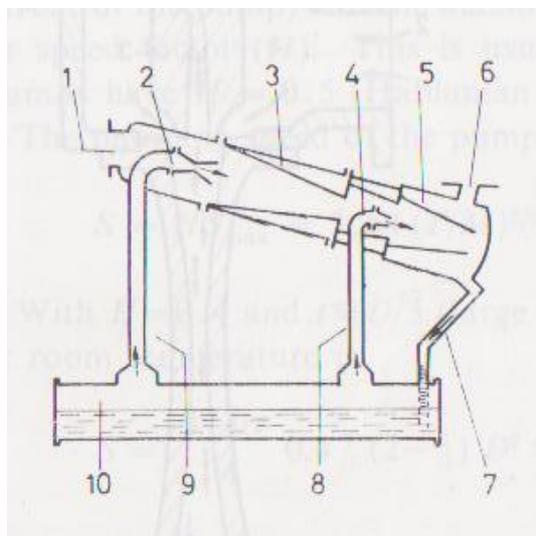


(a) diffusion pump: 1.High vacuum; 2.Water cooling; 3.First stage; 4.Second stage; 5.Pump oil; 6.Heater; 7.Fore-vacuum; (b) vapour pump; 1.Intake port; 2.Fore-vacuum; 3.Oil; 4.Heater.

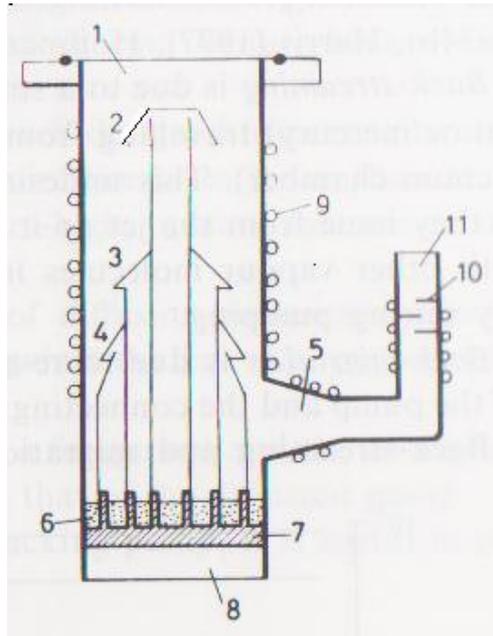
**Vapour Ejector pumps:**

Ejector pump work with oil or steam. The pump fluid is contained in the boiler (10) and the vapour flows through the jet chimneys (8,9) into the nozzle (2,4). Due to their special shape each of these nozzles produces a supersonic jet, which enters the nozzles (diffusers 3,5) and condenses on their cooled walls. The air to be pumped enters the pump through the high vacuum connection (1), and is carried with the jet and compressed. The process is repeated in the second stage. The air compressed to a pressure equivalent to that of the fore-vacuum line (6) is removed by the backing pump. The condensed fluid flows back through the return pipe (7).

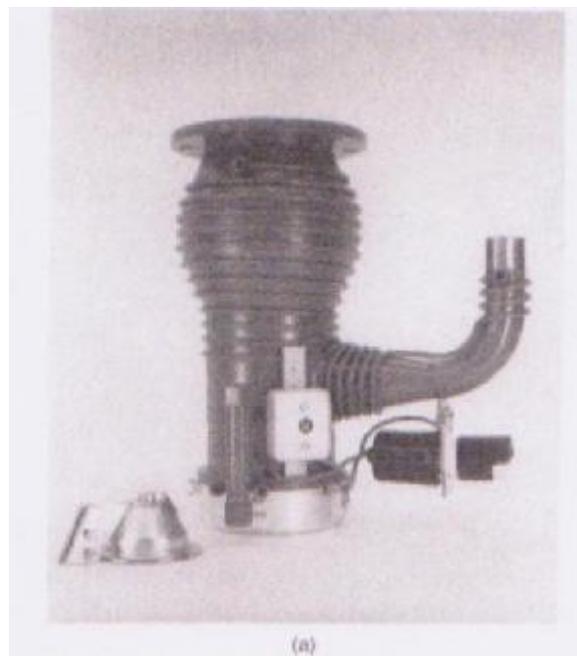


Double-stage oil-vapour ejector pum

**Diffusion pumps:**



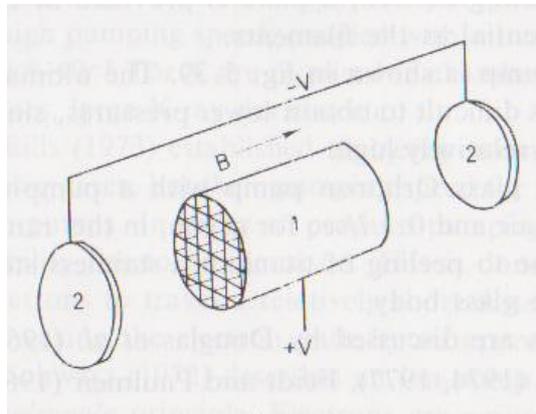
Oil diffusion pump; 1.Pump inlet, high vacuum; 2.First stage; 3.Second stage; 4.Third stage; 5. Ejector; 6.Pump fluid; 7.Fractionating boiler; 8.Heater; 9.Water cooling coil; 10.Forline baffle; 11.Backing line, fore-vacuum.



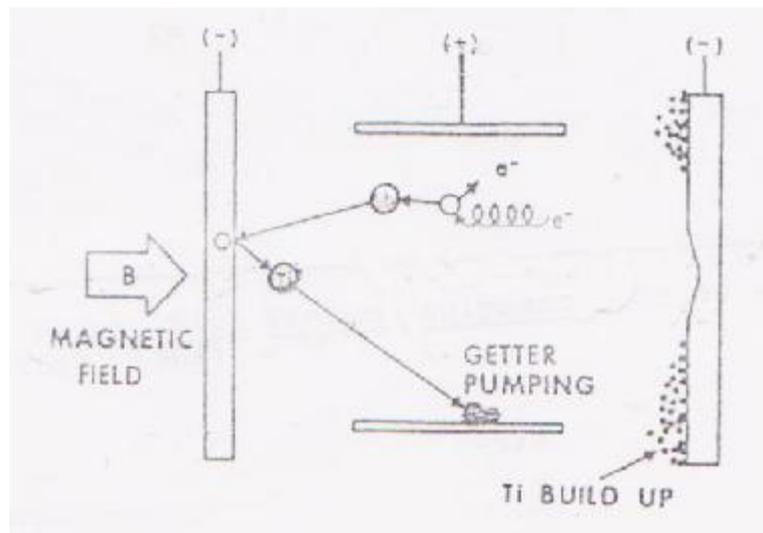
Diffusion pump (photograph)

### Sputter-ion-pumps:

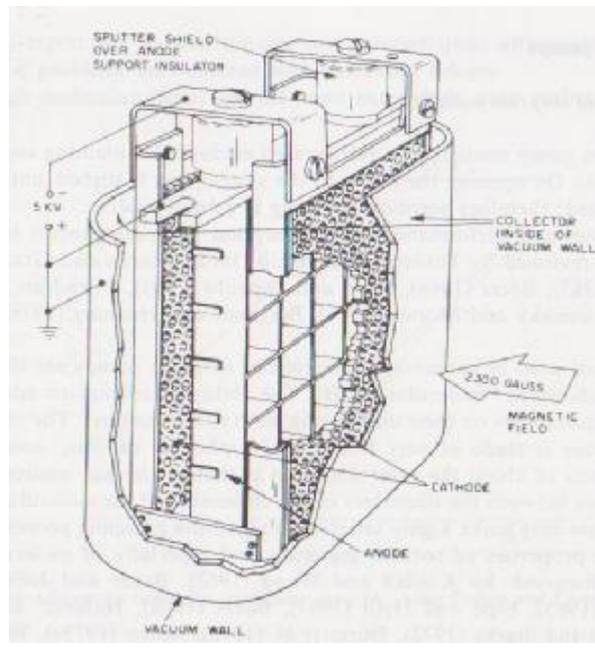
The sputter-ion-pumps are designed such that an electrical discharge occurs between the anode and the cathode at a potential of several thousands of volts in a magnetic field of a few thousand gauss. Since the magnetic field causes the electrons to follow a flat helical path, the length of their path is greatly increased. The gaseous ions so formed are accelerated to the titanium cathode, where they are either capture or chemisorbed. Due to the high energies they are propelled into the cathode plate and sputter cathode material (titanium), some of which settles on the surface of the anode where it also traps gas atoms.



