Transmission Electron Microscopy
7. Pumps and Holders

EMA 6518
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Outline

- Vacuums
- Roughing Pumps
- High/Ultra-High Vacuum Pumps
- Diffusion Pumps
- Turbomolecular Pumps
- Ion Pumps
- Cryogenic Pumps
- The Whole System
- Leak Detection
- Contamination: Hydrocarbons and Water Vapor
- Specimen Holders and Stages

Vacuums

- Strong scattering occurs in gases
- Vacuum: A vacuum is a volume of space that is essentially empty of matter
  - enable to send coherent, controlled electron beams
  - Keep the specimen clean
  - TEM: inside the column: 10^{-7}Torr; UHV TEMs: <10^{-9}Torr

Rough vacuums: 100-0.1Pa (1-10^{-3}Torr)

Low vacuums: 0.1-10^{-4} Pa (10^{-3}-10^{-6}Torr)

High vacuums: 10^{-4}-10^{-7} Pa(10^{-6}-10^{-9}Torr)

Ultrahigh vacuums: <10^{-7}Pa (10^{-9}Torr)

Perfect vacuum: 0Torr

Human Body in a Vacuum?

- You do not explode and your blood does not boil because of the containing effect of your skin and circulatory system.
- You do not instantly freeze because, although the space environment is typically very cold, heat does not transfer away from a body quickly.
- Loss of consciousness occurs only after the body has depleted the supply of oxygen in the blood.
- If your skin is exposed to direct sunlight without any protection from its intense ultraviolet radiation, you can get a very bad sunburn.

Roughing Pumps

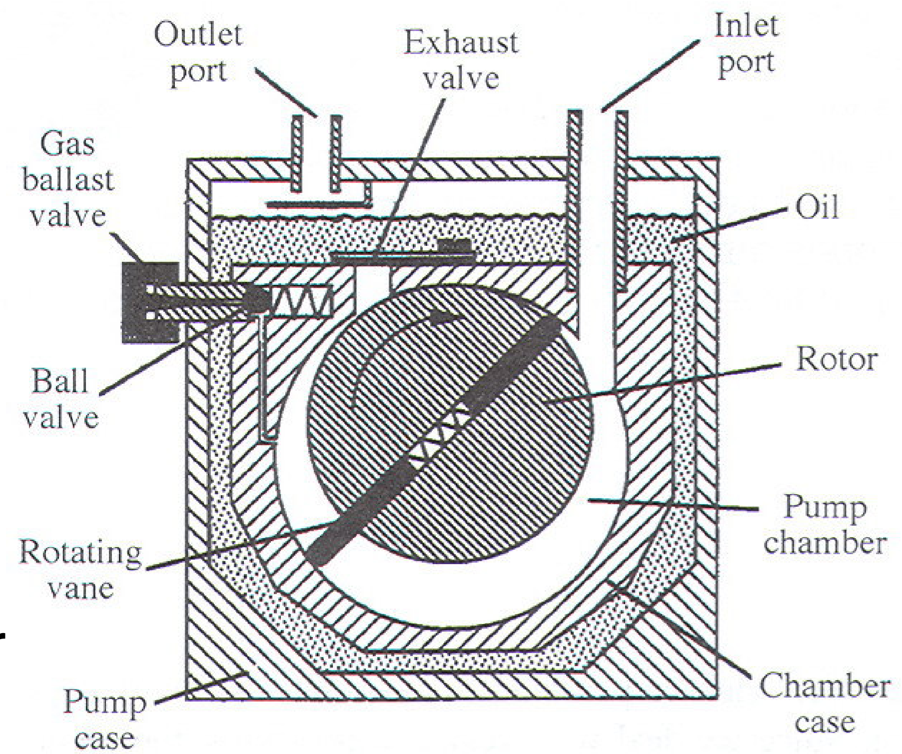
Rotary pump:

•Belt-driven

•The eccentric motion sucks air through an inlet valve into a chamber and expels it through an exit valve.

