Vacuum Systems & Thin Films
(Jaeger 6, Campbell 10&12, Ruska 7)

- Deposition of thin films involves vacuum system

Direct Deposition from Source

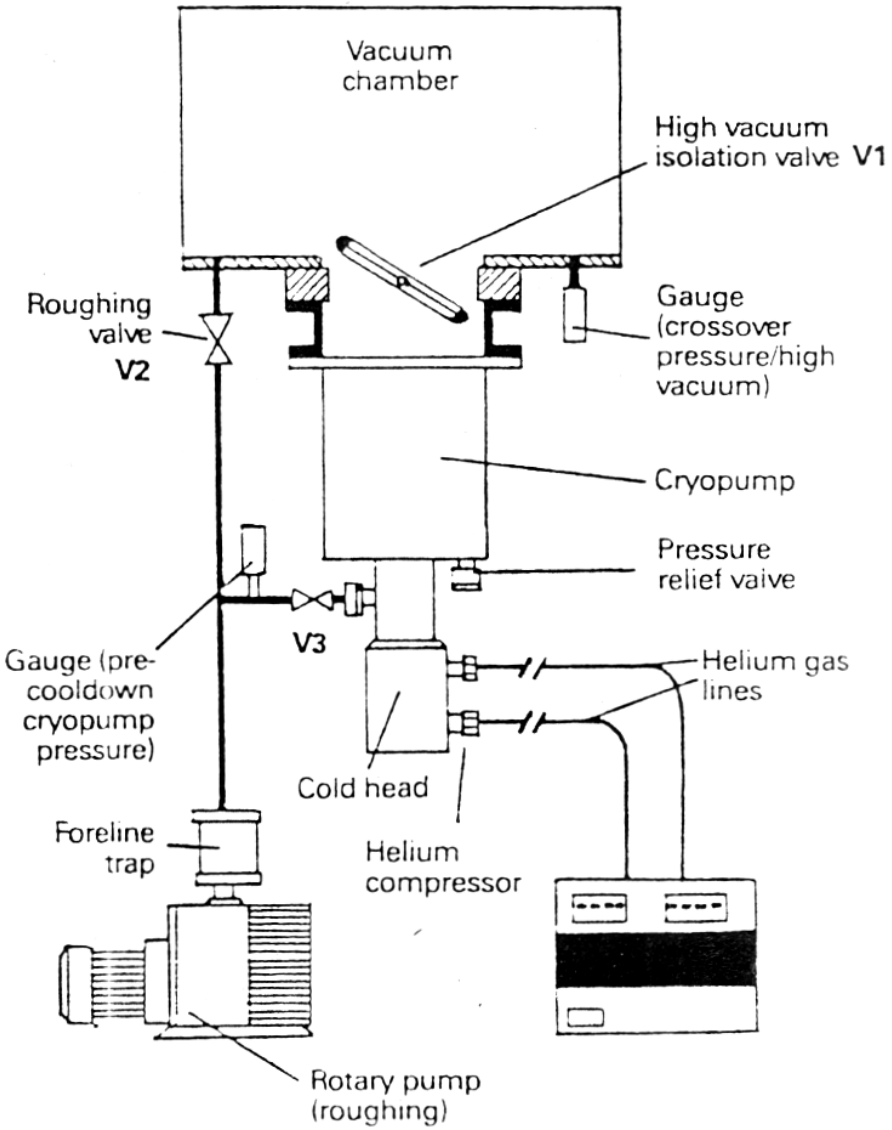
- Evaporation
- Sputter

Building up layers

- Chemical Vapour Deposition (CVD)
- Molecular Beam Epitaxy

Also Dry etching (Plasma, Reactive Ion Etching)

- All require an understanding of Vacuum & Vacuum systems



Vacuum and Gases

- Recall Ideal Gas law

$$PV = nRT$$

where P = pressure (atm)
V = Volume
N = number of moles of gas
R = Gas constant
T = temperature (^{o}K)

- RT = 22.4 l/mole at Standard Temperature & Pressure: STP
- Avogadro's Number = 6.03×10^{23} molecules/mole
- Use this to relate pressure and volume in vacuum systems

Table 1.1 Units of pressure and values of the gas constant in various units

Unit	Value of 1 atm	Basis
Atmosphere (atm)	—	Atmospheric pressure
Pascal (Pa)	1.013×10^5 Pa	1 N m^{-2}
Torr (or mm Hg)	760 torr	Pressure exerted by a mercury column 1 mm high
Micrometer (μm)	760×10^3	Pressure exerted by a (hypothetical) mercury column 1 μm high

Value of the gas constant R:

=	0.08205	liters atm $mole^{-1}$ $°K^{-1}$
=	8.314	J $mole^{-1}$ $°K^{-1}$
=	62.4	liters torr $mole^{-1}$ $°K^{-1}$
=	1.987	cal $molecule^{-1}$ $°K^{-1}$
=	22.40	liters atm $mole^{-1}$ at 273°K

Mean Free Path

- With temperature gases move around
- As pressure decreases they move longer distances
- Measure this with the Mean Free Path (MFP)

  Distance molecule moves before hitting another molecule

$$MFP = \frac{1}{\sqrt{2}\pi N d^2} \qquad (dm)$$

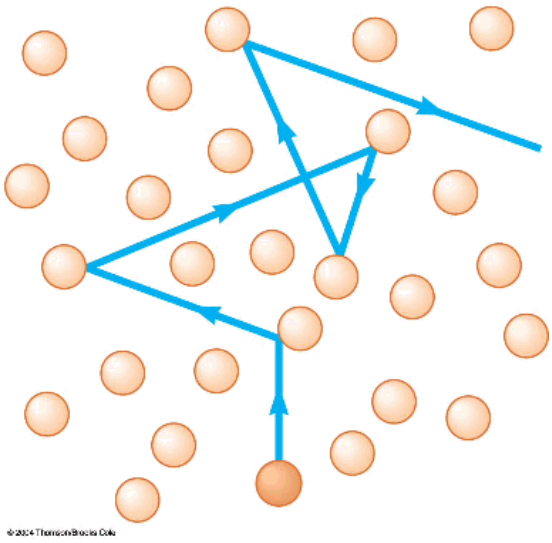
where N = molecules / l

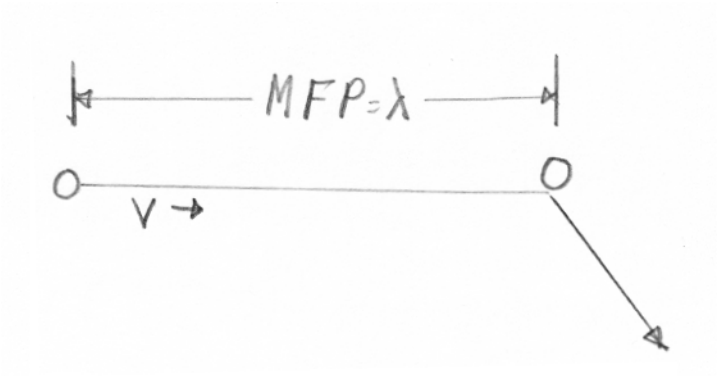
d = molecular diameter (dm=decimeter)

Table 7-2 Properties of gases of interest in thin-film deposition

Gas (formula)	Molecular weight (g/mole)	Molecular diameter† (nm)	Abundance in air	Boiling point (°K)	Comment
Nitrogen (N_2)	28	0.316	78.08%	77.4	—
Oxygen (O_2)	32	0.296	20.95%	90.2	—
Argon (Ar)	40	0.286	0.93%	87.4	Usual choice for sputtering
Carbon dioxide (CO_2)	48	0.324	0.031%	194.7‡	—
Water (H_2O)	18	0.288	Varies	373.15	Absorbs strongly on most materials
Hydrogen (H_2)	2	0.218	0.5 ppm	20.3	Formed by breakdown of water
Helium (He)	4	0.200	5 ppm	4.2	Used for leak detection

† Derived from gas viscosity (Ref. 14).
‡ Carbon dioxide sublimes rather than boils.





Mean Free Path and Gas Flow

- For air at 300°K the Mean Free Path (MFP) is

$$\lambda = \frac{0.05}{P(torr)} = \frac{6.6}{P(pa)}$$

where P = pressure

λ = Mean Free Path in mm

- 63% of molecules undergo collision in less than MFP

Viscous Flow

- At high pressure MFP is short
- Molecules collide with each other: creates Viscous Flow
- Slow near walls – fastest at the centre of chamber
- Slow moving molecules at wall makes for more collisions

Molecular Flow

- At low pressure MFP is longer than chamber narrowest part
- Molecule collision with walls dominate: Molecular flow

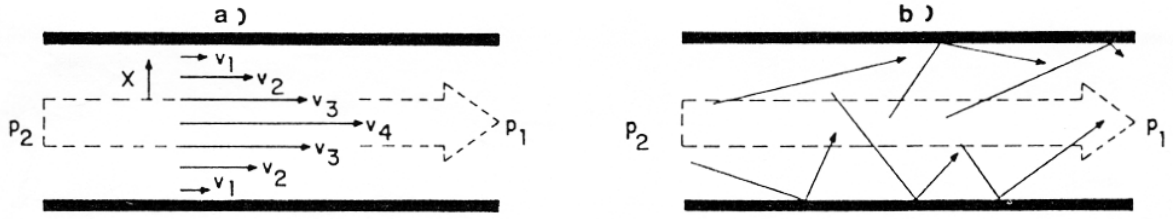


Fig. 2 (a) Schematic representation of particle velocities for viscous flow through a narrow tube. (b) Schematic representation of particle velocities for free molecular flow through a narrow tube. From L. Maissel and R. Glang, Eds., *Handbook of Thin Film Technology*, 1970[3]. Reprinted with permission of McGraw-Hill Book Company.

