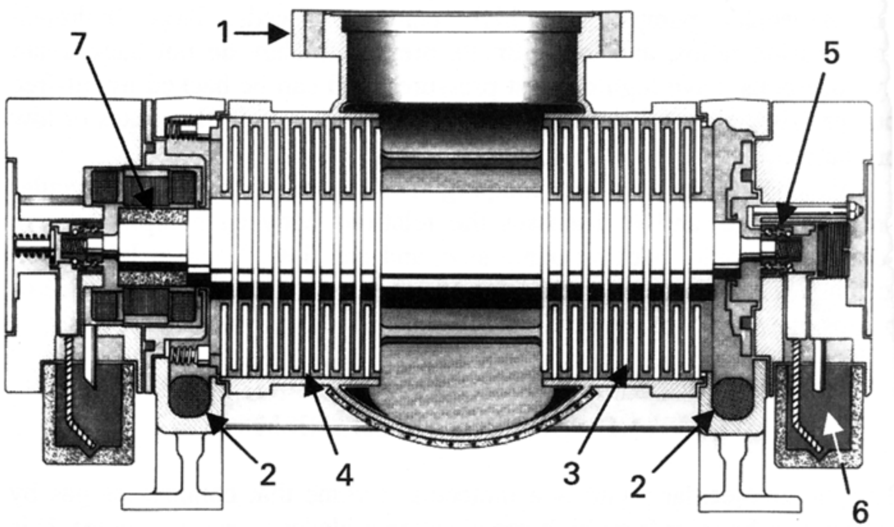
Rotary blade captures molecules, forces them out other side

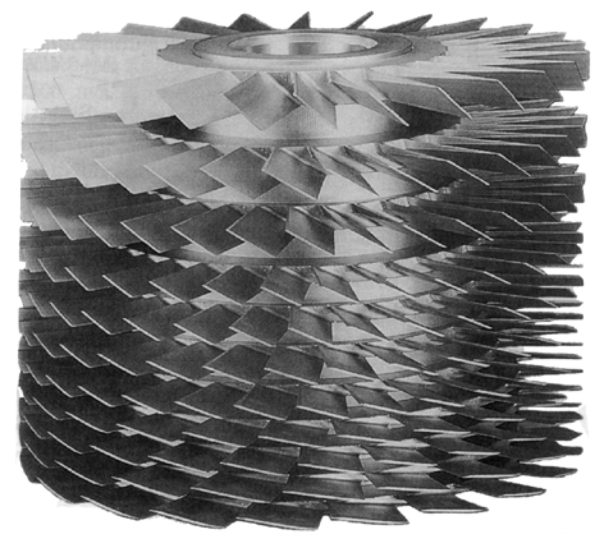
Can operate from ambient to 10^{-9} Torr

- In about an hour …
- Generally, you use a rough pump in conjunction with a turbo

Can introduce vibration







Ion pump

Electrons emitted from cathode

Spun around by a magnetic field

Incident gas is ionized

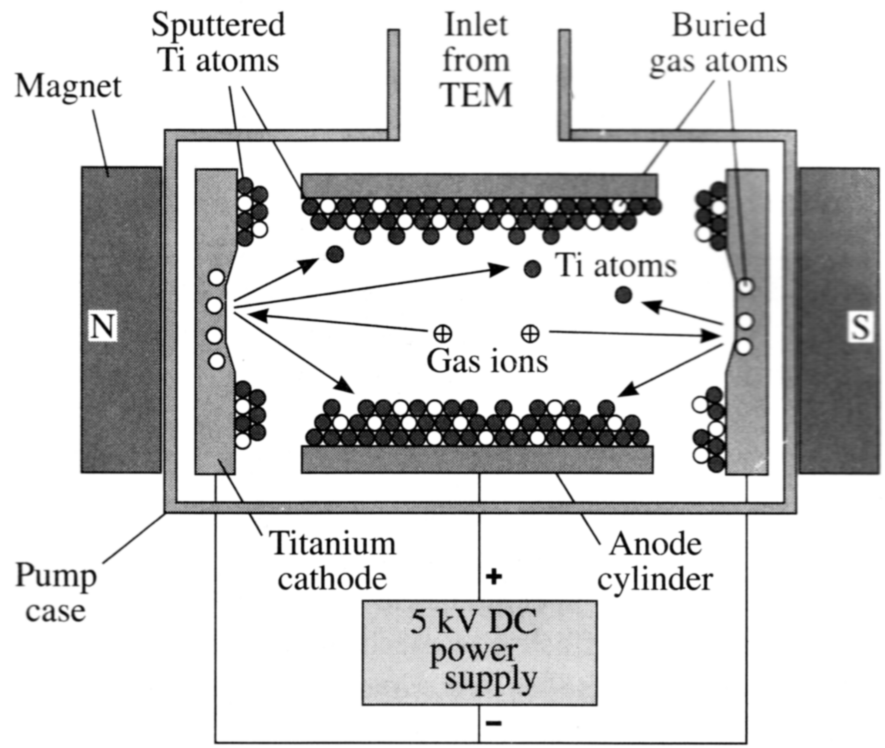
Ionized gas accelerated into pump walls, where it sticks

