The cup actually holds the specimen & thus provides the immediate environment which is seen by the strange and any X-rays coming down the column.

Cups are made by Be to minimize the generation of X-rays that would interfere with micro analysis.

The clamping ring or screw holds the specimen in the cup also made of Be and needs to be designed carefully.

It helps in holding the specimen firmly.

It should be such that it can be tightened easily without applying under pressure on the specimen.

Brittle disksmany break while loading them.

 Two kind of retaining rings are commonly used, screw thread rings which are easier to control and do not damage metals, but they break ceramics, they transfer shear stresses to disk. Others are spring clips; they offer more control over the load that we put on the specimen.

In the modern design jewel bearing is omitted so that the holder is supported at just one pivot point.

**Top entry holder:**

Less popular since they essentially prechede XPDS analysis in the TEM, also it is more difficult to design such holder so that the specimen can be manipulated, (eg. rotated or strained).

However the biggest advance is that they are much less susceptible to drift since they are not connected directly to the outside so early HRTEM request top to entry holders.

Currently all TEM’s up to 400KV use side entry holders.

**Tilt and Rotate Holder:**

1) Single tilt holder: - it is one of the basic types it can only be tilted around axis of the rod , cheap robust and can give at least some idea of the usefulness of tilting a specimen for diffraction contrast studies

2) Quick change Holder:- also a single tilt holder that clamps the specimen with a lever arm which we raise and lower on to the disk or grid.

It does not put high stress on the sample and also doesn’t hold it very strongly either should not be used for magnetic sample but good for ceramic samples.

Different retainer can be substituted for the clamp ass shown in fig. 8.8 creating a more multipurpose holder.

3) Multiple specimen holders: - usually a single tilt holder, but up to 5 samples can be loaded in the column at a time. Fig 8.9 show two specimen double tilt version.

Such holder is good if one is not good preparing specimen and also different specimen here to be compared under identical conditions.

However, in modern TEM’s specimen exchange is relatively quick in UHV instruments, where multiholder would probably be more useful though less common.

4) Bulk specimen holder:- used for surface imaging and diffraction.

The bulk specimen is longer than the traditional 3mm disk(usually ~10 nmX5nm)

5) Double tilt-holder:- most popular holder as it gives the maximum flexibility in orienting the specimen.

The tilt axis is fixed as two orthogonal directions. In some design the cup can be removed while the specimen is in place that means the specimen can be reinserted in the same orientation. Good for robust specimens.

6) Tilt rotate holder:- Useful when we want to orient the specimen parallel to the tilt axis (along the rod).

Its strength for the side –entry holder one tilt axis is always parallel to the rod of the holder which also gives the largest tilt angle.

7)Low background holder:- up and clamping ring made of Be to minimize the generation of `brem-strahlungX-ray and characteristics X-rays, they can be double or single tilt and may be cooled also.

8) Tomography Holder: - new design holder, sample can be tilted fully through 3600. It’s ideal for looking at needles.

9) In situ Holder: - Special holder that helps or allows changing the specimen while observing it in the TEM.

One can do expels on a specimen in the TEM i.e. heat, cool, strain, twist compress etc.

**Heating Holder** – Possible to go up to ~13000 C. temp measured by thermocouple attached to cup.Temp can go higher because of longer gap between pole pieces.

Calibration should be done carefully. Temp may be different for different specimens. Material under study should not form a entectic alloy with the material forming the holder

**Cooling Holder –** available for liq-N2 or liq-He temp. Holder can be single or double tilt, they minimize surface –borne contamination.

Useful for in situ studies of superconductive material and ideal for polymer or biological tissue, these typ of holders can also acts as small cryo-pump so that it actually attracts contamination, also specimen drift is possible.

**Cryo-Transfer Holder:;-** Useful for certain specimen that are prepared at cryogenic tempsuch as liqs, latex, emulsions and tissues. This holder permits the transfer of such cold specimens into the TEM without H2O vapor from atm condensing as ice on surface.

**Straining Holder:-** clamps sample at both ends then applies a load to one end via a load cell or screw thread mechanism as in fig 8.11.

Sample can be in shape of a small tensile specimen and it is thinned in the middle of the gauge length. The motion of dis locations, cracks etc. are then easily monitored. Load can be varied to study cyclic as well as tensile loading and the strain rate is another variable that is easily controlled.