Vacuum Units

- Standard units are Pascals: 1 Newton force per sq m
  official SI unit
- Everyone in the industry still uses torr (1 mm of mercury)
- Vacuum is called High when pressure is low (<1 mTorr)

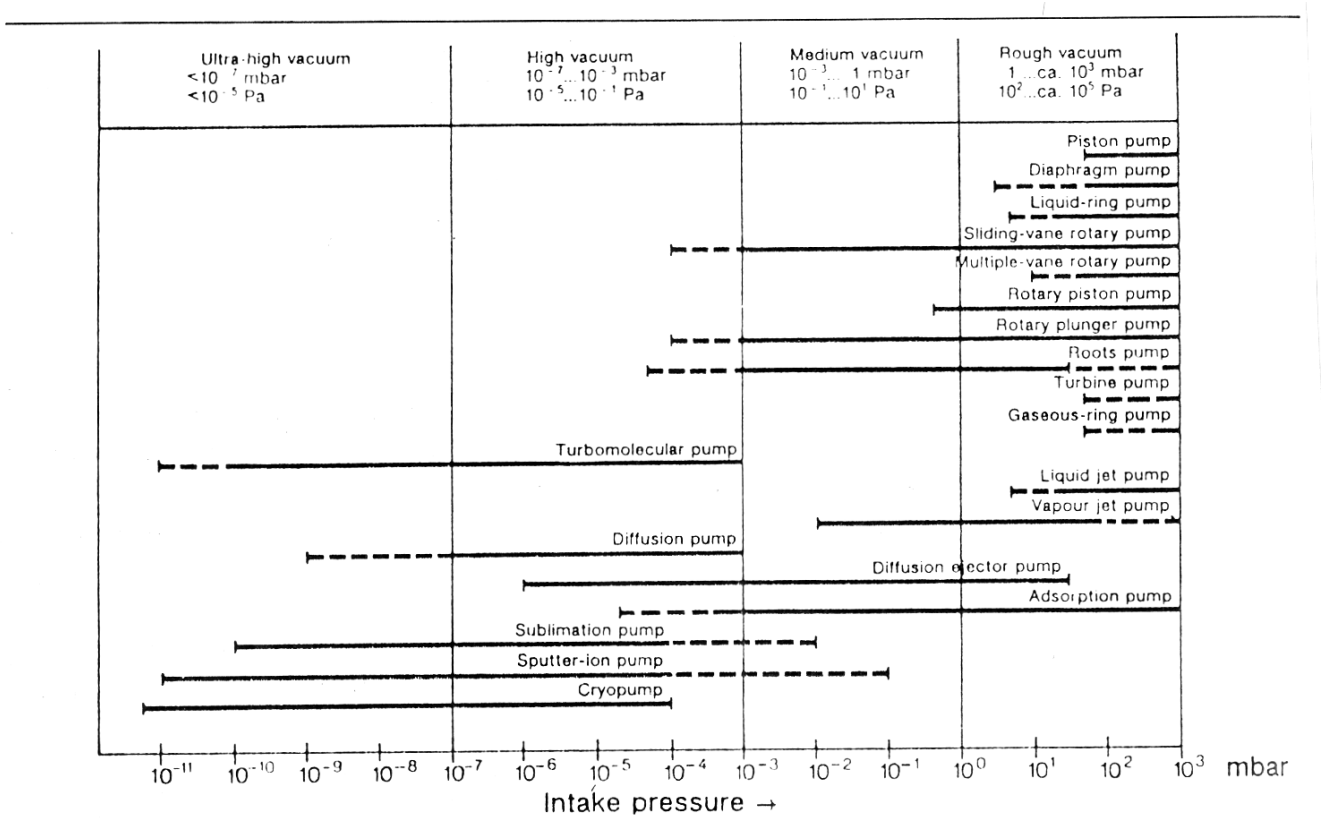
1 pascal = 7.5×10^{-3} torr = 7.5 microns of Hg
1 torr = 133.3 pascal
1 bar = 1×10^{5} Pa = 750 torr
1 atm = 1.013×10^{5} Pa = 760 torr

Pressure Ranges

Low	- 10^{5} Pa (750 torr)	to 3.3×10^{3} Pa (25 torr)
Medium	- 3.3×10^{3} Pa (25 torr)	to 10^{-1} Pa (7.5×10^{-4} torr)
High	- 10^{-1} Pa (7.5×10^{-4} torr)	to 10^{-4} Pa (7.5×10^{-7} torr)
Very High	- 10^{-4} Pa (7.5×10^{-7} torr)	to 10^{-7} Pa (7.5×10^{-10} torr)
Ultra High	- 10^{-7} Pa (7.5×10^{-10} torr)	to 10^{-10} Pa (7.5×10^{-13} torr)

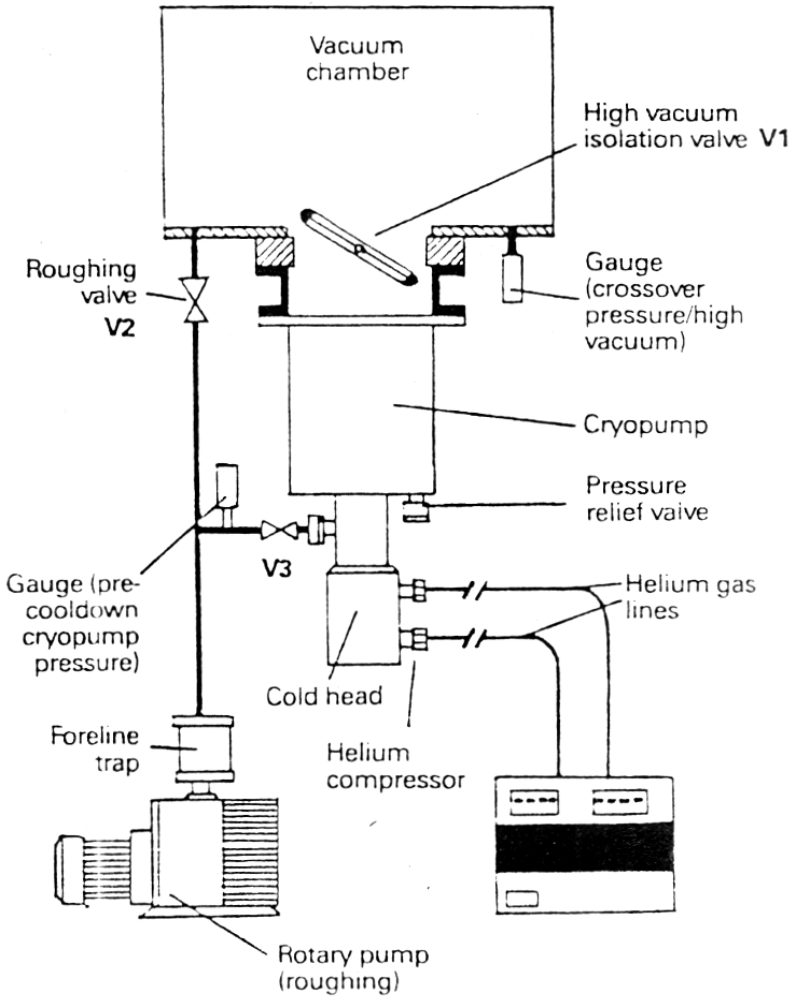
Vacuum Ranges

- Range of Vacuums
- Often described by the pumps used to achieve it
- Roughing: atm to 1 torr
- Medium vacuum: 1-10^{-3} torr used in many plasma processes
- High Vacuum: 10^{-3} to 10^{-7} torr: common for most deposition
- Ultra Vacuum: below 10^{-7} needed for molecular/epitaxial
- Most fab processes use either 10^{-2} to 10^{-6} Torr pressures Medium to High Vacuums
- Pumping systems needed to reach these important

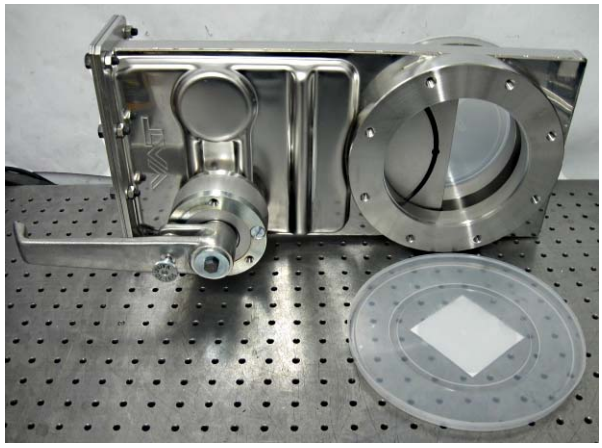


Typical Classic Vacuum System

- Systems usually contain two pump systems
- Medium vacuum section (Roughing or Fore pumps):10's mTorr
- High Vac pump (diffusion or cryo pumps) to microTorr range
- Pump chamber is switched between each using roughing and high vac values



Butterfly High Vac Valve



Gate High Vac Valve

Rotary or Foreing Pump

- Basic vacuum: Rotary or Foreing pump
- Also called mechanical or roughing pump
- Rotation of springed vanes sweeps air around in chamber
- Pump disk offset from chamber so as rotates volume shrinks
- Compresses air at outlet valve until it exceeds atm pressure
- Exhaust passes through oil (prevents backflow of gas)
- When pumping at atm pressure so much gas oil spews out exhaust
- Makes gurgling sound when this happens
- Pump flow rate is $Q = PS$

where Q = flow gas volume remove by pump (liter-atm/sec)

P = pressure (atmosphere)

S = pumping speed (liter/sec)

(sometimes time unit used is minutes instead of sec.)

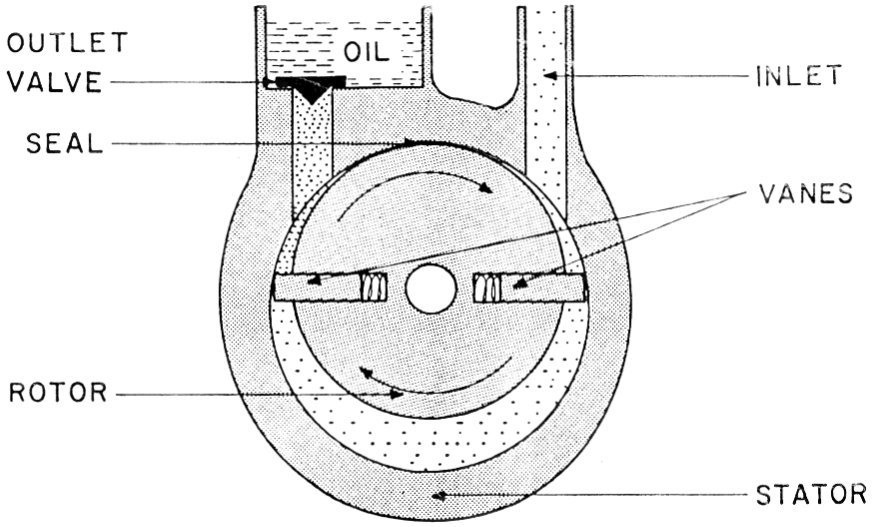


Fig. 1 Schematic of a vane-type rotary oil pump.