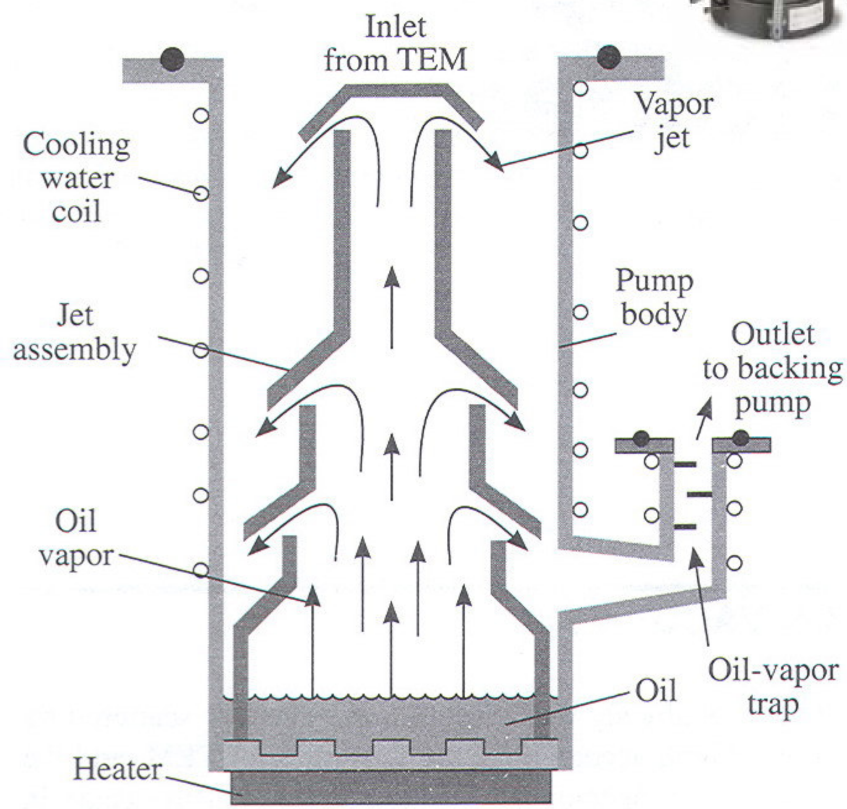
•Very reliable, relatively inexpensive, noisy and dirty

•Lower the pressure to 10^{-3} Torr



Diffusion Pumps

- It uses a hot plate to boil oil, which then forms a series of concentric vapor jets.
- The jets drag air molecules out of the microscope.
- Then condense onto a cold surface, freezing the air molecules which are extracted by the mechanical pump “backing” the diffusion pump.
- 10^{-3}-10^{-11}Torr



Turbomolecular Pumps

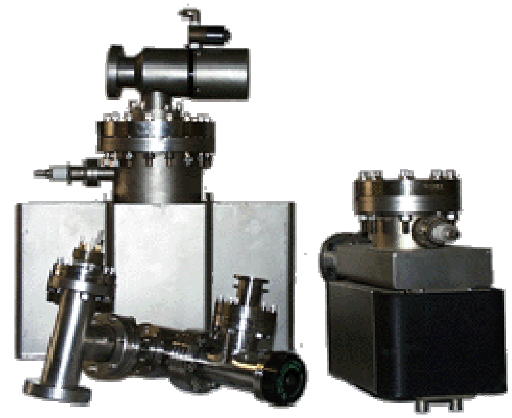
•Turbopumps use a turbine to force gases from the microscope.

•Many parts moving at high speeds (in excess of 20,000-50,000rpm)

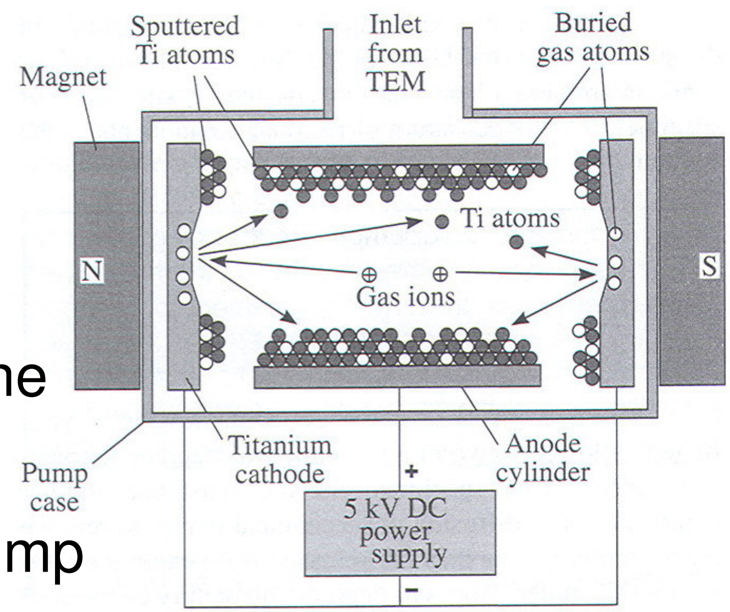
•Quite, almost vibration free, oil free
•Back the turbopump with a dry mechanical pump

•Able to generate intermediate vacuum (~10^{-4}Torr) up to ultra-high vacuum levels (~10^{-10} Torr).