Sputter-ion-pump (principle), 1.Stainless steel anode; 2.Titanium cathodes.



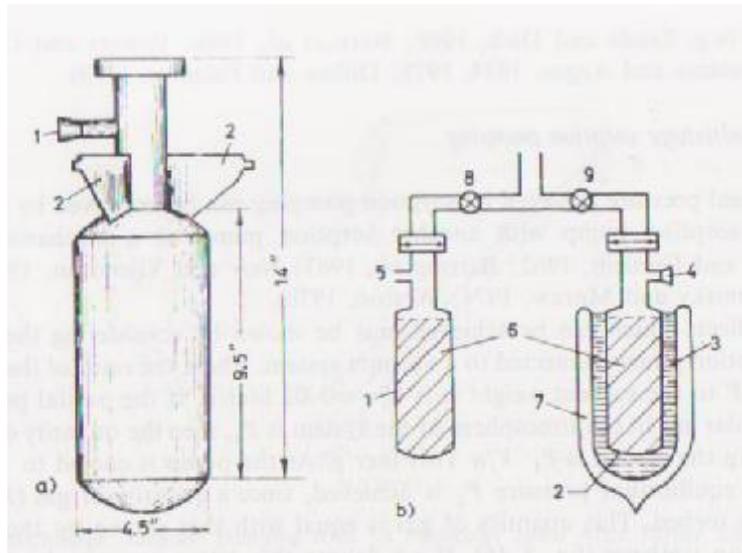
Sputter-ion-pump (principle)



Sputter-ion-pump (principle)

### **Sorption pumps:**

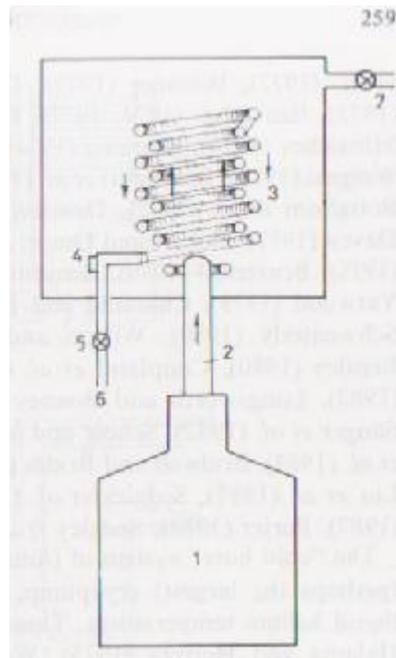
The sorption pump consists of a stainless steel body with internal copper fins to facilitate heat transfer to the zeolite charge. A liquid nitrogen container can be attached to three support brackets. The sorption pump is valved into the system, and immersed in liquid nitrogen, as the temperature of the zeolite (molecular sieve) falls, it sorbs more gas from the system to cause a reduction in the pressure. After pumpdown to the equilibrium pressure has been accomplished, the valve to the system is closed. At this stage the molecular sieve is saturated. The re-activation can be carried out by allowing the pump to warm at room temperature, care being taken to vent the pump. Adsorbed water is not readily evolved from the molecular sieve at room temperature, heats it up to 300°C.



- (a) Sorption pump, 1.Rubber stopper; 2.Liquid nitrogen container support.  
 (b) Two sorption pumps connected for multistage pumping, 1.Pump being re-activated; 2.Pump in operation; 3.Liquid nitrogen; 4.Rubber stopper; 5.Vent; 6.Molecular sieve; 7.Dewar; 8.Valve closed; 9.Valve open.

### **Cryopumps:**

A practical cryopump is illustrated in fig., it consists of a helix made of stainless steel tube, which acts as the condenser surface, mounted directly in the chamber to be evacuated. The coolant (liquid nitrogen, hydrogen or helium) is supplied from a Dewar to the helix through a vacuum insulated feed tube, and is made to flow through the coil by means of a gas pump at the outlet end of the coil. The coolant passes through the system, and hence the temperature to which the condenser is cooled, is controlled by a throttle valve mounted in the gas exhaust line. A temperature sensing element mounted on the condenser coils automatically controls the throttle valve setting. The cryopump is not used at pressures above  $10^{-2}$  Torr, partly because of the large quantities of coolant that would be required, and the thickness of solid built up during high pressure pumping would seriously reduce the pump efficiency at low pressures.

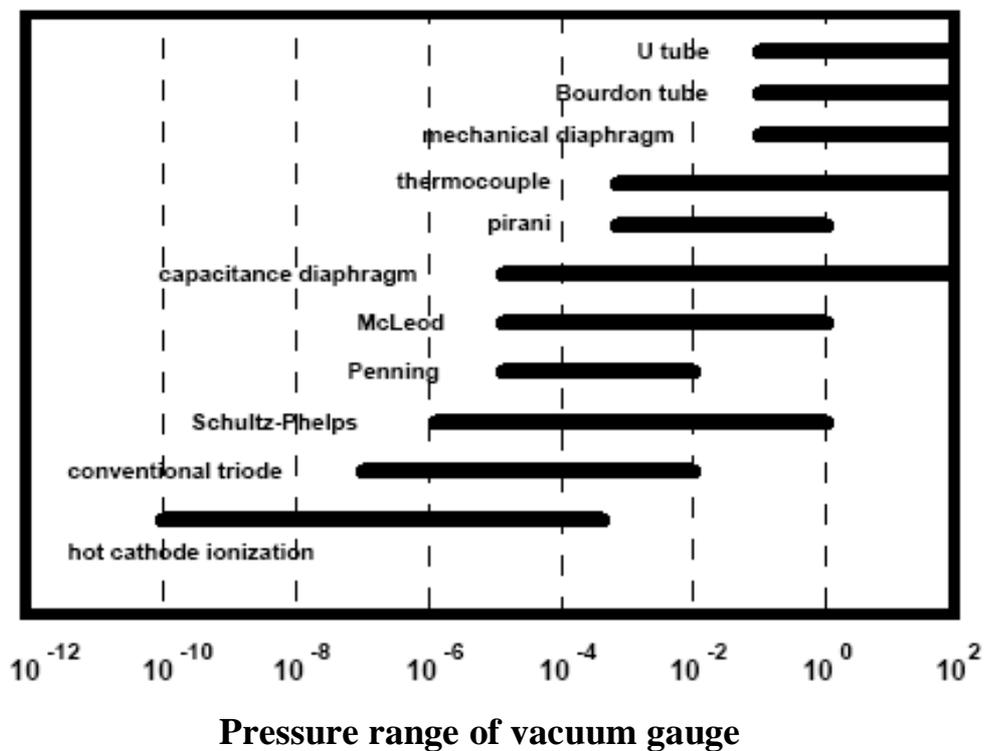


Cryopump(schematic), 1.Liquid nitrogen Dewar; 2.Insulated feed tube;  
3.Condenser coil; 4.Temperature sensing element; 5.Throttle valve;  
6.Connection to gas pump; 7.Connection to roughing pump.

# Measurement of low pressures

## Classification of vacuum gauges:

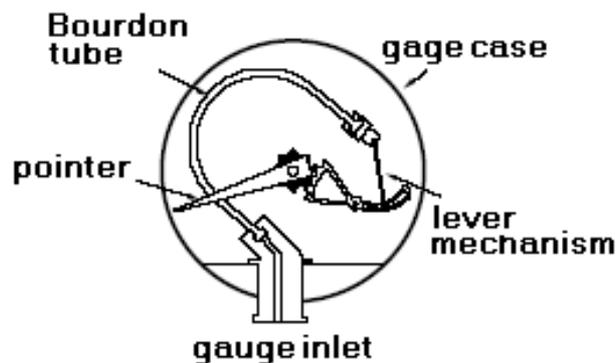
1. Mechanical - Bourdon gauge.
  - Diaphragm gauge.
2. Liquid - U-tube gauge.
  - Inclined gauge.
3. Gas compression – McLeod gauge.
4. Thermal conductivity -Pirani gauge.
  - Thermocouple gauge.
5. Ionization -Normal hot cathode gauge.
  - Bayard - Alpert gauge.
  - Penning gauge (cold cathode gauge).