Saturation of Roughing Pump

- Roughing typically saturates about ~10^{-3} torr (milliTorr)
- At limit pressure gas cannot be compressed to air pressure
- Results in sharp fall off in pumping speed (volume)
- Double stage pumps better
- Final chamber pressure depends on chamber leakage/outgas rate
- Gas leaking into chamber = pumping rate at limit

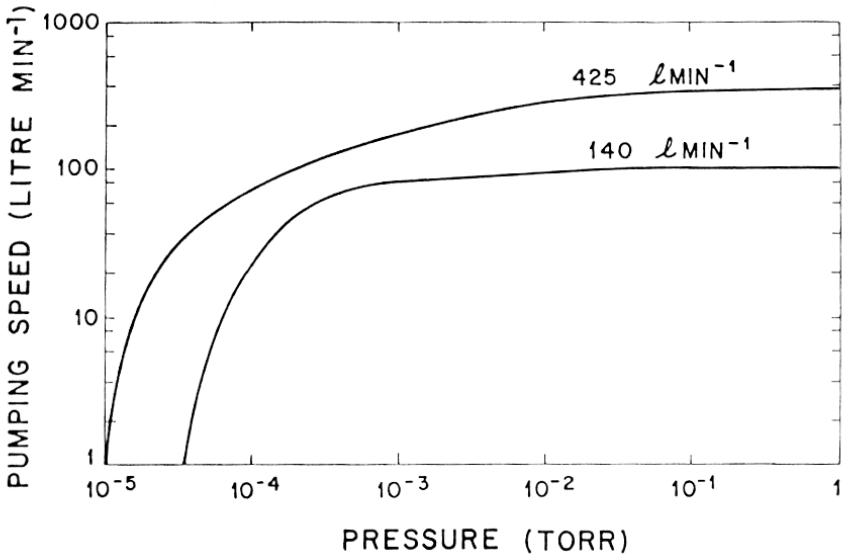
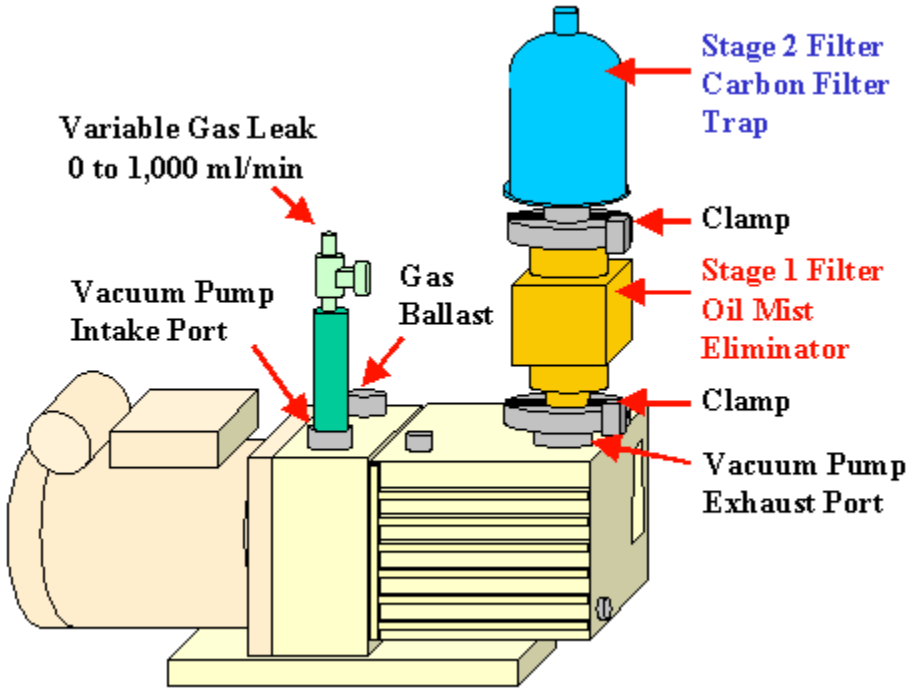


Fig. 2 Two-stage rotary oil pump speeds as functions of pressure (for models from Welch Scientific Co., Skokie, Ill.).



Diffusion Pump

- Start by roughing out chamber first:
- Diffusion pumps create High Vac: **Do not use above 10^{-2} torr**
- High vac pump operating by boiling pump oil
- Pump tower directs vapour downward
- Oil molecules push gas down by collision & momentum transfer
- Creates high pressure at foreline (above millitorr)
- Roughing pump on output foreline removes gases to atmosphere
- Water jacket cools pump walls: oil condenses for next evaporation

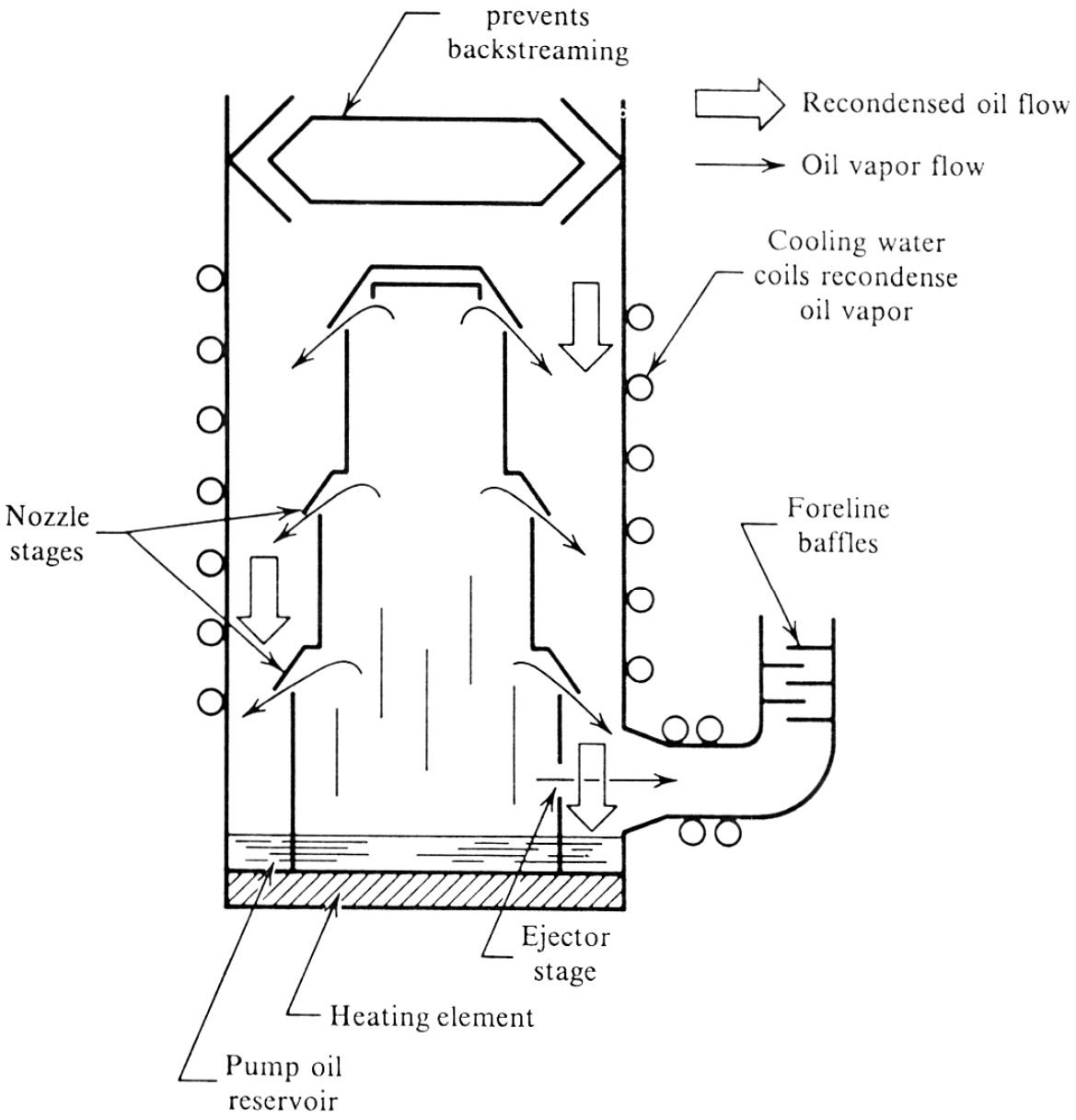
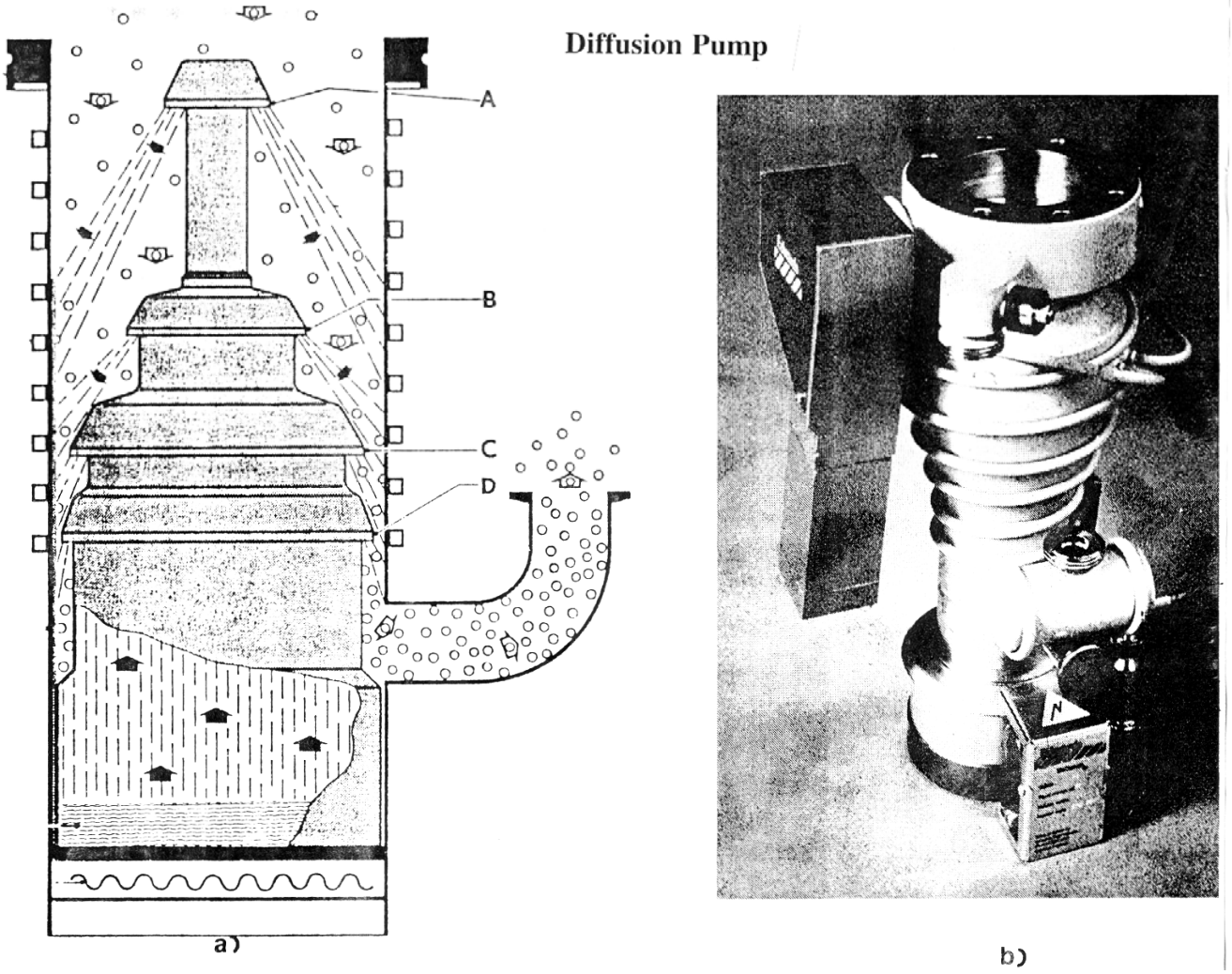