Ion Pumps



- Rely on ionization process to remove air
- Oil free, No moving parts
- It emits electrons from a cathode, these electrons spiral in a magnetic field and ionize air molecules, which are then attracted to the cathode
- Ion pumps remove gas atoms in two ways: by chemisorption on the anode surfaces and by electrical attraction to the cathodes
- Usually switched on after a diffusion pump has lowered the pressure to 10^{-5} Torr



Cryogenic (Adsorption) Pumps

- Cryogenic pumps (cryopumps) rely on liquid N_2 to cool molecular sieves with large surface areas.
- The cold surface efficiently removes air molecules from ambient pressure down to 10^{-6} Torr.
- Oil free
- They are used to back out ion pumps and prevent their accidental contamination through backstreaming from oil-bearing pumps.

Remarks

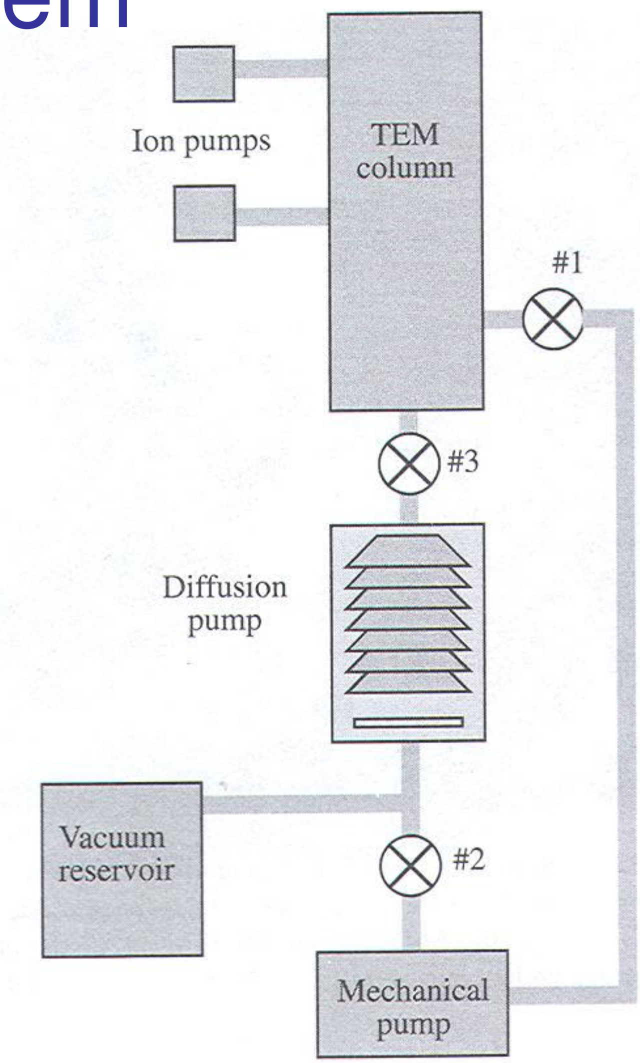
- Mechanical, diffusion, and turbopumps are all exhaust pumps; they pull in air from one end and expel it from the other
- Ion pumps and cryopumps are trapping pumps. They keep the air molecules within them and release them when turned off or warmed up, respectively.

The Whole System

•TEM has separate pumping systems. One that evacuates the column and one that pumps the camera and screen chamber.

• One of the primary causes of vacuum degradation since out gassing occurs from the emulsion that contains the AgI grains.

• There are three valves which are now all computer-controlled.

