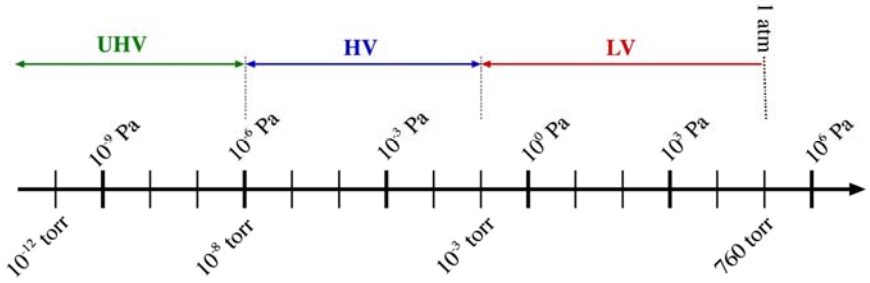
Instrumentation for Characterization of Nanomaterials (v12)

8. Vacuum Systems and TEM Holders

Vacuum terminology

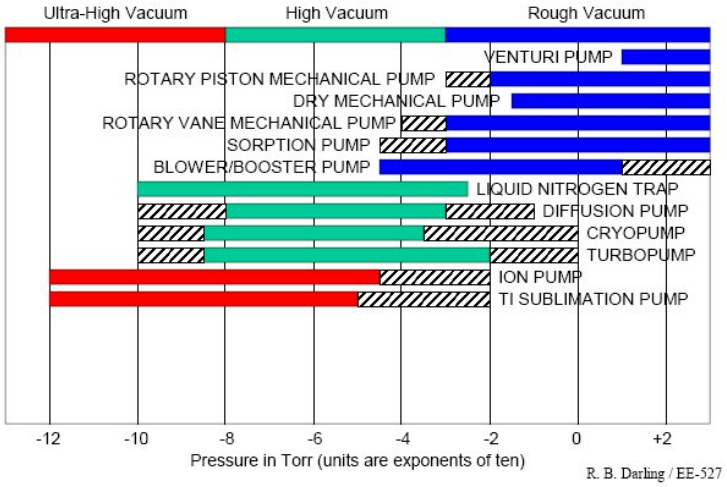
Many experimental techniques require low-pressure, vacuum conditions, instead of the high-pressure, oxygen-rich environment of the earth's atmosphere. Atmospheric pressure under standard conditions is assigned the value 1 atm. But there are several other options for expressing units of pressure - maybe too many. The metric unit of pressure is Pa. Atmospheric pressure is about 101 KPa. This is close to a nice, round number, so the unit bars was defined, with 1 atm exactly equal to 100 Kbar. For centuries, barometers measured pressure using the elevation by air pressure into an evacuated column of a liquid of precisely known mass density. For mercury (Hg), this is 760 mm at STP, which is used to define the unit of torr, where $1\ \text{torr} = 1\ \text{mm} \cdot \text{Hg}$.

The quality of a vacuum is inversely related to the pressure. So low vacuum means higher pressure than high vacuum. Low (or rough) vacuum (LV) usually means anything from just below 1 atm down to around 0.1 Pa. High vacuum (HV) usually spans from 0.1 Pa to about 10^{-6} Pa. Ultra-high vacuum (UHV) is anything below about 10^{-6} Pa. Vacuum pressures below about 10^{-10} Pa are extremely rare on earth; they may only exist in remote regions of intergalactic outer space.