Figure 7-2 A diffusion pump. Oil heated in the reservoir emerges from the jets and collides with gas molecules, driving them into the roughing pump.

Diffusion Pump

- Diff pumps rated by pump throat diameter (eg 6 inch, 8 inch)
- Diff pumps are very heavy
- Most diff oil in pump destroyed by air when hot
- Expensive silicone based oils better
- Do not bring up to air when pump hot
- Pumps have electric circuits to shut down if power fails
- Pump must be hot before high Vac valve opened
- May hear gurgling sound when high vac value first opened
- Comes from high pressure hitting the foreing pump



Turbomolecular Pumps

- High Vac pump alternative to Diffusion pump
- Multistage turbine running at very high speed 20-90 Krpm
- Rotor or Stator turbine blade pairs mounted in series
- Each stage compresses by transferring momentum to gas
- Give momentum to molecules by repeated collision with blades
- Starts at roughing pressure: 10^{-3} torr
- Can reach ultra high vac – 10^{-12} torr
- Exhausts to roughing pump just like Diff pump
- Advantage – no Diff pump oil to contaminate system
- Often faster to high vac than diff pumps
- Disadvantages – tends to disintegrate suddenly: total destruction
- Much costlier than Diff pumps
- Cannot be made to same size (bore) as diff pump: smaller systems

Cold Trap Systems

- Used to trap water vapour & oil from diff pump
- Cold trap filled with liquid Nitrogen (77 ^{o}K)
- Most important: stops back streaming of Diff pump oil
- If oil gets in chamber contaminates vacuum system
- Also freezes out water, some carbon dioxide
- Significantly reduces pressure – more than factor 10
- Must keep cold trap filled!
- Do not open high vac if trap warm

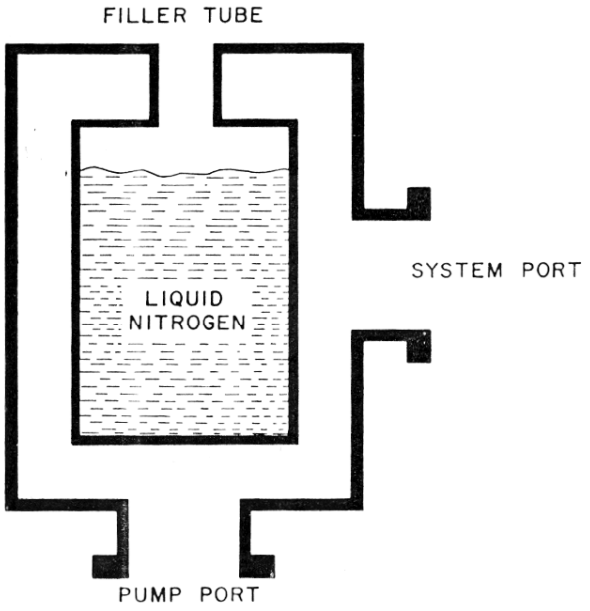


Fig. 9 Liquid-nitrogen container trap for oil diffusion pumps.

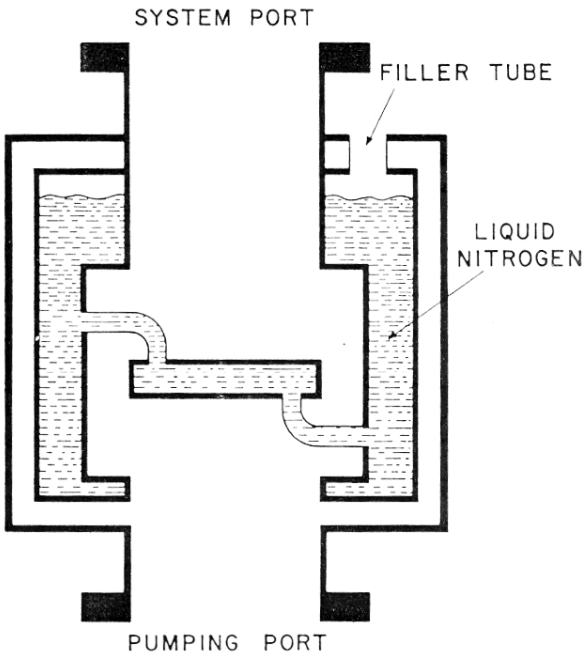


Fig. 10 Dewar trap with cooled baffle.

Cryopumps

- Extrapolation of the cold trap
- Entrapment of gases by chemical or physical action on surfaces
- Works with van der Waal forces Gas to walls
- 100K to remove water, but 20 K for air
- Get to 10^{-8} Torr or 10^{-4} Pa pressure
- Much cleaner than Diff pump – reaches lower pressures
- Most use Gifford and McMahon 2 stage Helium refrigeration
- 1st stage 50-75K cools radiation shield & baffels
- 2nd 10K inner cryopanel that traps gases in adsorbent material
- Regeneration cycle removes the captured gases (warm up)

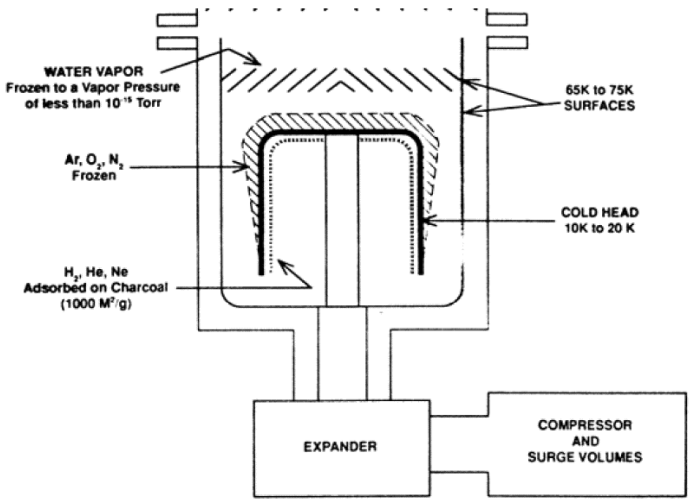


Fig. 6: Typical set-up of a two-stage refrigerator-cooled cryopump

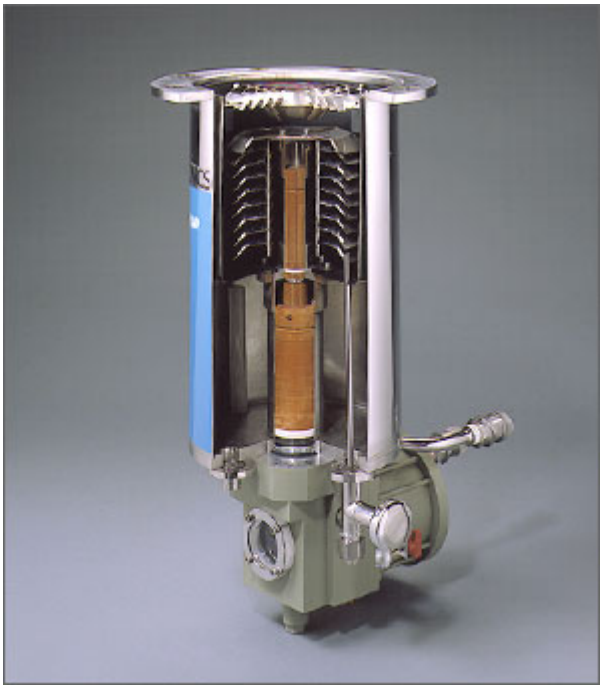


Table 2: Listed performance data (average taken from the catalogue of different manufacturers) for a typical 3000 l/s class two-stage GM cryopump

Parameter	Value
Pumping speed (l/s) water	9000–10500
Air	3000–3250
Hydrogen	4500–5200
Argon	2500–2700
Helium	1500–2300
Maximum throughput (Pa·m³/s) argon	1.0–2.5
Hydrogen	1.2
Pumping capacity (Pa·m³) argon	1.5×10^5–3×10^5
Hydrogen	1500–5000
Helium	10–100
Ultimate pressure (N_2 equivalent) (Pa)	$10^{-9} - 10^{-10}$
Cool-down time (h)	1.5–2.5
Crossover (Pa·m³)	35–50
Weight (kg)	30–50