## Mechanical gauge:

### 1. Bourdon tube gauge:

The Bourdon Tube Pressure Gauge is comprised of a leak tight case with a glass plate in front to allow a view of the pressure indicator dial and pressure scale. The curved metal tube elastically deforms and the end deflection is proportional to the differential pressure across its wall (think of a garden hose trying to straighten out when the water is turned on). This deflection is mechanically transformed into a rotation of the indicator dial by a rack and pinion mechanism. There are several variations of the basic design, some having evacuated cases and reference tubes that protect the mechanism inside the case from the environment of the vacuum system. Small, inexpensive Bourdon tube gauges (2" diameter faces) may be accurately read from atmospheric pressure down to 100 Torr. Larger, more sensitive gauges (8-9" diameter faces) can read down to 10Torr. Bourdon tube gauges are simple, inexpensive and relatively rugged.

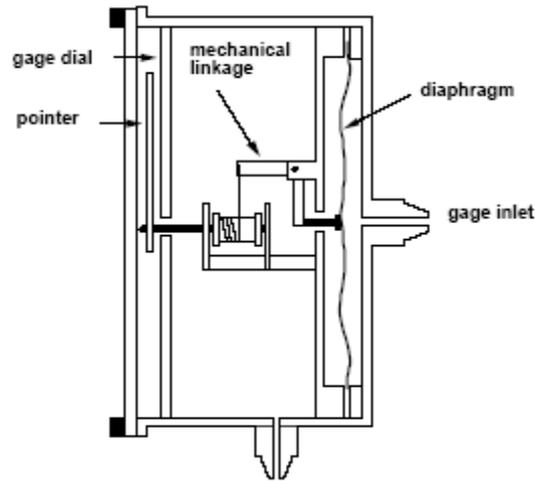


Bourdon tube pressure gauge.

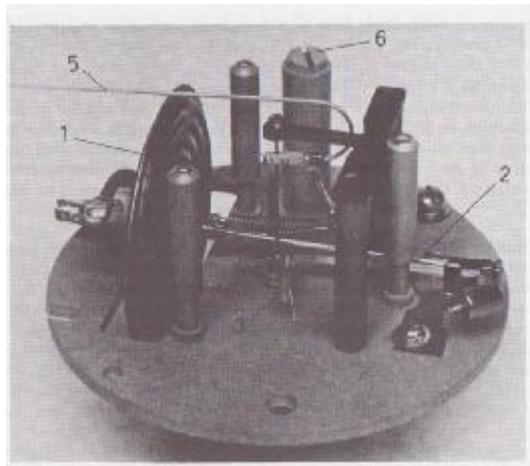
### 2. Diaphragm gauge:

#### mechanical diaphragm gauge:

The mechanically actuated diaphragm gauge makes use of a thin flexible metal diaphragm that deflects in proportion to the pressure differential across it. By means of levers and pulleys this deflection is amplified and transformed into rotational motion of a pointer in front of a calibrated dial face. Since the gauge side of the diaphragm is exposed to the environment of the vacuum system, care must be taken to control exposure of the gauge to oils, water, or reactive gases.



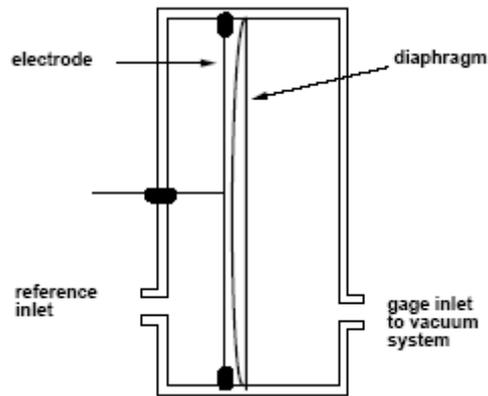
Cross-section of a mechanical diaphragm gauge



Diaphragm gauge with mechanical indication. 1.Pressure sensitive element;  
2.Push rod; 3.Geared sector; 4.Pinion; 5.Pointer; 6.Zero setting adjustment.

Capacitance diaphragm gauge:

Capacitance diaphragm gauges, or capacitance manometers, are another variety of pressure gauge that rely upon the pressure differential across a flexible diaphragm as a means of pressure measurement. In this gauge, the flexible diaphragm is made the variable element in a three-terminal capacitance potentiometer; for a given input voltage, the change in capacitance as a function of diaphragm deflection is measured, and translated into pressure units. The absence of mechanisms with backlash and counter forces means superior accuracy, repeatability over a mechanical diaphragm gauge. Capacitance manometer heads are available in a series of sensitivities; the less sensitive models being more rugged. Some of the most sensitive units can measure pressures as low as  $10^{-5}$  Torr. These gauges measure pressure as an aggregate kinetic manifestation of the molecules and hence are not gas-species sensitive.



Capacitance gauge head in cross-section.

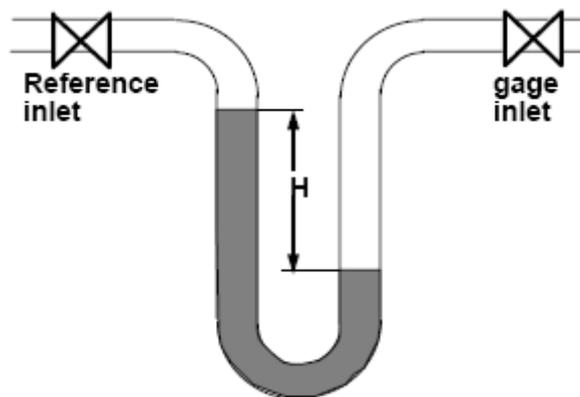
**Gauge using liquids:**

**1.U-tube manometers:**

Manometer using liquids consist of a U-tube partly filled with liquid (water , mercury, oil), having one end connected to the system in which the pressure is to be measured. The other end is either open to some reference pressure (usually atmospheric) or is closed off. The open end is connected to the system, so that the difference in level between the surfaces of the liquid in the two arms will be just proportional to the total pressure in the system. The difference in the level (h) is related to the pressure according to:

$$P = g \rho h$$

Where P is the pressure (dyne/cm<sup>2</sup>), ρ the density of the liquid (g/cm<sup>3</sup>), h (cm), and g = 980.7 cm/sec<sup>2</sup>. When the liquid is mercury, h, expressed in millimeters, is by definition equal to the pressure in Torr.



U-tube manometer

For lower values of the pressure, various causes produce errors:

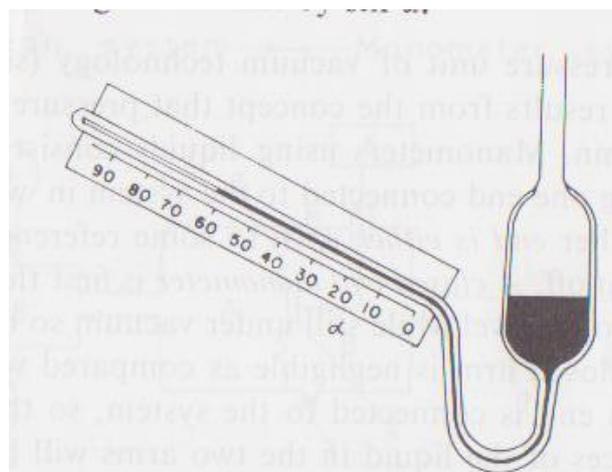
- 1.liquid sticking to glass (variable capillarity).
- 2.irregular light refraction in the glass.
- 3.dissolution of gases in the liquid(especially in oil).
- 4.temperature difference.

## 2.Inclinde manometers:

The scale of manometer can be extended by constructing it on an inclined side of the U-tube. In this arrangement the pressure P(Torr) is given by:

$$P = n [1 + (A_i/A_p)] \sin \alpha$$

Where n is number of millimetric division on the inclined scale,  $A_i$  and  $A_p$  are the cross section of the inclined branch and of vertical (pressurized) branch respectively, and  $\alpha$  the angle of the inclined branch to the horizontal. The ratio  $A_i/A_p$  has usually values of the 1/200, thus it can be neglected, and the magnification is given in fact by  $\sin \alpha$ .