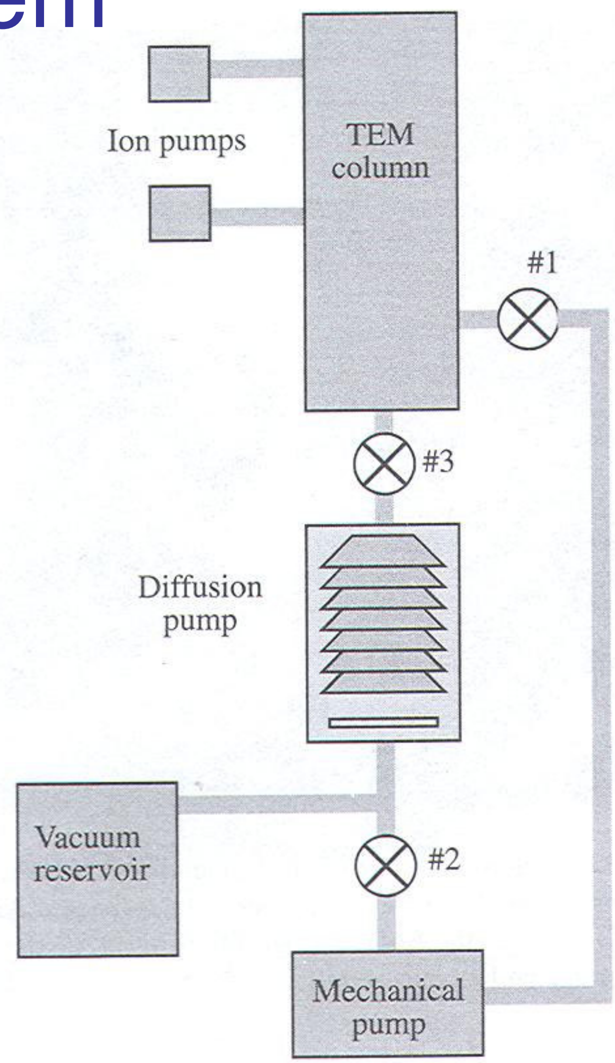


The Whole System

•Pumping down from atmospheric pressure: close #1, close #3, open #2

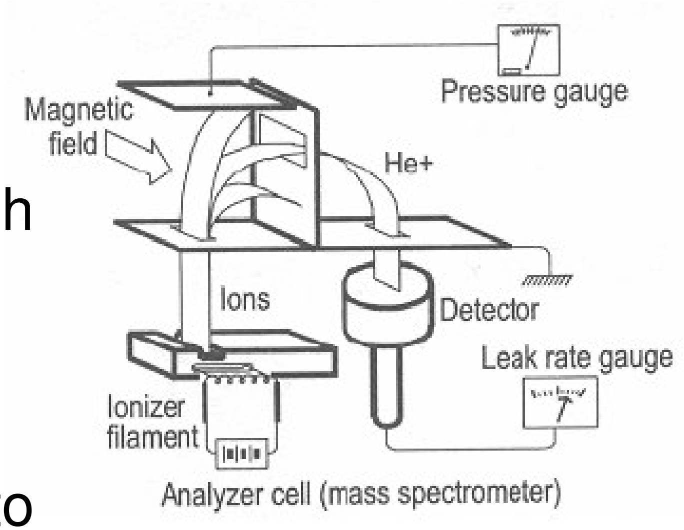
•When the diffusion pump is warmed up, rough out the column: close #2, close #3, open #1

•At a low enough pressure that the diffusion pump can be used: close #1, open #2, open #3



Leak Detection

- The most common cause of a leak is your specimen holder.
- Never touch the O-ring, make sure it doesn't dry out.
- "Bake" the column after repairing a leak (150-200ºC).
- For UHV system, you must bake to reach the ultimate vacuum, and the higher the temperature the better.
- However, since sometimes the TEM accessories, such as XEDS and EELS systems, are not designed to be baked to the same high temperature as the column.



Contamination: Hydrocarbons and Water Vapor

- The vacuum can be a source of contamination, particularly residual hydrocarbons from the pump oil which crack under the electron beam.
- Carbonaceous material then deposits on your carefully thinned specimen, making it difficult to do sensible high-resolution imaging or microanalysis.
- A clean vacuum is essential.
  - Heating the specimen to >100ºC
  - With a halogen lamp in the prepump chamber
  - Cooling the specimen to liquid-N_2 temperatures in a cooling holder
  - Using oil-free pump
  - Plasma ashing of the specimen holder and speicmen prior to insertion in the TEM
  - Never touch anything that will enter the vacuum

Specimen Holder and Stages

- The traditional side-entry holder is a rod with a motor attracted to tilt and/or rotate the specimen and a lead connecting it to a power supply and control box, or liquid-N_2 dewar
- The traditional top-entry holder is a cartridge which you load into the TEM but is detached from the outside world when you use the microscope
- Side-entry holders are now the standard, although their design has changed quite radically
- Top-entry holders are becoming less common because they essentially preclude XEDS analysis in the TEM