Medium Vacuum Pressure Gauges

- Rough Vacuum gauges for above 10^{-3} torr
- Work by measuring heat loss to air
- Pirani Gauge: compare heat loss to standard low pressure tube
  uses a Wheatstone bridge setup
- Thermocouple gauge: measure T of hot wire:
- Low pressure higher Temp: uses thermocouple attached to wire
- Both low cost ($50) & robust
- Electronics cost about $1000 but supports many guages

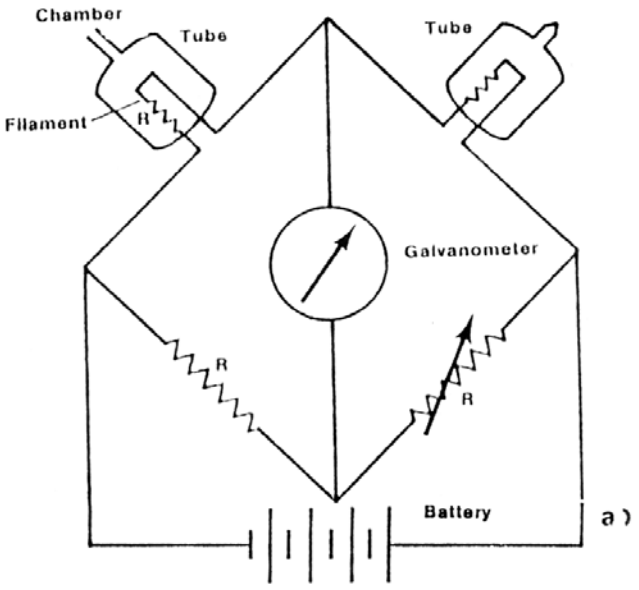
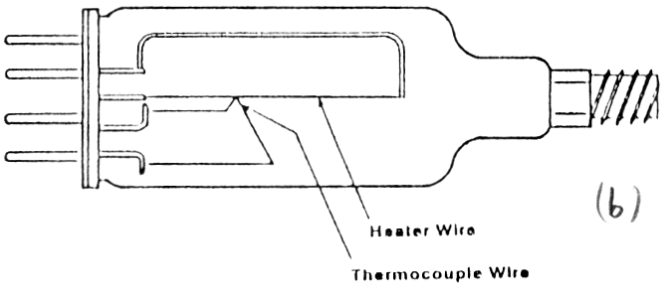
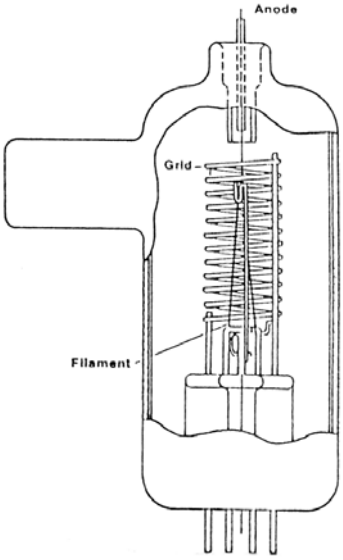


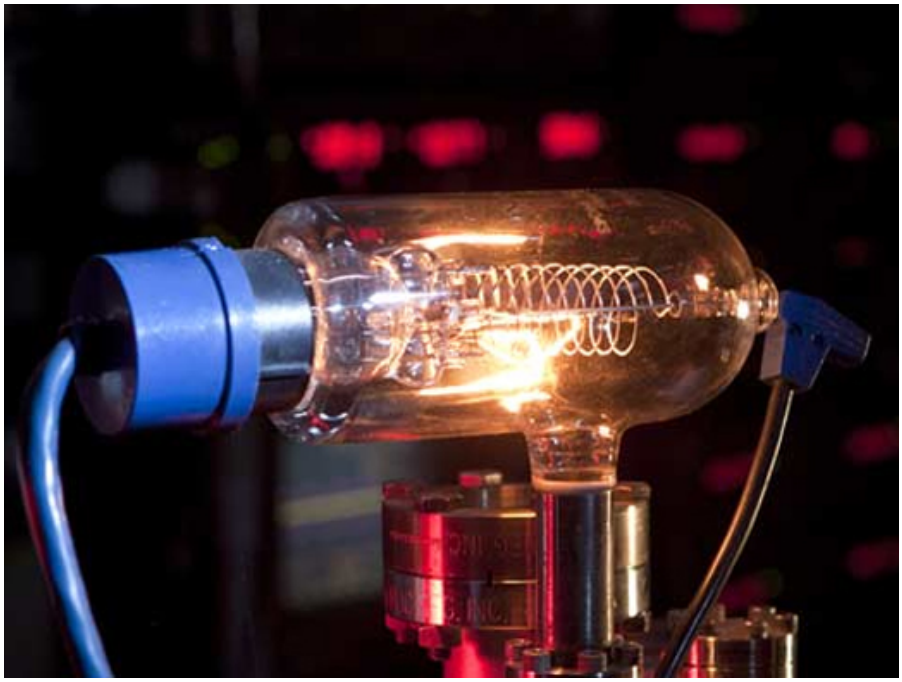
Fig. 18 (a) Pirani gauge. (b) Thermocouple gauge.



Ionization High Vacuum Gauges

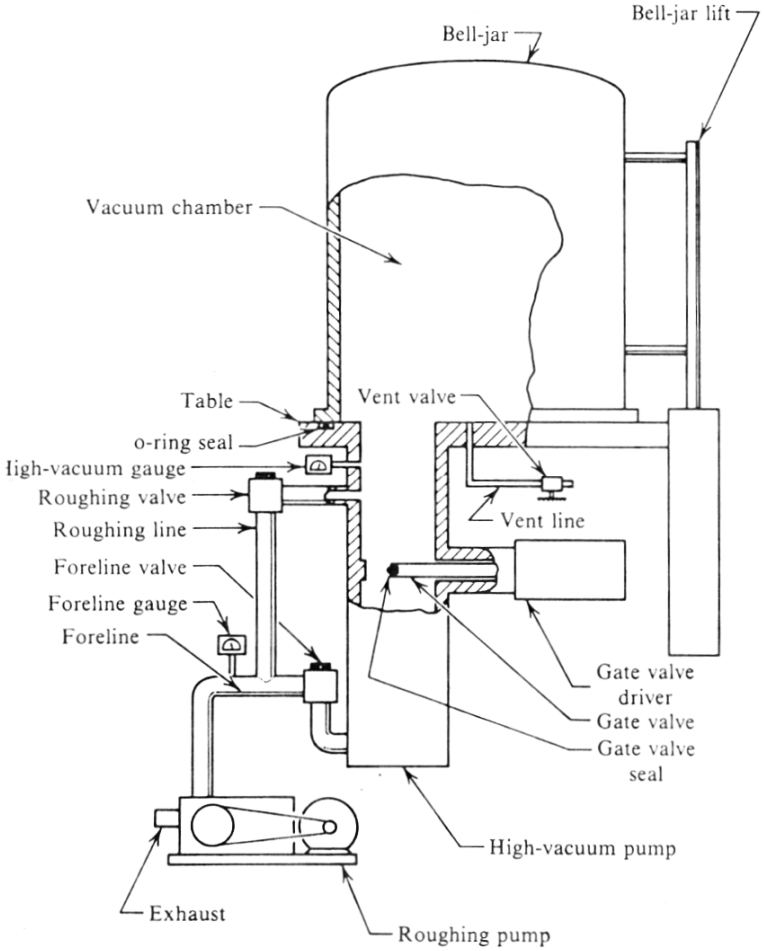
- Ionization gauge: works only a pressure $<10^{-3}$ torr
- Heater filament ionizes gas
- Measure current flow to anode
- Good till about 10^{-8} torr
- Burn out if brought into roughing pressure (10^{-2} torr)
  electronics have cutoff for high pressure: not always fast enough
- Walls and electrodes collect gases at higher pressure
- For true pressure must outgas
- Done by running heater in tube
- Baking out system drives off gases/water (for gauges & chamber)
- Expensive: tube costs $300 - $500
- Electronics about $2000

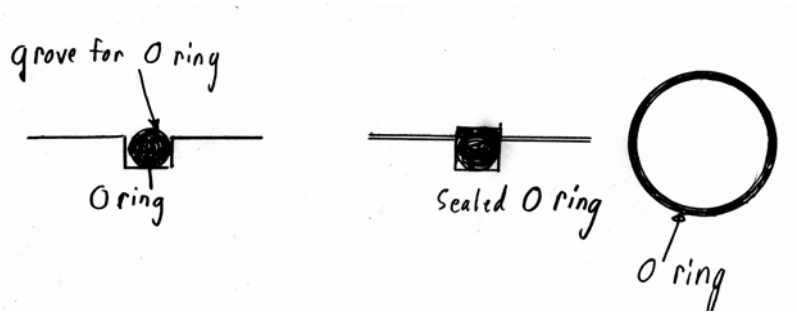




Bell Jar Evaporator

- Simple Bell Jar system has large glass bell jar chamber
- Chamber raised to open & set up operation
- Medium vacuum uses roughing pump
- High vacuum diffusion pump
- Seals done with O rings: rubber rings that flatten to seal groves





Typical Vacuum System Operation

- Close chamber & make certain seals tight
- All vac valves closed at this point but pumps running

Roughing Cycle

- Rough out chamber: open Foreline value to roughing pump
  Note: diff pump isolated: roughing value to diff closed!
- When first open foreline gurgles & may put out oil
- If pressure does not drop much then leak
- Watch thermocouple guage: get pressure to millitorr level

High Vacuum Cycle

- Diff pump must be hot & cold trap filled
- Close foreline value, open roughing line to diff pump
- Then open high vac value
- Turn on ionization guage
- Degas ionization when pressure gets $<10^{-5}$ torr
  & before final pressure reading

To Bring to Atmosphere

- Close High Vac, pause then close roughing to diff pump value
- Make certain foreline value to chamber closed
- Possibly back fill chamber with nitrogen (to keep clean)
- Bring to Atm, and break chamber seal