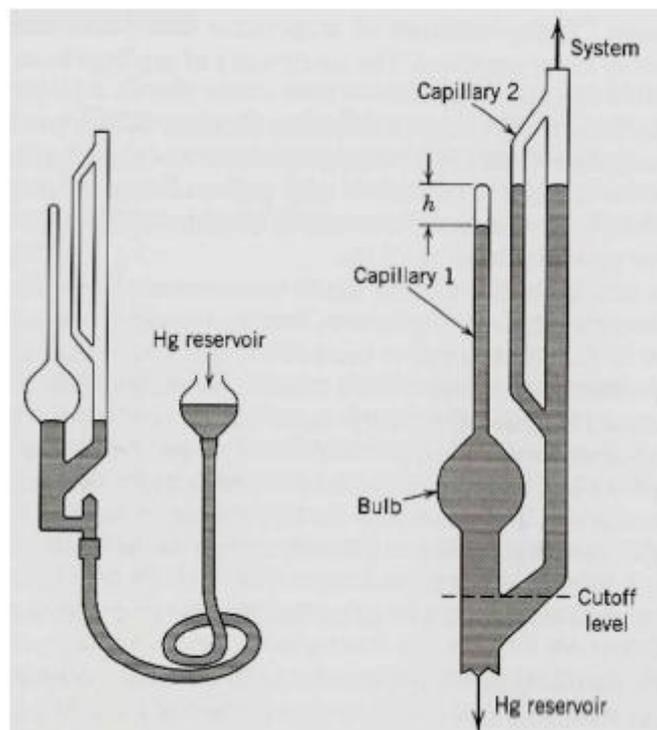


**Inclined manometer**

### Gas compression -McLeod gauge:

The pressure range over which liquid level gauges read can be extended if a sample of the gas to be measured is isolated from the vacuum vessel and compressed in a well controlled manner to amplify the force per unit area thus making the pressure easier to measure accurately. A McLeod gauge accomplishes this through the use of a movable mercury reservoir, a bulb of known volume, a set of capillary tubes and a tube allowing for connection to the vacuum vessel. Lowering the mercury reservoir will allow gas from the vacuum vessel to fill the bulb of known volume situated directly below the closed capillary. This sample of gas is then isolated from the vacuum vessel by the rising mercury reservoir at the cutoff level. At this same time, the captured gas is compressed into the sealed capillary tube. It can be shown for a calibration constant  $k$ ,

$$P = k h^2$$

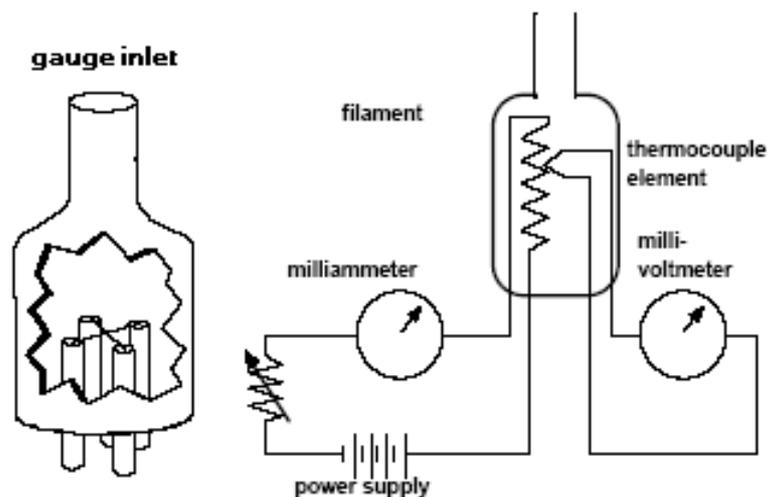


McLeod Gauge principle

## Thermal Conductivity Gauges:

### 1. Thermocouple Gauge:

In this gauge a filament is heated electrically and its temperature is measured directly by means of thermocouple. The heating current which is passed through the hot filament is kept constant at a standard value independent of the temperature of the filament. As the pressure increased, the heat conduction through the gas increases and the temperature of the filament decreases. The thermocouple (usually spot welded to the midpoint of the filament) responds to the temperature of the filament and provides a direct reading of the pressure.



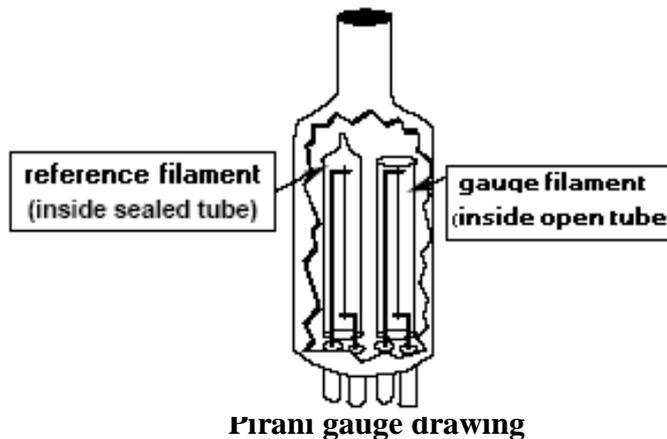
**Cutaway view of a thermocouple gauge and a schematic of the gauge and control circuitry.**

Thermocouple gauges are very widely used in the vacuum industry due to their low cost, ease of installation, use, ruggedness, and small size. The major disadvantage of the gauge is its inherent slow response to pressure change.

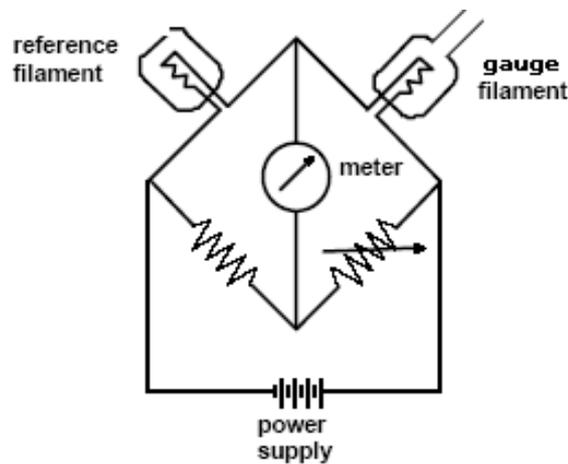
### 2. Pirani Gauge:

In the Pirani gauge, the reference filament (or compensator) is enclosed in a leak tight glass envelope evacuated to a pressure of less than 1 Torr. In a similar glass envelope which is open at the gauge inlet end is housed the gauge filament. As gas density exposed to the gauge filament changes, the gauge filament, which is heated using a constant electrical current flow, experiences a change in electrical resistivity and this is measured in the Wheatstone bridge circuit and displayed in

pressure units on the readout dial. As with other gauges that measure the thermal conductivity of gases, the Pirani gauge does not read pressure changes instantaneously. Some time is required for the heated filament to respond to changes in its environment.



**Pirani gauge drawing**



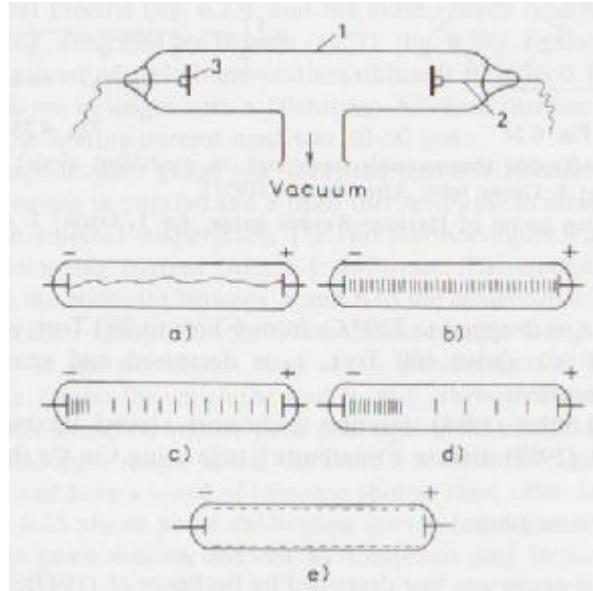
**Pirani gauge control circuit**

### **Ionization gauge:**

#### **The discharge tube:**

The discharge tube is an elementary form of ionization gauge in which a potential difference of several thousand volts is applied between two electrodes in a narrow glass tube connected to the vacuum system. The ionization in the tube produces a glow discharge, whose form is characteristic for the pressure existing in the tube. The color of the discharge is characteristic for kind of gas existing in the tube. At pressure 1 to 20 Torr, a spark (steamer) of discharge passes from one electrode to the other. At about 1 Torr the spark widens to a glow discharge. As the pressure is still further decreased definite regions (striations) in the glow discharge can be observed. When the pressure reaches

about  $10^{-2}$  Torr the number of collision is not sufficient to maintain an easily visible discharge. The electron, however, bombard the wall of the tube fluorescence of the glass may be observed. The fluorescence disappears at about  $10^{-3}$ Torr, a condition which is known as "black-out".