Can coat walls with Ti, to help that process

- Ti sublimation pump
- Ti coating renewed every day

Range: 10^{-5} to 10^{-11} Torr

No vibrations



Cryo Pumps / cold fingers

Cold surfaces attract & retain residual molecular species

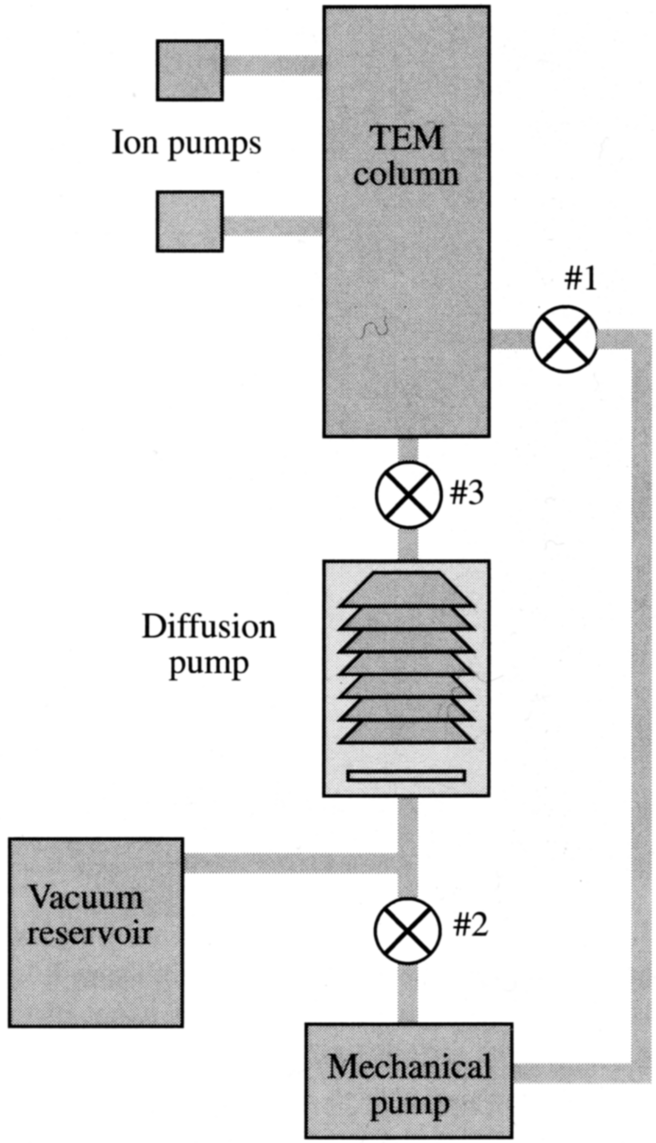
- Cryo-condensation
  - Molecules attracted to condensations sites on cold surface, as T decreases, their residence time there increases
- Cryo-sorption
  - Weak van der Waals attraction of molecules to surface
- Cryo-trapping
  - Trapping on one gas within the frozen porous condensate of another