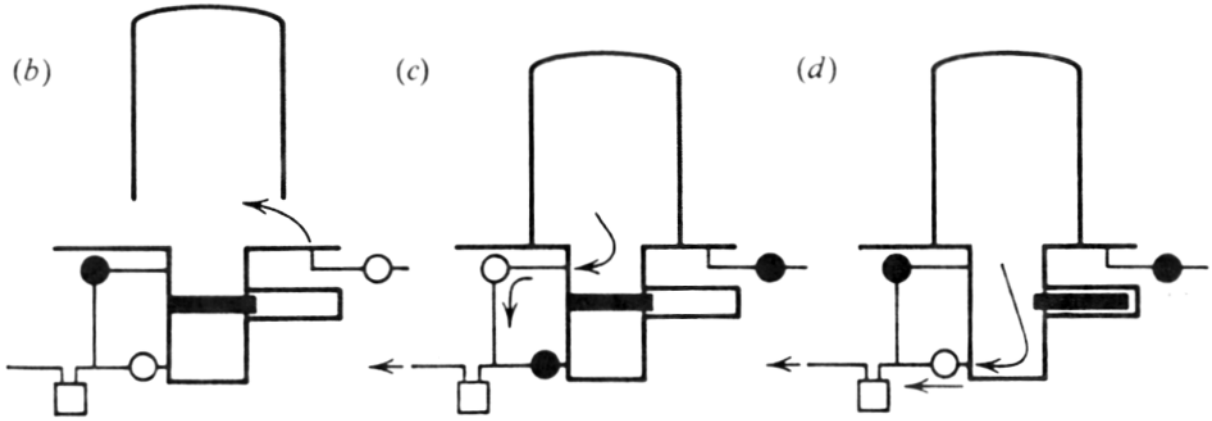


Figure 7-1 A typical apparatus for achieving high vacuum. (*b*) Valve configuration when chamber is open. (*c*) Valve configuration when rough pumping chamber from atmospheric pressure. (*d*) Valve configuration in high-vacuum operation.

Simple Evaporation

- Typical heating sources Ni-chrome heating elements
- Place sample eg Al wire in heat source
- Slowly turn power until melt covers source
- Then set for evaporation rate
- Best for pure materials

Problems

- Mixed materials eg alloys
  different materials come off at different rates
- Not very uniform coverage
- Materials with dissociate
- Difficult for dielectrics
- Need good vacuum:
  react with residual gas during evaporation

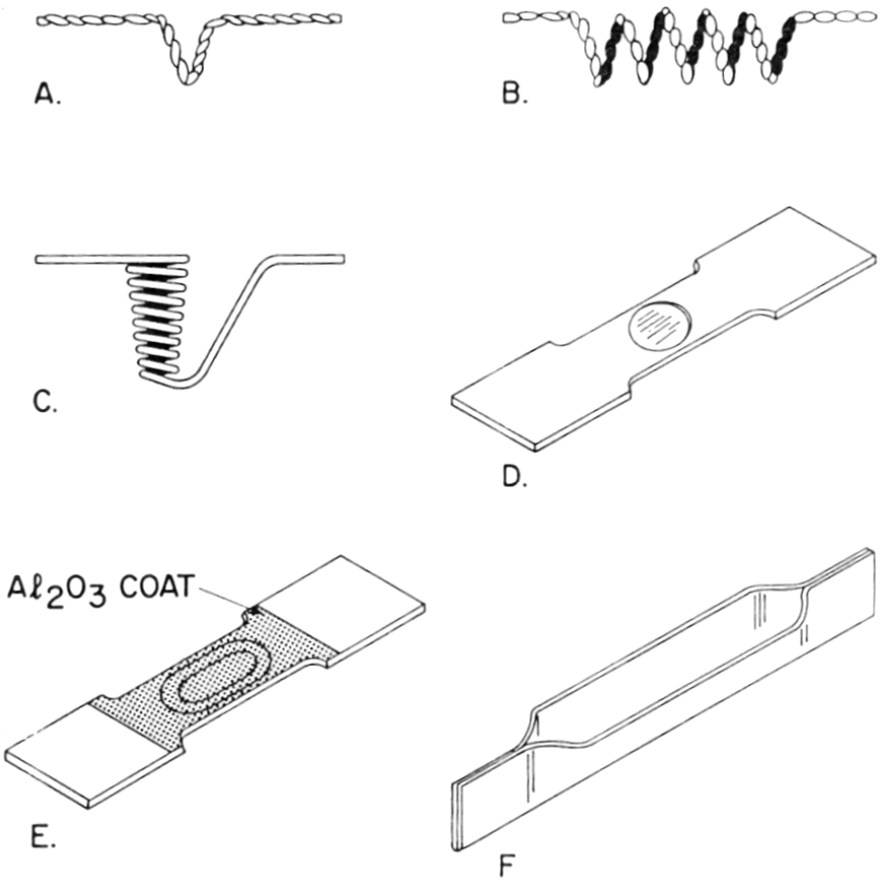


Fig. 13 Wire and metal-foil sources. (*A*) Hairpin source. (*B*) Wire helix. (*C*) Wire basket. (*D*) Dimpled foil. (*E*) Dimpled foil with alumina coating. (*F*) Canoe type.

Flash & E-beam Evaporation

- Flash Evaporator: feed wire or power
  get better alloy mixture
- E-beam: better heating: more uniform

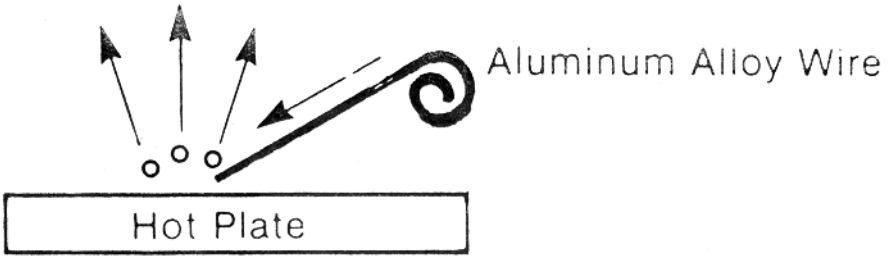


Figure 13.12 Flash evaporation source.

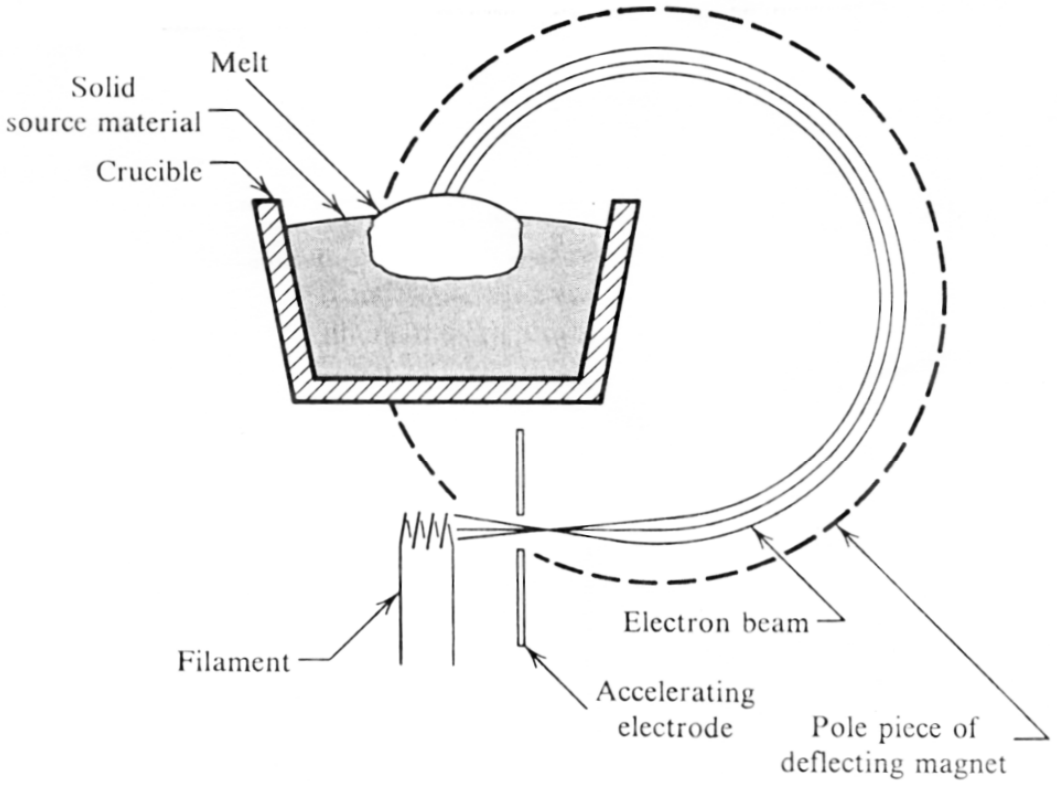
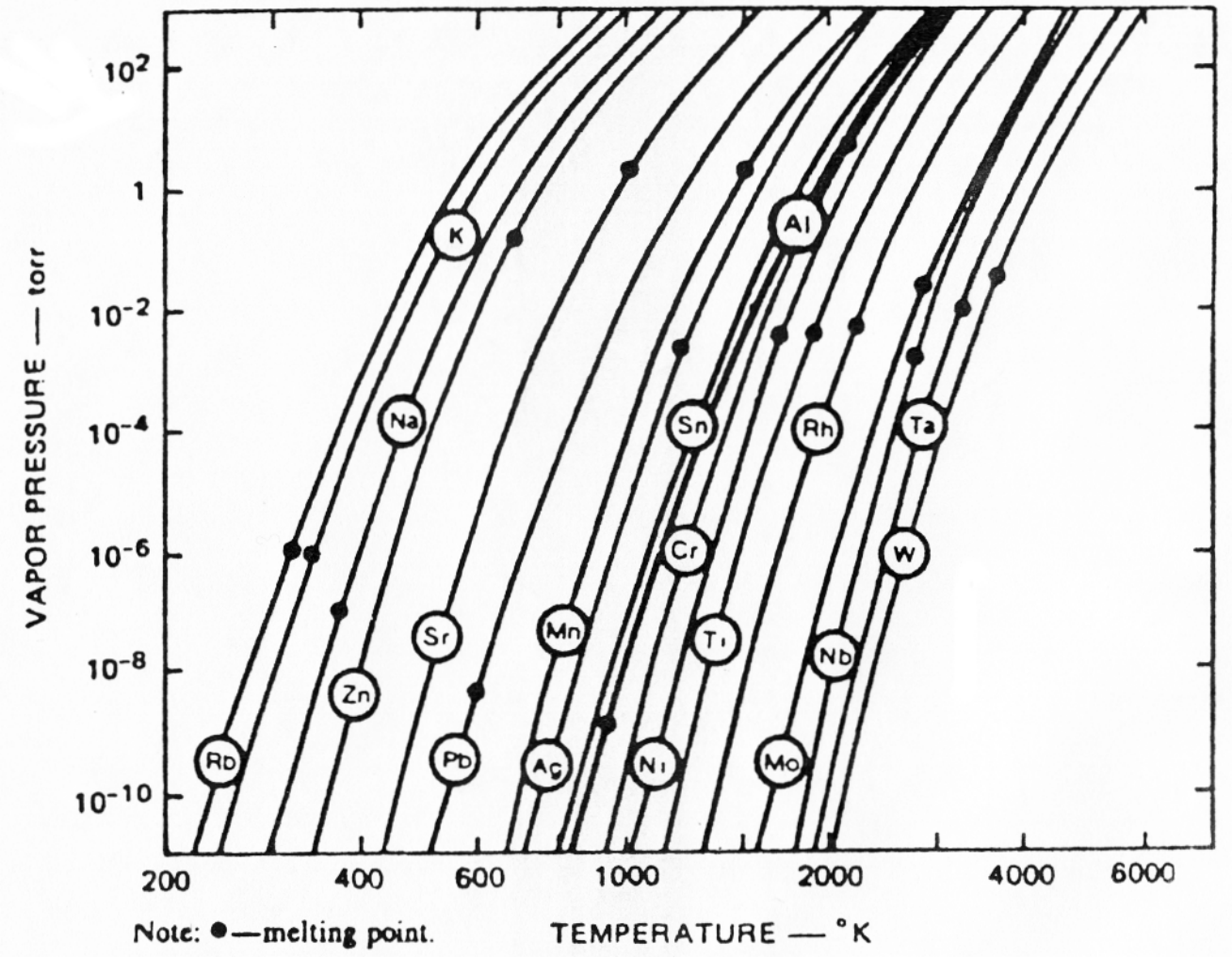