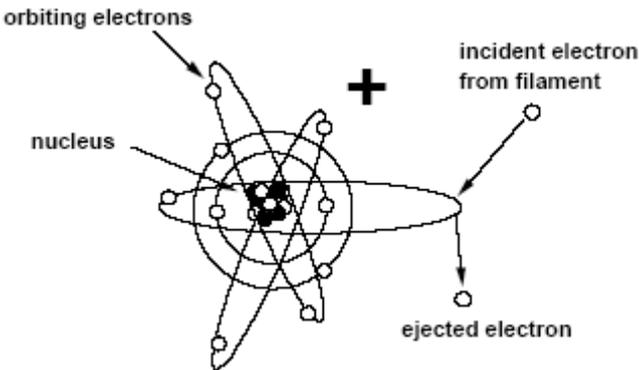
**The discharge tube and the appearance of the discharge at various pressure 1.Glass tube; 2.Current lead-in; 3.Electrode(Ni). (a) Spark = 5Torr; (b) Discharge of color specific to gas = 1 Torr; (c) Striations =  $5 \times 10^{-1}$  Torr; (d) Diminishing striations =  $1 \times 10^{-1}$  Torr; (e) Fluorescence of glass wall =  $10^{-2}$  Torr.**

To improve the correlation between the observation of the glow discharge and the pressure, two main techniques have been adopted. The first technique is to measure the applied potential difference across the discharge tube in terms of the length of the spark between polished metals spheres of a given size. The second technique is to include a fluorescent screen in the discharge tube, and use the intensity of the luminescence of this screen as the indicator of the pressure.

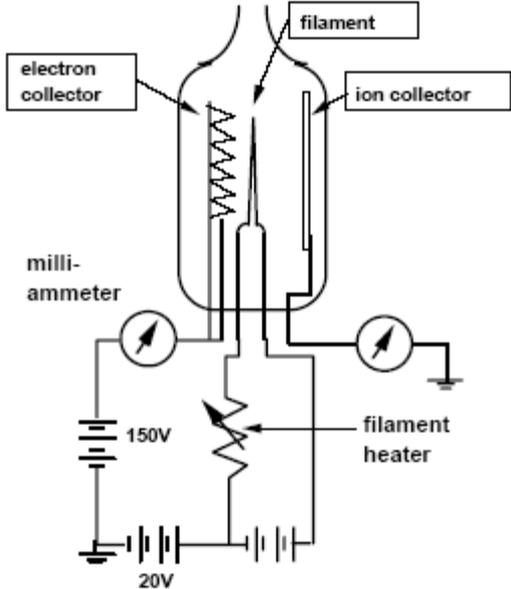
### **Hot-cathode ionization gauges:**

Also called the Bayard-Alpert gauge, the hot cathode ion gauge is a simple and reliable gauge that is widely used in vacuum processing industries. The triode (three electrode) design is easily understood if we examine each component independently, become familiar with its function, then see how they work together. The filament is usually a thin ribbon of tungsten which is in the shape of a coil or inverted. Low voltage electric current from the gauge power

supply is passed through the filament which heats up much like the filament in an incandescent light bulb. In addition to heat and light, the filament emits enormous quantities of electrons which can collide with gas atoms and in that collision, eject an electron from the gas atom making it an ion. Electrons from the filament are attracted to a helical "grid" or electron collector which is maintained at a positive voltage of approximately 150V with respect to the filament. The additional energy input into the electrons by the electron collector bias is to insure efficient ionization of gases in the gauge. Finally, the gas ions created are collected on an ion collector operated at zero volts with respect to the electron collector.



**a Mechanism of Electron Loss**

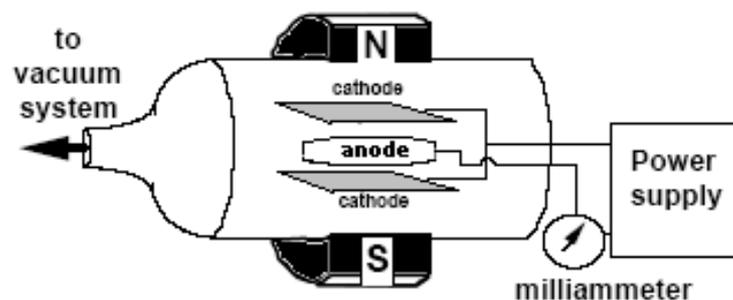


**Hot cathode ionization gauge and control circuit**

The operating range of hot cathode ionization gauges is from  $10^{-3}$  Torr to  $10^{-9}$  Torr. These gauges are small in size, relatively easy to operate and accurate to  $\pm 10\%$  of the reading in the pressure range in which they are designed to operate.

**Cold-cathode ionization gauges:** (the penning gauge)

Two parallel connected cathodes are used and midway between them is placed the anode. The cathodes are metal plates while the anode is a loop of metal wire whose plane is parallel to that of the cathode. A potential difference of about 2 kV is maintained between the anode and the cathode. In addition a magnetic field of the order of 500 Oersteds is applied at angles to the plane of the electrodes by a permanent magnet. An electron emitted by the cathode is accelerated towards the anode by the electric field, but the action of the magnetic field causes its path to be in the form of a helix. The electron generally passes through the plane of the anode loop until its path is reversed by the electric field due to the second cathode. The electron continues to oscillate in this manner about the plane of the anode loop. Due to the very long path of the electron the ionization probability is high even at low pressure. The positive ions created are captured by the cathodes, producing an ion current in the external circuit.

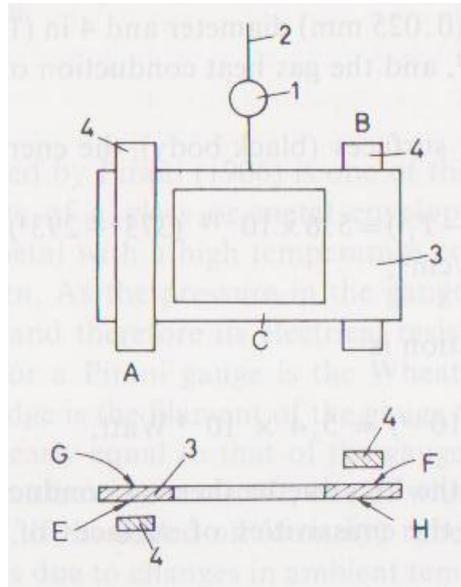


**Penning gauge**

**Radiometer (Knudsen) gauge:**

The Knudsen gauge consist of a light vane C supported vertically at its center point by a torsion wire, and of two plates A and B heated to temperature  $T_1$ . Surface E and F receive molecules of velocities corresponding to  $T_1$  while

surfaces G, H molecules from the walls of the vessel, at T. if  $T_1 > T$  there is a net couple on the vane, and the resultant torsional twist in the suspension wire is measured by the conventional mirror, lamp and scale.

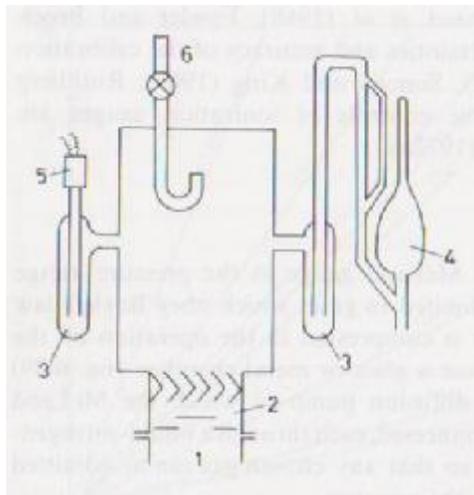


**Knudsen gauge**

## Calibration of vacuum gauge:

### McLeod gauge method:

Calibration may be effected against a McLeod gauge in the pressure range  $10 - 10^{-5}$ . the usual practice is to use a glass or metal chamber evacuated by a liquid nitrogen-trapped diffusion pump to which the McLeod gauge and the gauges to be calibrated are connected, each through a liquid-nitrogen-cooled trap. A needle valve is provided so that any chosen gas be admitted to the system at a controlled rate to vary the pressure.



**System for calibrating vacuum gauges against a McLeod gauge. 1.Diffusion pump;  
2.Liquide nitrogen and water-cooled baffles; 3.Liquid nitrogen trap; 4.McLeod gauge;  
5.Gauge to be calibrated; 6.Dry gas inlet.**

## **High vacuum technology:**

### **General:**

For the construction of vacuum systems or vacuum devices it is conventional to use metals, glasses, ceramics, and some rubbers and plastics. The materials that become part of the vacuum system, forming the enclosure (vessels, pipes) must have sufficient mechanical strength to withstand the pressure difference, must be impermeable enough to gases, must have low vapour pressures, and good resistance to special working conditions.