Fig shows presence of furnace to the heat the sample while under load.

**Probing Holder: -** useful for poking the specimen

**EBIC and CL Holder: -** Essential feature in the electrical feed through that allows controlling the charge recombination in semiconductor or certain minerals specimens by applying a bias across the specimen surface.

**Plasma Cleaner: -** used for removing surface contamination and modifying surface (e.g.-changing wettability). The rod of holder is placed inside a plasma chamber (fig 8.14B).

Surface of glass, semicond and other ceramic, metals and even polymers and biomaterial can be cleaned. basic idea to remove hydrocarbon . Plasma consists of a mix of energetic electrons and ions that bombard the surface and break the C\_H bonds.

Gases used are O2, N2 and Ar, selected based on sample.

**The Vacuum: -** 10-5 Pa needed to avoid scattering of electron and also it keeps the specimen clean,

Better the vacuum less is the contaminations, vacuum represented in terms of rough (100-0.1Pa) low (0.1-10-4Pa) high(10-4 -10-7) and ultrahigh(10-7 &above)

1Torr = 130Pa, 1Bar =760Torr, equivalent to 10-5Pa, different pumps useful for different levels of vacuum.

**High and Ultrahigh Pumps: - Diffusion Pumps**

It uses a hot plate to boil oil which then forms a series of concentric vapor jets. There jets drag air molecules out of the microscope as in fig 8.2 , then condense to a cold surface, freeing the air molecule which are extracted by the mechanical pumps ‘backing’ the diffusion pumps.

While this may seem an efficient way to more air, diffusion pumps can in fact transport more than a hundred litres of air per sec, which is quite sufficient to pump out a TEM column.

With no moving parts, diffusion samples are inexpensive and very reliable, but they need external water cooling to aid condensation of vapour.

Failure of the coolingH2O supply and burn out of the hot plate are about the only possible causes of failure.

No moving parts because ensure vibration free operation.

Oil diffusion pump can contaminate the vacuum in the TEM if oil vapour were to escape into the column.

This can be minimized by using non-hydrocarbon oils with low vapour pressure. AliqN­2 cold trap sits on top of the pumpand condenses out any residual oil molecule. The cold traps should be full of liq N2to maintain a clean system.

Diffusion pumps are capable of very efficient pumping from 10-1-10-9Pa (10-11Torr) and if properly trapped, will provide a clean UHV system that is very reliable.

**Terbomolecular Pumps:-**

Uses a turbine to force gases from the microscope.

Have many moving parts at high speed(in excess) of 20,000 -50,000rpm in common so they are more liable to fail than diffusion pump.

Mechanics very simple, do not use oil so they don’t introduce hydrocarbon to contaminate the microscope, best models are quite and almost vibration free.

Modern turbo pumps are being used to prepump the specimen chamber when this is eritical as in the cryotransfer technique.

It can start (slowly) at ambient pressures increasing speed as the pressure is lowered; ultimately providing UHV conditions are high enough speeds.

It is used to back the terbopump with a dry mechanical pump

**Ion Pump:-**

Penning trap, swirling electron cloud produced by an electric discharge stored in anode region.

Ion pump do not contain oil, no moving parts.

Relies solely on the ionization process to remove air. The ion pump emits electron from a cathode. These ions spiral in a magnetic field and ionize air molecule, which are then attracted to the cathode.

The energetic gas ions sputter Ti atoms from the cathode and they condense throughout the pump chamber, mainly on the cylindrical anode, trapping gas atoms.

Thus ion pumps remove gas atoms in two ways by chemisorption on the anode surface and by electrical attraction to the cathode.

Smaller the ion current between the electrodes the lower the vacuum, so the pump act as its own vacuum gauge.

Efficient only at high vacuums, so are usually switched on after a diffusion pump has lowered the pressure to <~10-3Pa (10-5Torr).

It is common to add ion pumps directly to the stage or gun chamber of TEM’s to focus their pumping action on these important regions.

Fig shows how they operate.

**Cryoscopic(Adsorption) Pumps: -**

These pumps (Cryopump) rely on liq N2 to cool molecular sieves with large surface areas.

The cold surface efficiently removes air molecules from ambient pressure down to

10-4Pa(10-6Torr).