Figure 7-3 An evaporation source using electron-beam heating. The beam is generated out of the line of sight of the source and is focused into it by a magnetic field. A heated filament supplies electrons, and the accelerating electrodes form them into a beam.

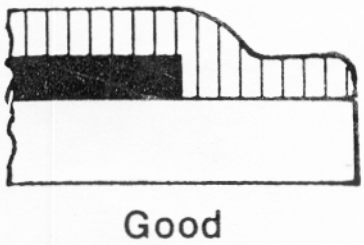
Vapour Pressure of Common Metals

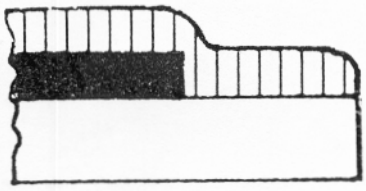
- Many metals (eg Al) can reach millitorr pressure at modest temp
- Hence evaporation easy for many materials



Planetary Rotation for Uniformity

- Problem: vapour comes off unevenly
- Rotate wafers for more uniform deposition
- Called a Planetary rotation system
- Rotate wafers on one holder, while rotate several holder
  Wheels within wheels





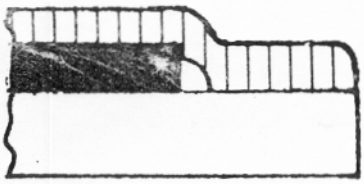


Figure 13.13 Step coverage.

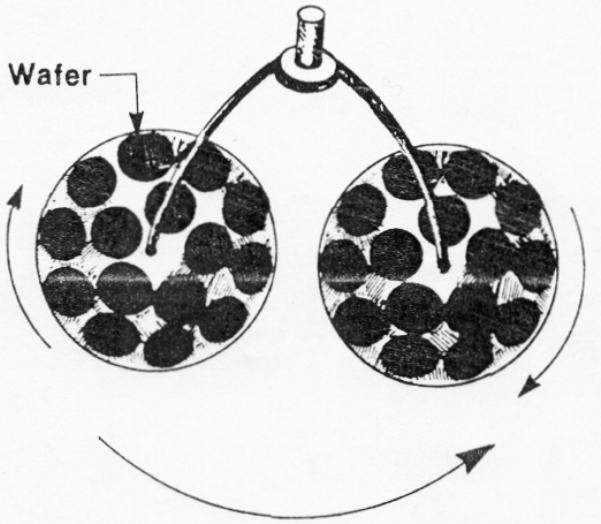


Figure 13.14 Planetary wafer holder.

Figure 13.15 Slope etch step coverage.

Planetary Rotation for Uniformity

- Planetary also puts wafers at angle
- Gives much better edge coverage
- Note many wafers loaded into planetary:
  better for smaller wafers (100, 200 mm)

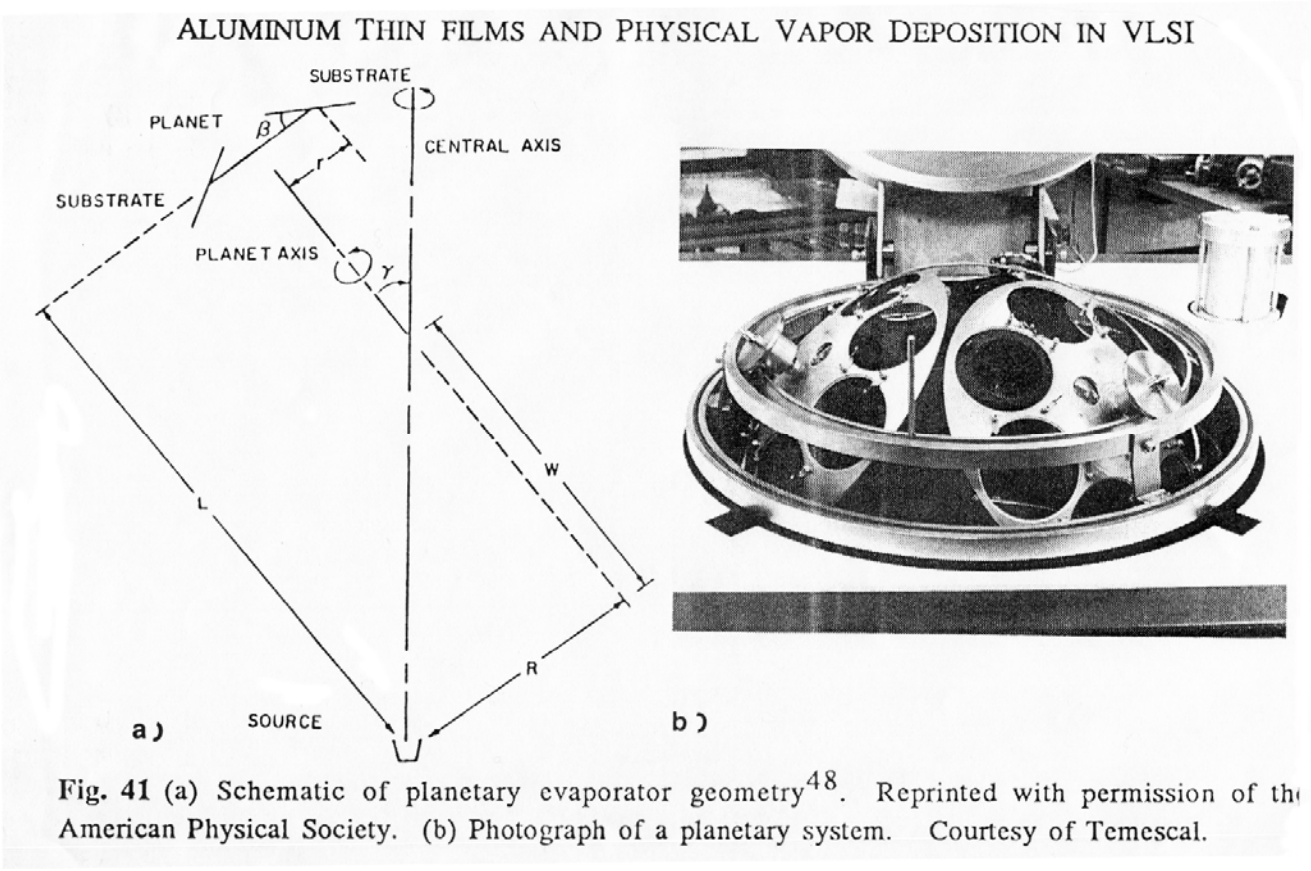


Fig. 41 (a) Schematic of planetary evaporator geometry[48]. Reprinted with permission of th American Physical Society. (b) Photograph of a planetary system. Courtesy of Temescal.

Step Coverage

- Build up at edges shadows bottom of line
- Get cracks in line: not continuous conductors
- More difficult as smaller holes/thicker lines
- Note: planetary methods used for many depositions
  e.g. sputter deposition and chemical vapour deposition

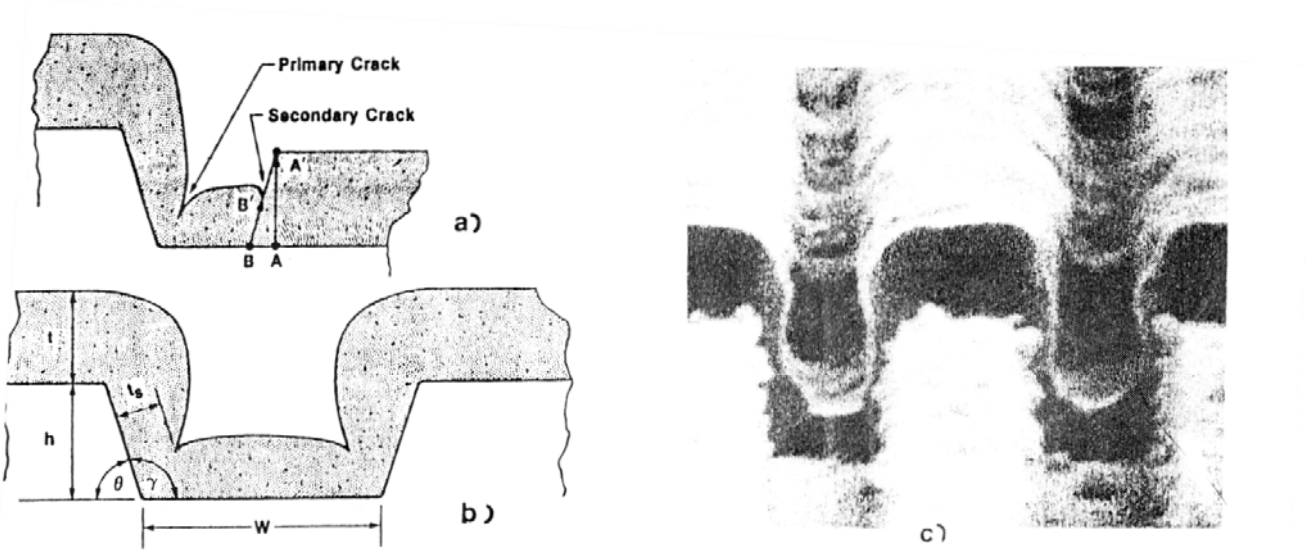


Fig. 32 Computer simulated film growth on a step: (a) Point source planetary system; (b) dc magnetron sputtering system[47]. Reprinted with permission of Solid State Technology, published by Technical Publishing, company of Dun & Bradstreet. (c) Example of poor metal step coverage.