Used in EM in two major ways

- "Cold fingers" or "anti-contamination devices"
- To help pumps (older microscopes)

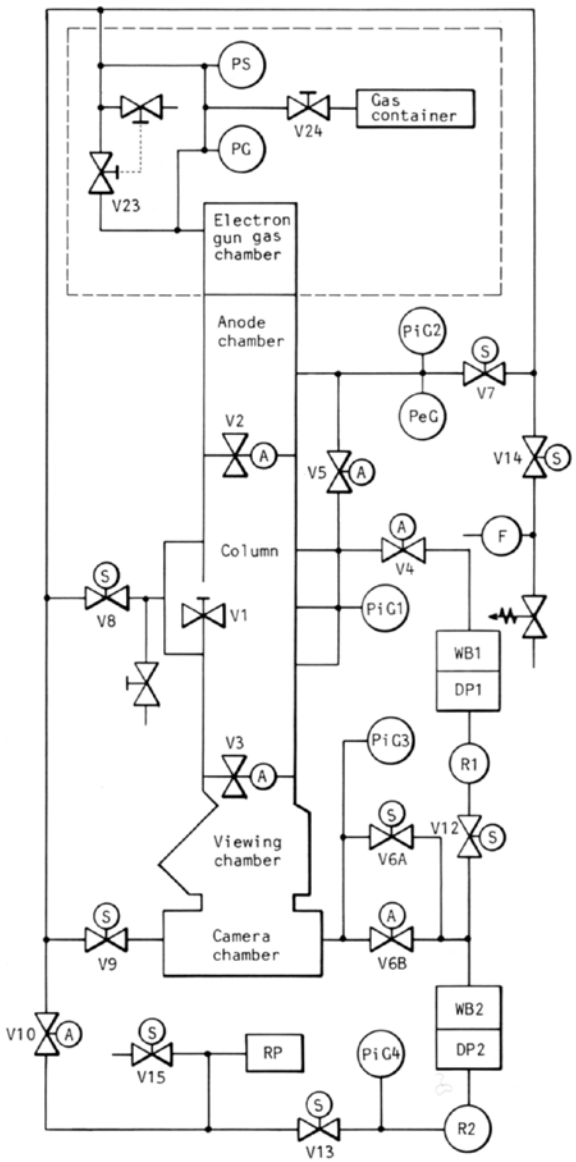
Residual contaminants lost when surface warms up

Vacuum system



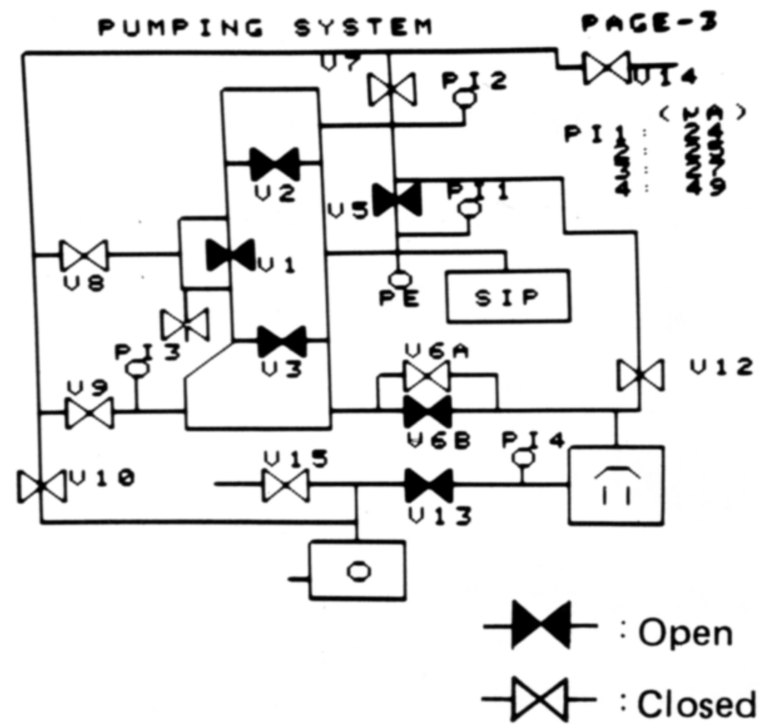
Actual vacuum system

2000FX



Symbols

- DP: Oil diffusion pump
- F: Filter
- PeG: Penning gauge
- PiG: Pirani gauge
- PG: Pressure gauge
- PS: Pressure switch
- R: Vacuum reservoir
- RP: Oil rotary pump
- WB: Water-cooled baffle
- : Release valve
- : Manual valve
- : Pneumatic valve
- : Solenoid valve