### **Mechanical strength:**

Vacuum enclosure are made up from cylindrical, plane and hemispherical parts. All these parts tend to deform inward as a result of the difference between external (atmospheric) and internal pressures. Cylindrical parts tend to collapse easier if their length is greater than the critical length  $L_c$  defined by

$$L_c = 1.11D(D/h)^{1/2}$$

Where D is the mean diameter and h the wall thickness.

### **Permeability to gases:**

The metallic, glass or rubber of vacuum vessels or pipes are more less permeable to gases. The quantity of gas which permeates the walls can be really large as in the case of porous ceramics or casting or low as for the case of gas diffusion through " non-porous" walls.

### **Vapour pressure and gas evolution:**

The materials used in vacuum systems should have a low vapour pressure at the maximum working temperature. Some metals (Zn, Cd, Pb) have at 400-500C vapour pressures exceeding the pressures required in high vacuum system and therefore these metals (or their alloys) cannot be used. For ultra-high vacuum work the choice of metals is only stainless steels, high nickel alloys. The gas evaluation from the metal surface should be low. To meet this requirement

previously degassed materials are recommended, and the outgassing rate should be decreased by cleaning and baking.

Material	Pressure (Torr)				
	760-1	$1-10^{-2}$	$10^{-2}-10^{-5}$	$10^{-5}-10^{-7}$	$10^{-7}-10^{-10}$
Iron, steels	good	good	good	only after degassing	only stainless steels
Cast iron, copper or aluminum	good	good	bad	bad	bad
Rolled copper or alloys	good	good	good	only after degassing	only OFHC copper
Nickel and alloys	good	good	good	good	good
Aluminum	good	good	only after degassing		not recommended
Glass, quartz	good	good	good	good with degassing	only thick-walled
Ceramics	good	good	only with vitreous coating		only special types
Mica	good	good	only after strong degassing		not recommended
Rubbers	good	good	only degassed	bad	bad
Plastics	good	only special types		only Teflon, Araldite	not recommended

### Useful range of materials for vacuum vessels and pipes

#### Glass vessels and pipes:

Glass is used as the envelope of many vacuum devices (lamp, electron tubes), as bell jars in small evaporation plants, as reaction vessels and connection pipes, and in the construction of some diffusion pumps and gauges. The vacuum range covered by glass is quite large. If the glass is exposed to steady temperature differences between the two faces, thermal gradients are developed through the glass. These are even more dangerous to the integrity of the glass than sudden cooling.

#### Cleaning techniques:

Cleaning generally means the removal of undesirable materials lying on the surface. In vacuum technology, the cleaning must be regarded not only as the

removal of the visible dirt from the surface, but including the subsequent removal of all the contamination physically stuck on the surface (oil, grease, dust) or resulting from a chemical reaction (oxides, sulphides). The degree of cleanliness must be higher, for higher vacuum. The oxides and other similar surface layers can be removed by mechanical and / or chemical methods, as abrasive blasting, wire brushing or pickling and etching.

Table 7.5.  
Pickling solutions.

Metal (Alloy)	Pickling solution	Remarks
Aluminum	NaOH (10 % sol.) saturated with NaCl (if blackening appears, Al has Cu; in this case subsequent pickling in HNO <sub>3</sub> (20–30%) required; good washing)	At 80°C, 15–50 sec. Subsequent immersion in HCl (10%) for shining surface.
	NiCl <sub>2</sub> (25% sol.) diluted 5 : 1 with HCl (1.16)	50–100 V, up to 600 V 3–15 mA/cm <sup>2</sup> , 20–40 V about 30 min.
	Electrolytic etching in sol. of 100 g H <sub>3</sub> BO <sub>3</sub> in 1000 ml dist. water plus 0.5 g Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> Electrolytic etching in 5–10% sol. of chromic acid	
Beryllium	NaOH (or KOH) 50–100 g/1000 ml. dist. water at 20°C, electrolytic etching	2.5–7 A/dm <sup>2</sup>
Constantan (CuNi55/45)	H <sub>2</sub> SO <sub>4</sub> (10% sol.) 50–60°C	
Copper	250 ml. HNO <sub>3</sub> (1.40) with 600 ml. H <sub>2</sub> SO <sub>4</sub> (1.83) and 20 ml. HCl (1.16) in 130 ml. dist. water	Bright dip
	500 ml. HNO <sub>3</sub> (65%) with 500 ml. H <sub>2</sub> SO <sub>4</sub> (conc.) and 10 ml. HCl (37%) and 5 g carbon black	Immersion (2–3 sec) immediate rinsing
	1000 ml. HNO <sub>3</sub> with 1000 ml. H <sub>2</sub> SO <sub>4</sub> and 15 g NaCl and 20 g carbon black. To be diluted 1 : 1 with dist. water 24 hr before use	Immersion (1–5 sec)
	10% Fe <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> in citric acid (0.1–1.0%) or in acetic acid (0.3–0.5%) HCl dil. immersion for 5 min followed by immersion in sol. of 100 g Cr <sub>2</sub> O <sub>3</sub> , 7 ml. H <sub>2</sub> SO <sub>4</sub> (conc.) in 1000 ml. dist. water	Bright dip
Invar (FeNi 64/36)	Sol. (1) : 40 ml. H <sub>3</sub> PO <sub>4</sub> , 15 ml. HNO <sub>3</sub> , 1.5 ml. HCl, 20 g NH <sub>4</sub> NO <sub>3</sub> , 45 ml. dist. water. Immersion for 3–4 min. After rinsing immersion in Sol (2); 65 ml. glacial acetic acid, 30 ml. H <sub>3</sub> PO <sub>4</sub> (1.75), 5 ml. HNO <sub>3</sub> (1.42). Immersion about 1 min until gas evolves evenly all over Cathodic etching in 1 vol. HCl (37%) with 1 vol. H <sub>2</sub> SO <sub>4</sub> (96%) and 1 vol. HNO <sub>3</sub> (70%) in 1 vol. dist. water. 26 mA/cm <sup>2</sup> . Carbon anode	Sol. 1 at 35°C Sol. 2 at room temp. Bright dip

(contd.)

Metal (Alloy)	Pickling solution	Remarks
Iron	50% sol. of HCl or 5-15% sol. of H <sub>2</sub> SO <sub>4</sub> . Recommended to add hydrogen evolution inhibitor (e.g. Ferrocleanol).	
Iron-Chromium	500 g Cr <sub>2</sub> O <sub>3</sub> with 5 ml. H <sub>2</sub> SO <sub>4</sub> filled up with dist. water to 1000 ml. Anodic etching in 335 ml. acetic acid with 240 ml. perchloric acid (HClO <sub>4</sub> ) in 100 ml. dist. water. 6 V, Cathode of graphite	8-10 min
Stainless steel (FeNiCr)	1-3% HNO <sub>3</sub> (conc) with 25% HCl (1.16) in dist. water. Temp. 65° C 7% H <sub>2</sub> SO <sub>2</sub> (1.83) with 3% HCl (1.16) in dist. water. Temp. 65° C 30 pbw Fe <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> with 16 pbw HF (48-52%) in 380 pbw dist. water. Temp. 70° C 27 pbw HCl (1.16) with 23 pbw H <sub>2</sub> SO <sub>4</sub> (1.83) in 50 pbw dist. water. Immersion 60 min at 45° C, followed by immersion for 20 min in a sol. of 11 pbw H <sub>2</sub> SO <sub>4</sub> (1.83), 13 pbw HCl (1.16), 1 pbw NO <sub>3</sub> H (1.40) in 75 pbw dist. water, at 60° C.	Bright dip
Kovar (FeNiCo)	1 pbw HNO <sub>3</sub> (65%) with 1 pbw acetic acid (50%) 75 g (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> in 100 ml. H <sub>2</sub> SO <sub>4</sub> (20%) at 50-100° C 1 vol. HCl (10%) with 1 vol. HNO <sub>3</sub> (10%) at 70° C. Stirring required Electrolytic etching: 1% NaCl in HCl (10-15%) a.c. 10-12 V, 1.6 A/cm <sup>2</sup> . Electrode graphite or Kovar	About 50 sec 3-5 min 2-5 min
Molybdenum	HF (40%) or HCl (5-8%) 2000 ml. H <sub>2</sub> SO <sub>4</sub> with 37.5 g CrO <sub>3</sub> , 100 ml. HF, 10 ml. HNO <sub>3</sub> (conc) at 90° C 10 g NaOH in 750 ml. dist. water with 250 ml. H <sub>2</sub> O <sub>2</sub> (30-35%) at 40° C Electrolytic etching in 20% KOH sol. d.c. 7.5 V. Carbon electrode	10 sec 2-5 min