Specimen Holder and Stages
Side-entry holders

Cup:2.3mm or 3.05mm

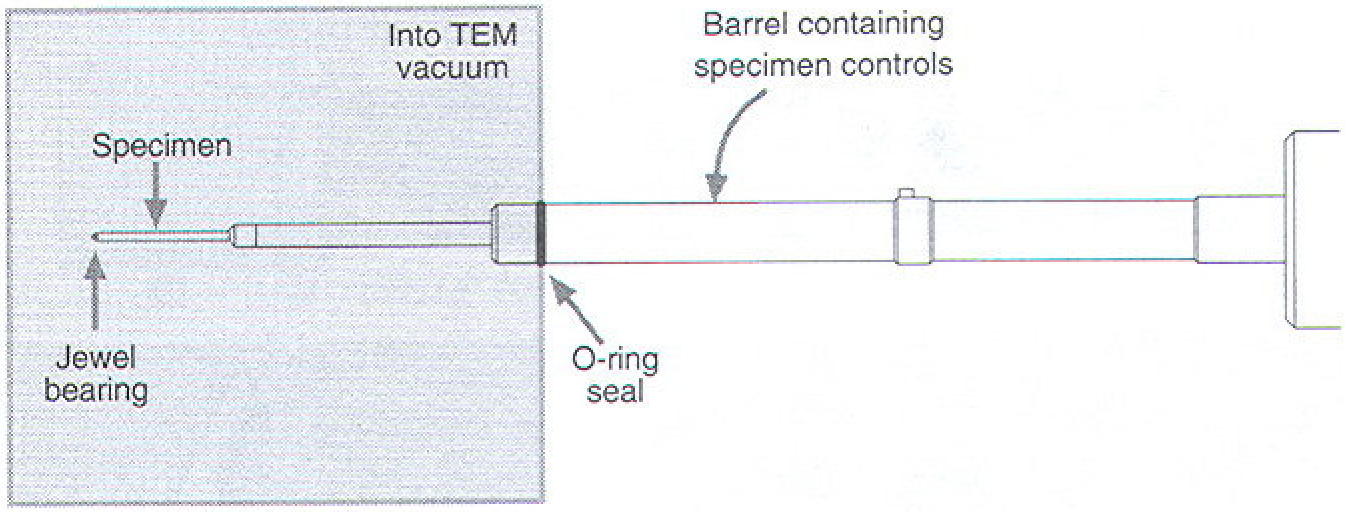
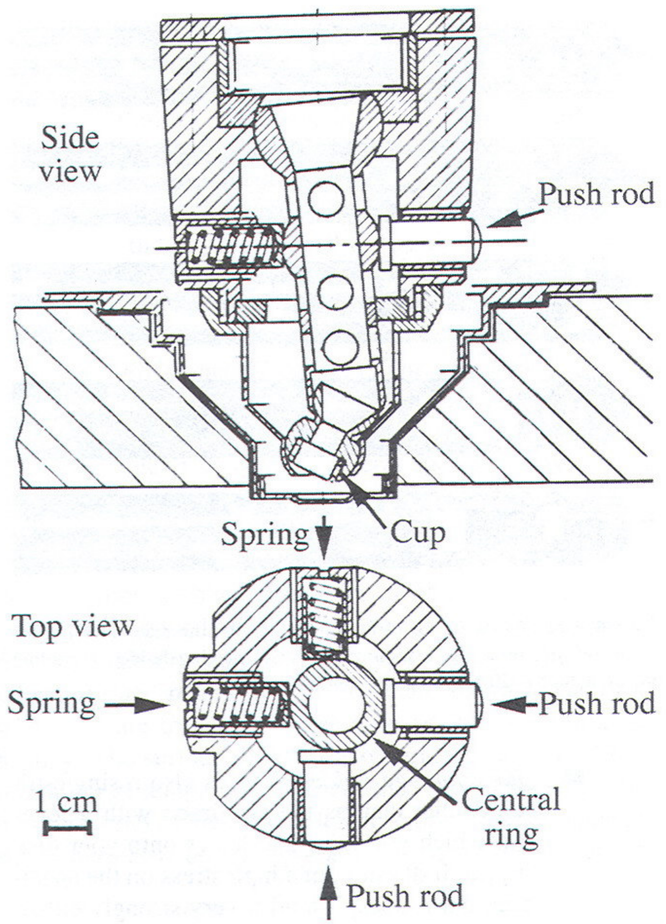


Figure 8.6. Principal parts of a side-entry holder that is held in the goniometer stage. The specimen is clamped into the cup at the end of the rod. A small jewel at the end of the rod (usually sapphire) fits into another jewel bearing in the stage to provide a stable base for manipulating the specimen. The O-ring seals the end of the holder inside the vacuum. Manipulating the specmen is accomplished from outside the column via controls within the rod.