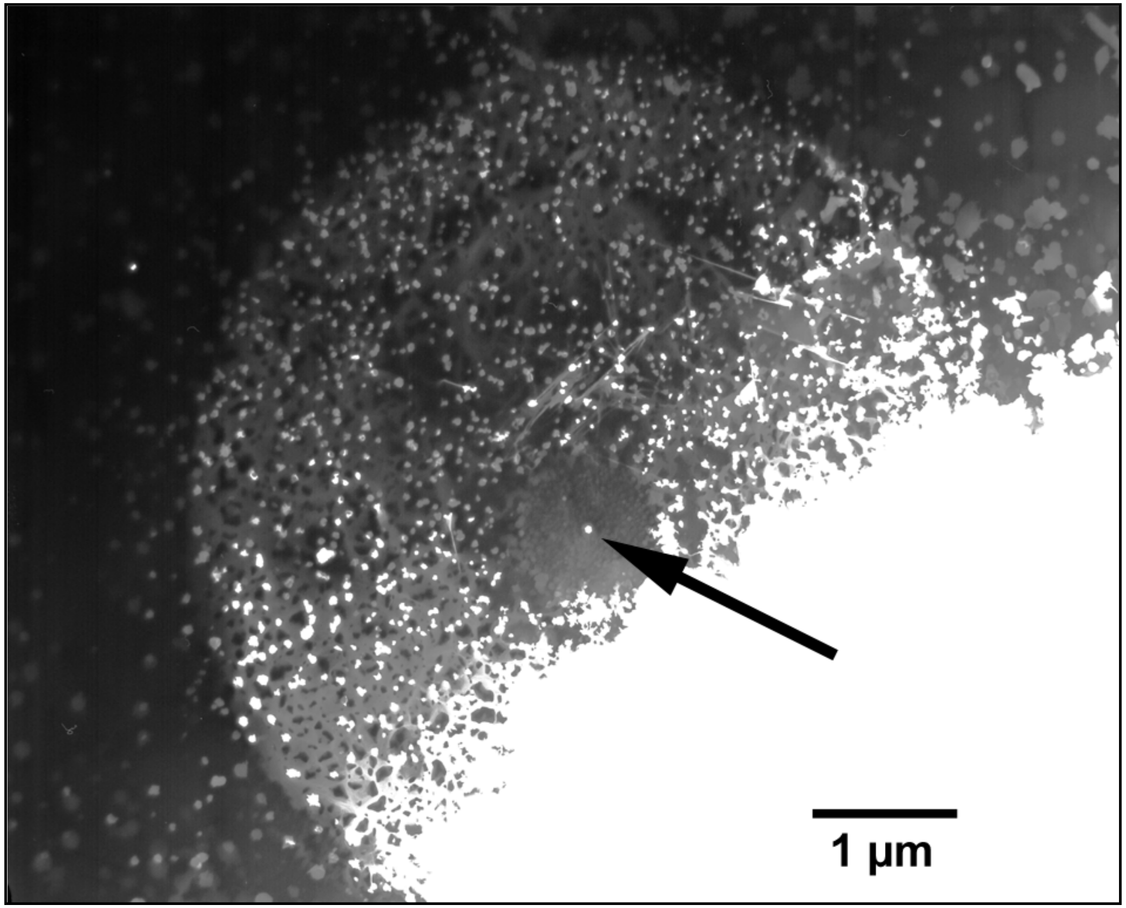
Contamination

Main worry is residual hydrocarbons

- Pump oil
- Specimen & specimen holder

You can help

- Use gloves, use care (don't touch past the o-ring)



Residual water

- Film