Metal (Alloy)	Pickling solution	Remarks
Nickel	HNO <sub>3</sub> sol. 10%, at 70° C 150 ml. H <sub>2</sub> SO <sub>4</sub> (1.83), 225 ml. HNO <sub>3</sub> (1.3), 3 g NaCl in 100 ml. dist. water, at 20-40° C Electrolytic etching in sol. of 130 ml. H <sub>2</sub> SO <sub>4</sub> , 25 g NiSO <sub>4</sub> in 200 ml. dist. water 6-12 V, nickel electrode Acetic acid sol. 10%	1-2 min 5-20 sec
Tantalum	Hot HF Anodic etching in 75-98% H <sub>2</sub> SO <sub>4</sub> (or HCl) with 2-7% HF, in dist. water. 40-160 mA/cm <sup>2</sup>	1-2 sec
Tungsten	50 ml. HNO <sub>3</sub> with 30 ml. H <sub>2</sub> SO <sub>4</sub> in 20 ml. dist. water Boiling H <sub>2</sub> O <sub>2</sub> (3% sol.) Anodic etching in 250 g KOH and 0.25 g CuSO <sub>4</sub> in 1000 ml. dist. water	

Pickling is the chemical removal of oxides and other surface layers, leaving the cleaned part with a metallic appearance with a smooth or rough finish, depending on the concentration of the solution and the pickling time. After pickling the part should be always thoroughly rinsed and subsequently neutralized in an alkaline bath, and dried with hot air.

Electrolytic etching and polishing is the anodic (or cathodic) treatment of metal surfaces in appropriate etching solution.

Metal (Alloy)	Bath composition (ml.)	Voltage (V)	Current density (mA/cm <sup>2</sup> )	Temp (°C)	Time (min)
Aluminum	40 ml. H <sub>2</sub> SO <sub>4</sub> , 40 ml. phosphoric acid 20 ml. dist. water	10-18	720	95	5
	165 ml. perchloric acid, 785 ml. acetic acid, 50 ml. dist. water	50-100	30-50	<50	15
	45 ml. perchloric acid, 800 ml. ethanol, 155 ml. dist. water	100-200	2000-4000	<35	30
Beryllium	100 ml. orthophosphoric acid, 30 ml. H <sub>2</sub> SO <sub>4</sub> , 30 ml. glycerine, 30 ml. ethanol	—	2000-4000	—	—
Copper	670 ml. orthophosphoric acid, 100 ml. H <sub>2</sub> SO <sub>4</sub> , 270 ml. dist. water	2-2.2	100	22	—
	7 g CrO <sub>3</sub> , 22 g sodium dichromate, 7 ml. acetic acid, 6 ml. H <sub>2</sub> SO <sub>4</sub> , 58 ml. dist. water	20-60	—	—	—
Iron	530 ml. orthophosphoric acid, 470 ml. dist. water	0.5-0.2	6	20	10
Molybdenum	35 ml. H <sub>2</sub> SO <sub>4</sub> , 140 ml. dist. water	12	—	50	—
Monel	200 ml. HNO <sub>3</sub> , 400 ml. methanol	2.4-2.6	125-150	20-30	10
Nickel	60 ml. orthophosphoric acid, 20 H <sub>2</sub> SO <sub>4</sub> 2 dist. water	10-18	900	60	5
	210 ml. perchloric acid, 790 ml. acetic acid	22	180	20	—
Carbon steel	50 ml. H <sub>2</sub> SO <sub>4</sub> , 40 ml. glycerol, 2 ml. HCl 8 ml. dist. water	10-18	50	10	60
	185 ml. perchloric acid, 765 ml. acetic acid, 50 ml. dist. water	50	40-70	<30	5-10
Stainless steel	50 ml. H <sub>2</sub> SO <sub>4</sub> , 40 ml. glycerol, 10 ml. dist. water	10-18	300-1000	30-90	3-9
	133 ml. glacial acetic acid, 25 g CrO <sub>3</sub> , 7 ml. dist. water	20	900-2500	18	4-6
Tantalum	90 ml. H <sub>2</sub> SO <sub>4</sub> (conc), 10 ml. HF	—	100	34-45	9
Tungsten	100 g NaOH, 900 ml. dist. water	—	30-60	20	20-30

### **Cleaning of glass:**

The glass may have rough appearance due to weathering or to devitrification. Weathering is a result of the influence of the atmospheric vapour, and consists of the hydrolysis of the alkali silicates, forming alkali hydroxides and colloidal silicic acid. The alkali hydroxides react with the carbon dioxide from the air, forming a film of alkali carbonates, with separation of silica. Due to this process the glass loses its transparency and becomes brittle.

Glass vessels and pipes (new or weathered) can be washed by immersing them in hydrochloric acid solution (1 – 5%) for 3 – 10 sec, followed by a subsequent rinsing in water (40 – 50 °C) and drying.

Chromic acid is satisfactory for cleaning glass (subsequent to a washing in water). The usual cleaning solution known as chromic acid contains about 50 ml of saturated aqueous sodium dichromate in a liter of concentrated sulphuric acid. The chromic acid solution should be used only if it has its brown color. If the color is changed the solution is decomposed.

A solution which is much more effective than the chromic acid solution consists of 5% HF with 35% HNO<sub>3</sub> in 60% water.

Acetone or alcohol may also be useful for drying glass parts.

### **Cleaning of ceramics:**

Suitable cleaning of ceramic parts is obtained by firing the ceramic parts in air at 800 – 1000 °C. alternatively an alkaline cleaning solution can be used, followed by immersion in dilute nitric acid (2 – 5 min). chromic acid or other glass cleaning solution are also satisfactory.

### **Cleaning of rubber:**

The rubber must be cleaned with a solution of KOH (20%) at 70 °C with subsequent washing with distilled water and drying with clean air, and/or degassing in vacuum at 70°C for 4 – 5 hours.

## **Baking:**

The most efficient method of reducing the out gassing rates of the parts of vacuum systems is their baking. The useful range of baking temperature is 400 – 500 °C (for metal or glass systems), and the efficiency is much reduced if only 150 – 200 °C is used .

## **Thin Film Deposition Processes**

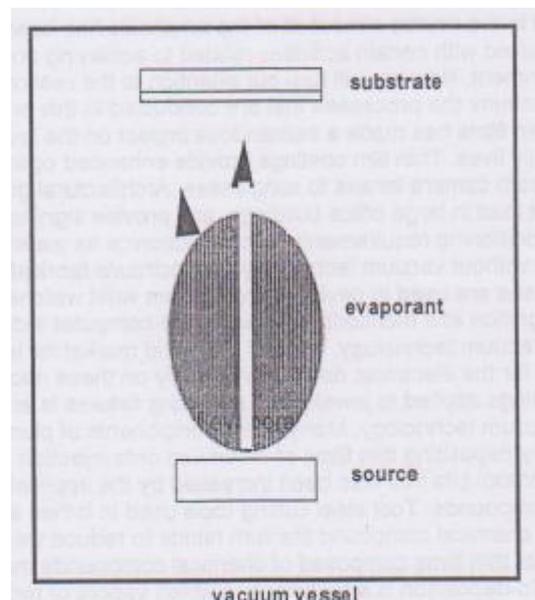
Thin film coatings provide enhanced optical performance on items ranging from camera lenses to sunglasses. Architectural glass is often coated to reduce the heat load in large office buildings, and provide significant cost savings by reducing air conditioning requirements. Microelectronics as we know them today would not be possible without vacuum technology. Microcircuits fabricated in multi-step vacuum processes are used in devices ranging from wrist watches to microwave ovens to automobile ignition and monitoring systems. The computer industry would not exist if it were not for vacuum technology. In 1990 the world market for integrated circuits was \$50 billion; and for the electronic devices which rely on these microcircuits, \$0.9 trillion.