Specimen Holder and Stages
Top-entry holders



Different Types of Holders

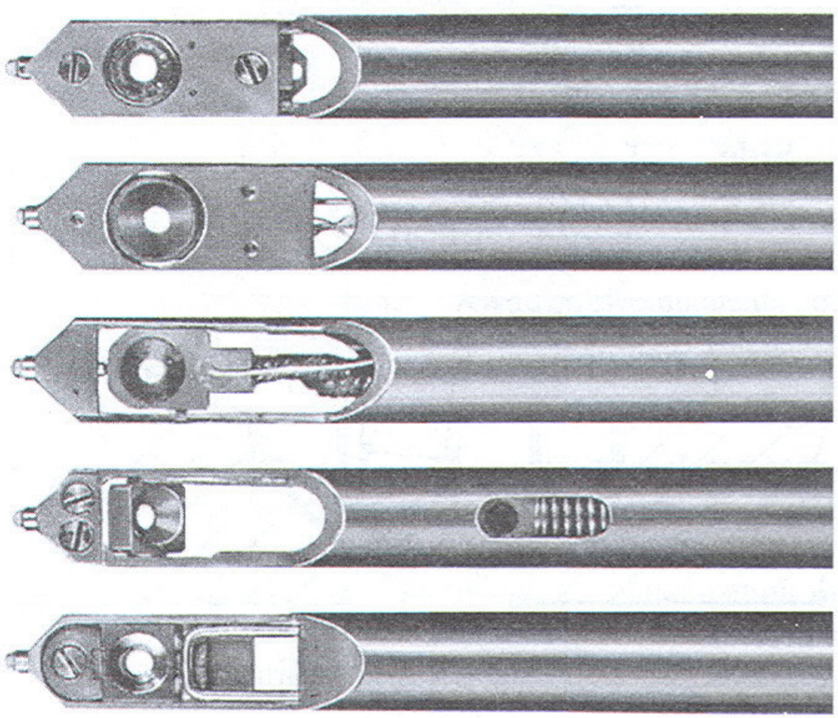


Figure 8.8. Examples of different designs for the side-entry holder. From the top, they are: a rotation holder, a heating holder, a cooling holder, a double-tilt holder, and a single-tilt holder.

- Single-tilt holder
- Quick change holder
- Multiple-specimen holder
- Bulk specimen holder
- Double-tilt holder
- Tilt-rotate holder
- Low-background holder
- Heating holder
- Cooling holder
- Cryo-transfer holder
- Straining holder
- EBIC and CL holder

Different Types of Holders

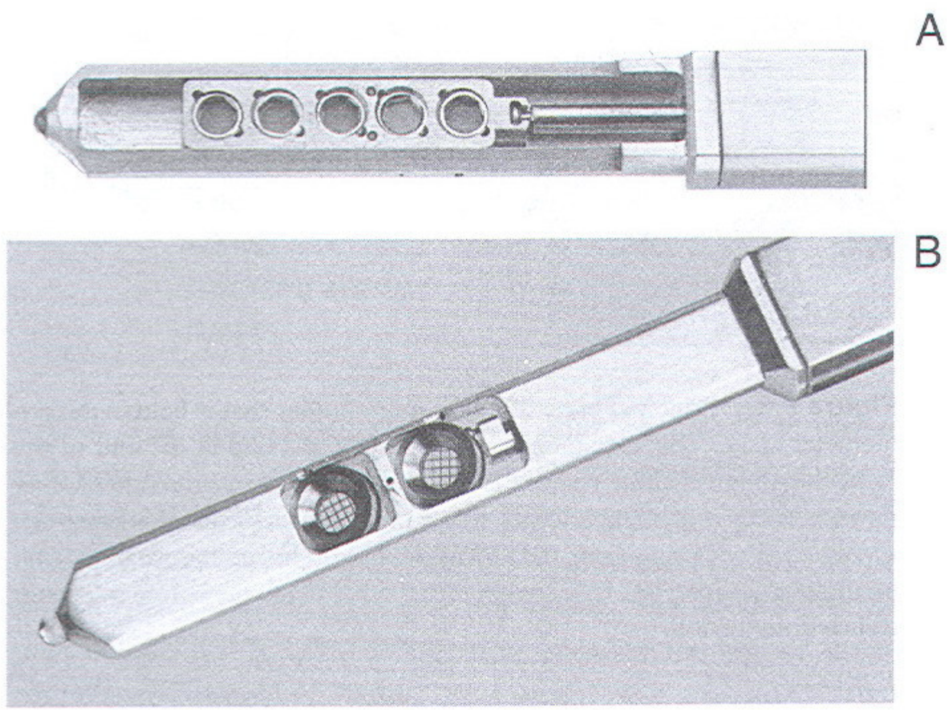


Figure 8.9. Multiple-specimen holders. (A) Five-specimen single-tilt and (B) two-specimen double-tilt.

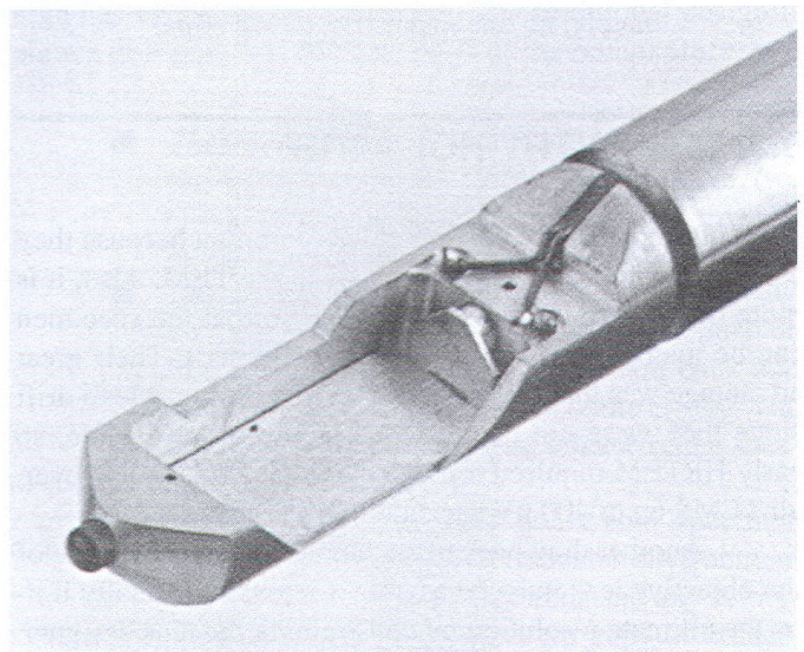


Figure 8.10. A bulk holder for large specimens.