Heating Substrate for Step Coverage

- Heated substrate causes deposited material to flow
- Combination of angle deposit and heat give better step coverage
- Needed for steep sidewalls
- Also relieves stresses in films

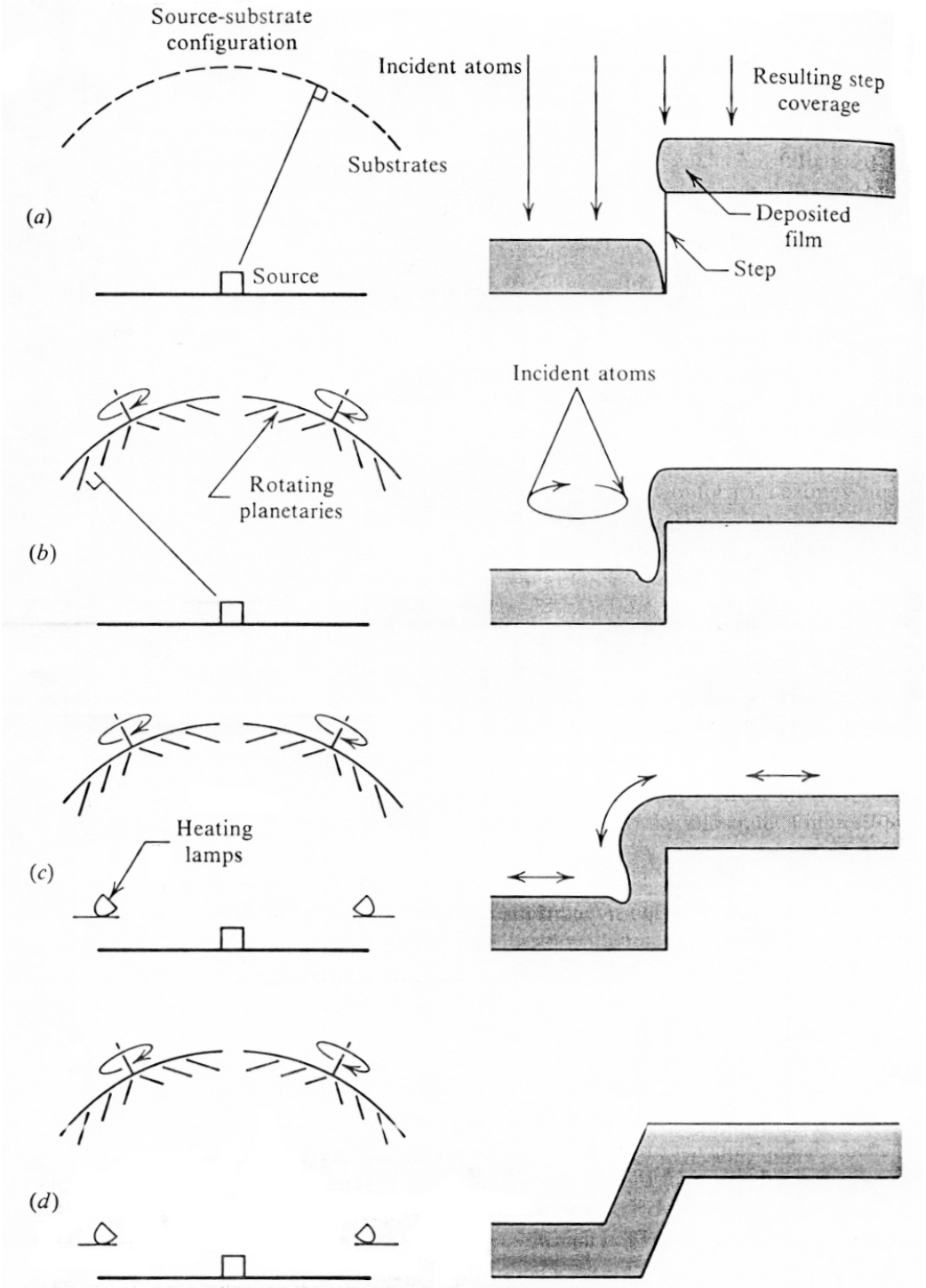
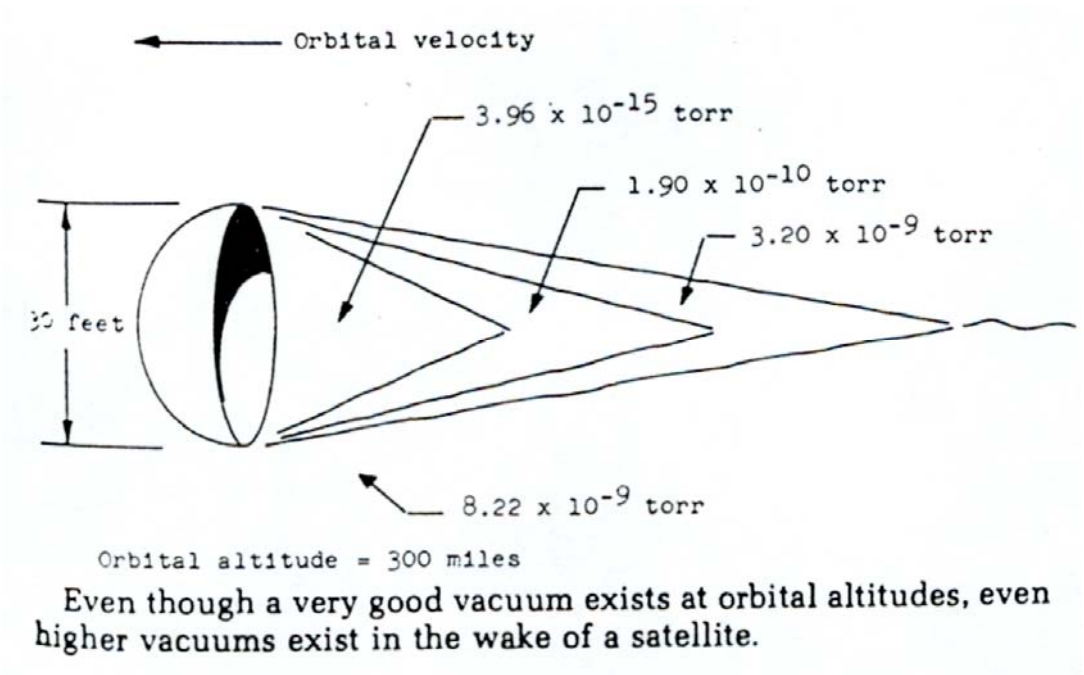


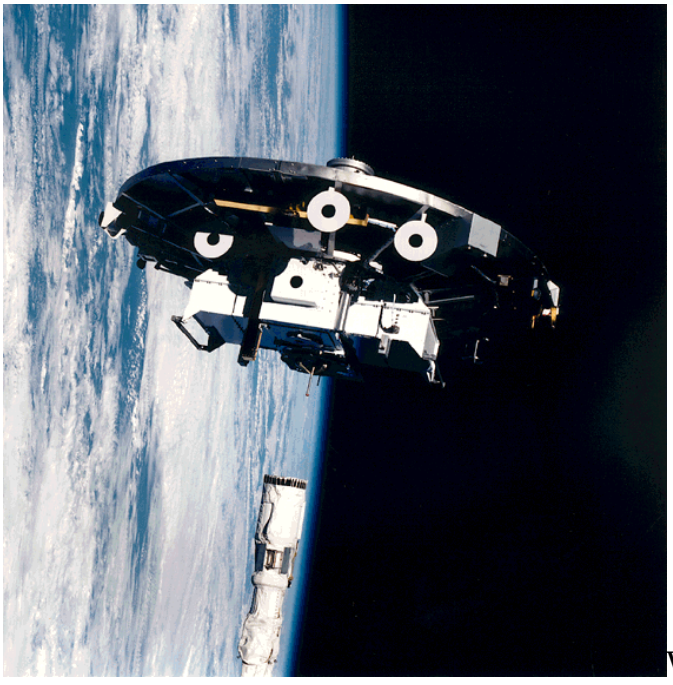
Figure 7-4 Factors governing step coverage in evaporation. (*a*) Perpendicular step on perpendicular substrate. No coverage. (*b*) Rotating planetaries with some substrate inclination. Improved coverage. (*c*) Same configuration with substrate heating. Further improvement. (*d*) Reduced slope of step, plus rotation and heating. No thinning over step.

Orbital Microfab Processing Vacuum

- Pumping down to Vacuum consumes large amounts of energy
- What about doing processing in Low Earth Orbit
- LEO has native pressure 10^{-8} torr: good for most processing
- Can get lower with a Wakeshield
- Shield facing in direction of orbit – at orbital speed 8 km/s
- Reduces vacuum to ultravacum – $4x10^{-15}$ torr
- Great for Epitaxy deposition



Even though a very good vacuum exists at orbital altitudes, even higher vacuums exist in the wake of a satellite.



Wakeshield launch from space shuttle

FabSat Project

- Project with Boeing to develop orbital Microfab
- Use a dry photolithography process
- Thus almost all work done in the vacuum
- Means wafers do not need to be constantly wet cleaned
- Reduces energy for processing by 96%!
- Designed for quick prototyping
- Wafers delivered to earth in small capsules