Decorative coatings applied to jewelry and plumbing fixtures is another large industry based upon vacuum technology. Many of the components of plumbing fixtures are manufactured by depositing thin films of chromium onto injection molded plastic parts. The useful life of tool bits has also been increased by the application of thin films that are chemical compounds. Tool steel cutting tools used in lathes and mills are often coated with the chemical compound titanium nitride to reduce wear of the cutting edges. The deposition of thin films composed of chemical compounds may be performed in several ways. Co-deposition is a technique in which vapors of two different materials are generated simultaneously. These two vapors condense together, forming an alloy or compound. Other techniques for deposition of compounds include thermal evaporation of the compound (as is performed for salt coatings), sputtering of the compound, and reactive sputtering or evaporation. In the

reactive processes, atoms of the evaporant (typically a metal) chemically react with gas species which are intentionally injected into the process chamber. Each of these processes will be described in detail.

### **Thin Film Deposition in a Vacuum Environment**

The techniques for thin film deposition have evolved in approximately this order: thermally induced evaporation (by electrical resistance heating, induction heating, and electron beam heating), sputtering (diode, triode, magnetron, ion beam), arc processes, and most recently, laser ablation. In general, there are three steps in any physical vacuum deposition (PVD) process: creation of an evaporant from the source material, transport of the evaporant from the source to the substrate (item to be coated), and condensation of the evaporant onto the substrate to form the thin film deposit. There are two reasons why this process is best conducted under vacuum: 1. the process of evaporation involves significant amounts of heat, if oxygen were present, any reactive metal would form oxides; 2. collisions with gas molecules during the transport of evaporant from source to substrate would reduce the net deposition rate significantly, and would also prevent growth of dense films.



The three basic steps in any physical vapor deposition process:  
evaporation from the source, transport of evaporant, and condensation of the  
evaporant

Upon arrival at the substrate, evaporated material condenses on the substrate in a complex sequence of events that determine many of the physical properties of the deposited film. The steps in the growth of thin films are generally referred to as nucleation and growth. In nucleation, the atoms and molecules which are arriving (called ad atoms) at the surface lose thermal energy to the surface, and the surface absorbs that energy. Depending on the amount of thermal energy the ad atoms and the surface have, the ad atoms move about on the surface until they lose the thermal energy required to move about the surface (referred to as Adam mobility). As nuclei continue to form, the film grows into a continuous sheet covering the substrate. Chemical interactions between the ad atoms and the surface determine the strength of the bond between the film and substrate. Gold, for example, does not form a chemical bond with silicon dioxide, and therefore, the adherence of gold films on glass are very weak. Improvement of this adhesion may be made by first depositing a thin (500Å thick) "Binder" layer of chromium or niobium, then depositing the gold over the binder layer.

Chromium and niobium do form chemical bonds with the silicon dioxide in glass, and also form metallic bonds with the following gold layer. Once a few monolayers of evaporant have condensed on the substrate, the film continues to grow in thickness as if the entire substrate were made of the material being deposited. During film growth the microstructure of the deposited film will be developed. This microstructure may be described in terms of grain size, orientation, porosity, impurity content, and entrained gases. Normally, vacuum deposition processes are selected over other processes (electrochemical deposition, flame spraying, etc.) to achieve the following desirable properties:

- 1) High chemical purity.
- 2) Good adhesion between the thin film and substrate.
- 3) Control over mechanical stress in the film.
- 4) Deposition of very thin layers, and multiple layers of different materials.
- 5) Low gas entrapment.

For each of the vacuum deposition process described in this unit, keep in mind

the ultimate goal is to provide a means for depositing a thin film having the required physical and chemical properties. The parameters one can control to achieve the specified goals are:

- 1) Kinetic energy of the ad atoms.
- 2) Substrate temperature.
- 3) Deposition rate of the thin film.
- 4) Augmented energy applied to the film during growth.
- 5) Gas scattering during transport of the evaporant.

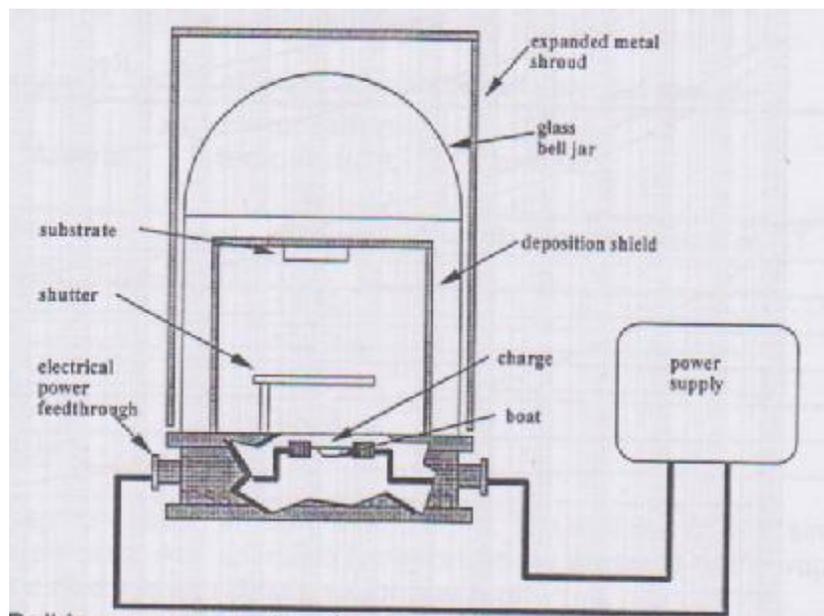
By varying these parameters one can generate thin films of a given material that have different mechanical strength, adhesion, optical reflectivity, electrical resistivity, magnetic properties and density.

### **Thermally Induced Evaporation**

In this process, heat is input into the source material (often called the charge) to create a plume of vapor which travels in straight-line paths to the substrate. Upon arrival at the substrate, the atoms, molecules, and clusters of molecules condense from the vapor phase to form a solid film. The heat of condensation is absorbed by the substrate. On a microscopic scale the localized heating from this process can be enormous. It is common, in the development of metal coating techniques for thin cross-section plastic parts, to melt substrates during the initial deposition runs. With experience, one can select source-to-substrate distances and deposition rates which will allow coating of temperature sensitive substrates without melting. There are several methods by which heat can be delivered to the charge to cause vaporization: electric resistance heating, induction heating, and electron beam heating. Deposition of thin films by laser ablation and cathodic arc could be grouped in this section with thermal processes, but there are some unique characteristics of these techniques which are beyond the simple model of thermally induced evaporation. For this reason we will cover these two deposition techniques separately.

## Resistance Evaporation

Evaporation of material by electrical resistance evaporation is very likely the easiest of the thermal evaporation techniques. Quite simply, in a vacuum environment the charge (which may be an elemental metal, an alloy, a mixture or a compound) is heated to become a vapor. Low voltage, high current power is brought into the vacuum vessel using electrical power feed through. The electrical power is passed through a filament which is in intimate contact with the charge. Filaments are often heated to 1000 to 2000° C. A materials requirement for efficient thermal evaporation is that the charge have an appreciable vapor pressure at the operating temperature of the filament.

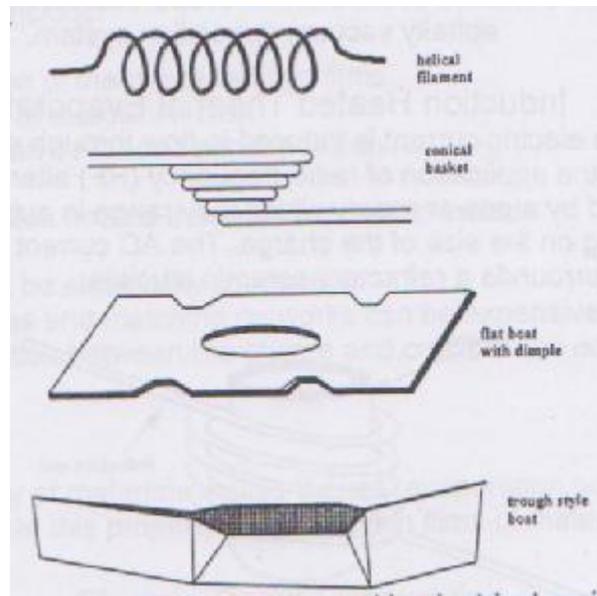


Bell jar vacuum system for deposition of thin films by electrical resistance heated thermal evaporation.

Filaments are usually made of refractory metals such as Tungsten, Tantalum, or their alloys. Some of the requirements for a good filament material are:

- 1) High melting point.
- 2) Low solubility for the charge materials.
- 3) Filament should be wettable by the charge materials.
- 4) Filament should withstand thermal shocks well.

There exists a great variety of filament configurations ranging from straight and coiled wires to "boats" and boxes:



Configurations for filaments used in electrical resistance heated thermal evaporation.