Don’t always think that you are limited to 3-mm specimens

Different Types of Holders

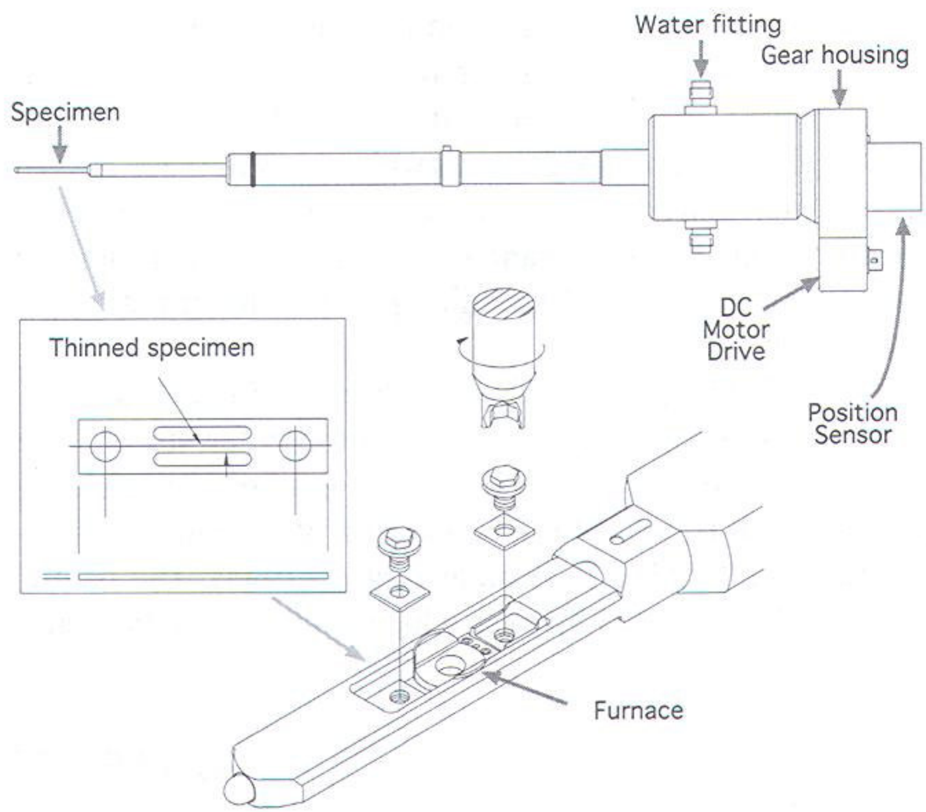


Figure 8.11. A side-entry combined straining and heating holder. The specimen looks like a miniature tensile specimen (inset) and is clamped at either end by hex screws. There is a screw-thread arrangement for pulling the specimen contained within the rod. The furnace surrounds the central thin portion of the specimen.

Different Types of Holders

- Heating and straining holders, in particular, can produce effects in thin foils that are totally uncharacteristic of your bulk specimen.
- Often, surface reactions will dominate internal reactions when you are trying to induce a phase transformation by heating. The surface may also stop grain boundaries from migrating at temperatures where they would do so in the bulk material.
- Defect motion under applied stress may also be strongly affected since the 3D stress field will be very different in bulk specimens compared to thin foils.