As they are oil free these are also used to back ion pumps and present their accidental contamination through back streaming from oil bearing pumps.

Cold surface are also used to enhance vacuums in the stage of most non-UHV-TEM, such cold fingers or anticontaminator provides an alternative site (rather than your specimen) for condensation of residual component in the vacuum.

**Exhaust Pump: -**

Mechanical, diffusion and turbo pumps are all exhaust pumps they pull in air from one end and expel it from the other.

**Trapping Pump:-**

Ion pumps and cryopumps are trapping. They keeps the air molecules within them and release them when turned off or warmed uprespectively.

**The whole System: -**

The modern TEM’s have at least 2 separate pumping systems: One that evacuates the column and one that pumps the camera and screen chamber.

Camera is pumped separately because the film is one of the pri causes of column degradation since outgassing occurs from the emulsion that contains the AgI grains, so this part of the TEM is usually pumped by a combination mechanical/diffusion pump.

The stage is oftenpumped by a separate ion pump, turbo or cryopump or combination of these,

Each part of vacuum system consists of roughening pumps (mechanical / turbo) that pumps out the appropriate part of the microscope to give the vacuum where the HV/UHV pumps start to operate controlled

1. !st connects the mechanical pumps to column.(the roughening value)
2. 2)connect the mechanical pumps to the bottom of the diffusion pump(the backing value)
3. 3)connect the diffusion pump directly to the TEM column(the butterfly value)
4. When pumping down from atmospheric pressure, 1st the mechanical pump is used to back out the diffusion pump till it get to flow enough pressure so its heater can be safely switched on without oxidizing so close

Fig 8.5 shows three value, now close1, open 2 and close 3.

When diffusion pump is warmed up, we rough out the column open 1, close2 & 3 until the column is at a low enough pressure that the diffusion pump can be used.

At this point, close1, open2 & then 3 so the diffusion pump is open to TEM and may be continuously backed by the mechanical pump.

A better approach incorporates a vacuum reservoir between the mechanical and diffusion pumps.

When the reservoir is <0.1Pa, the mechanical pump is closed off and diffusion pump exhaust in to reservoir.

 When the pressure builds in the reservoir the mechanical pump will automatically switch on and lower the pressure,

Similar arrangements works for other pumps eg a diffusion pump may be used to lower the pressure in the stage and gun sufficiently for the ion pumps to be switched on and so on.

In most TEM’s the stage and gun have significantly better vacuum then the camera region so the camera / screen is isolated from rest of the column by a differential pumping aperture.

**Leak defect:- “**Nature abhors a Vacuum” so the pumps must keep pumping the TEM leaks.

Sometime the leaks are too large for pumps to handle.

**Specimen Preparation: -**

The specimen when made for TEM analysis should be electron transparent (usually) and representative of the material you want to study.

Mostly it should be uniformly thin, stable under the electron beam and in the lab environment conducting and non-magnetic.

The specimen can be divided into two groups

1)Self- supported specimen and

2) Specimen resting on a support grid or thin washer, the grid washer is usually Cu but could be Au, Ni, Be, C and Pt.

The most imp is i.e. while preparing sample is safety.

The time for specimen preparation can be vary from few min to days.

Samples like superconductive yBa2Cu3O6+x should be crushed in mortar and pestle using a non-aqueous solvent. Catch the small particles on a carbon filling and put the specimen in the TEM.

Thin slices of sample can be cut using a diamond saw, thin it on a grinding wheel, dimple the thinned disk, then ion mil to electron transparency at liq.N2 temperature, then carefully warm the specimen to RT in a dry environment and put it in TEM.

**Safety: -**

Four favoriteliqfor polishing are HCN, HF,HNO3 and HClO**4**

TEM: -

Imaging- forming DP’s and Images

Viewing DP: - for this we have to work in the diffraction mode.

To see the DP we have to adjust the imaging. System lenses so that the BFP of the objective lens acts as the object plane for the intermediate lens. Then DP is projected on to the viewing screen / CCD.

**Viewing Image: -**

For this, the intermediate lens is readjusted so that its object plane is the image plane of the objective lens.

The image is then projected onto the viewing screen / CCD

Fig 9.12A & B. Two ways Bright Field and Dark Field Imaging.