Different Types of Holders

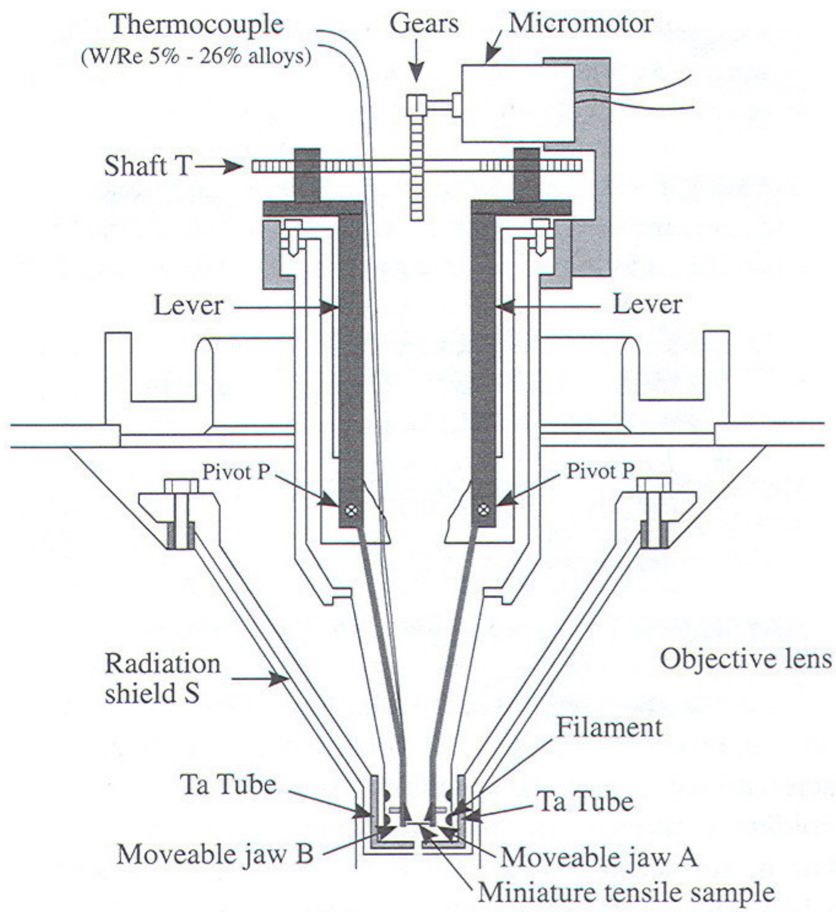


Figure 8.12. A top-entry, heating-straining holder which can be used at temperatures up to 2300 K in a 3-MV HVEM.

Different Types of Holders

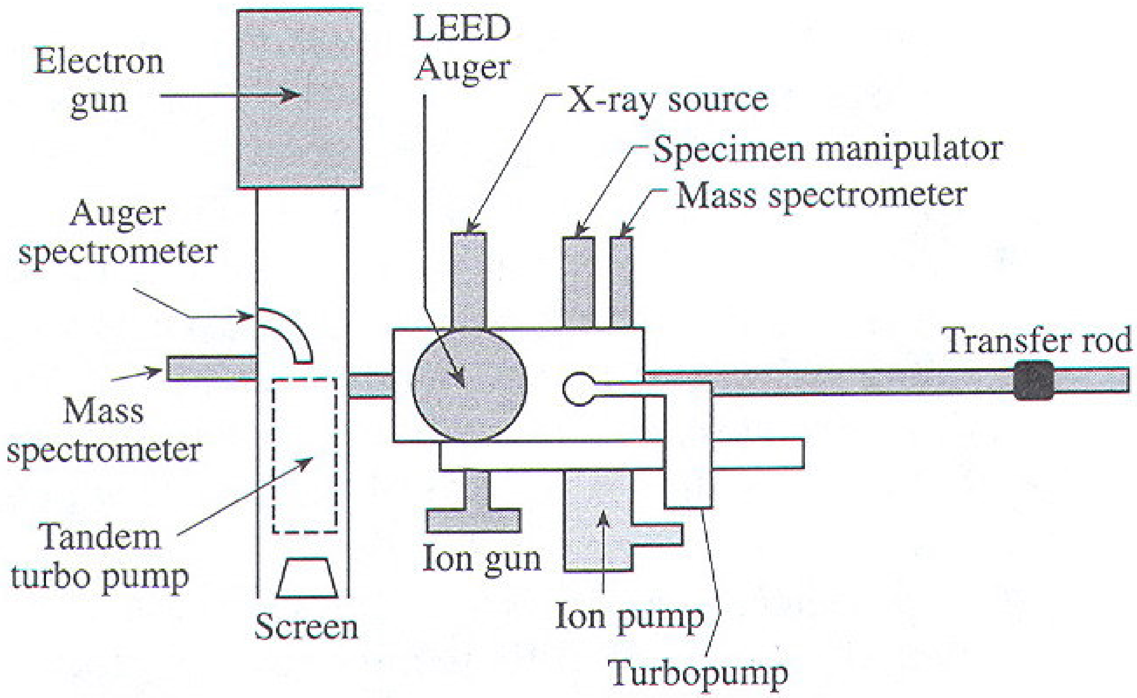


Figure 8.13. Schematic diagram showing the Hitachi H9000 UHV TEM. This instrument is equipped with a prechamber with LEED, Auger, and an ion gun which can be used to clean the specimen, allowing UHV surface analysis to be carried out on the TEM specimen. The holder has to transfer the specimen through a prepump chamber where it is ion-cleaned before going into the column.