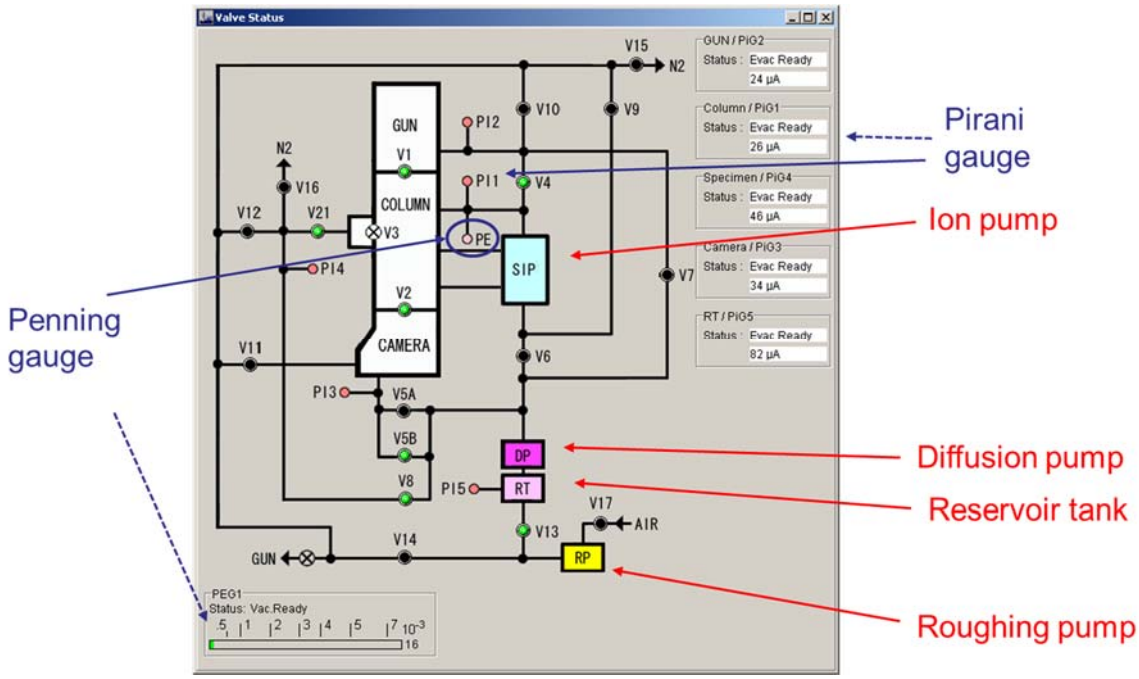
Ranges for different pumps

We will discuss a few common types of vacuum pumps. They each have their advantages and disadvantages, relating to qualities such as pumping capacity, ultimate vacuum achievable, maintenance requirements, cleanliness, tolerance to contaminants and hostile materials, volume and vibration levels, size, and cost. A comparison of some types of pumps is shown below (from R. B. Darling.)



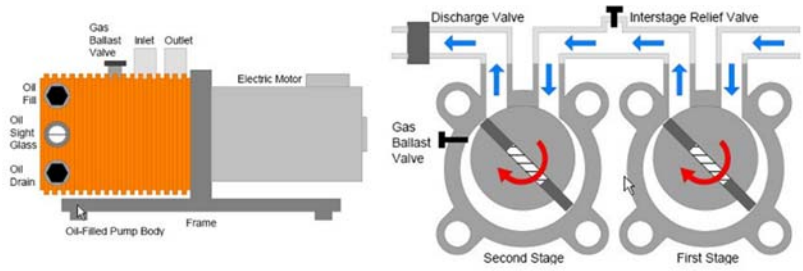
Multiple pumps in a system

In all but the simplest vacuum systems, it is necessary to have multiple pumps acting on different aspects of the instrument, or working in series to achieve an ultimate vacuum within a critical volume. For example, a TEM may use a mechanical roughing pump, an oil diffusion pump, and a sputtering ion pump to provide required vacuum levels within various chambers and allow sample exchange into the microscope column. Along with these are various vacuum gauges, particularly Pirani gauges and Penning gauges, which monitor the pressure, as needed. It is often the case that a roughing pump backs a diffusion pump, which backs an ion pump, which is the only pump directly pumping on the microscope column during operation. A reservoir tank is sometimes used as a buffer to maintain a vacuum at the the outlet of high vacuum pump, increasing its pumping efficiency and capacity.



Rotary, mechanical, roughing pump

The most common and familiar type of pump is the “roughing” pump, which is suitable for low vacuum. The operation is fairly simple to understand and it has appeal to the mechanically inclined. Within the pump is an electric motor, which drives one or two sets of rotary vanes that are essentially immersed in oil. The rotor axis is off center within the pumping stage. The vanes are connected to the axis by springs that keep them pressed flush against the interior seals, so that they slide inwards and outwards from the rotor axis as the shaft rotates. This causes the rotation of the vanes to compress gas taken from the inlet, and then allow the gas to expand into the exhaust on the outlet. The oil serves to cool and lubricate, while also sealing the gap between the end of a vane and its enclosing cylinder. The oil has to be kept full and changed periodically, or the pump may seize. he ballast is a one-way (check) valve that may be opened to inject air or other gas in the low-pressure (isolation) portion of the pumping cycle. Materials that have entered the inlet as vapor can become condensed in the oil during the compression cycle of the pump. The ballast allows them to return to the vapor stage and be expelled through the outlet.

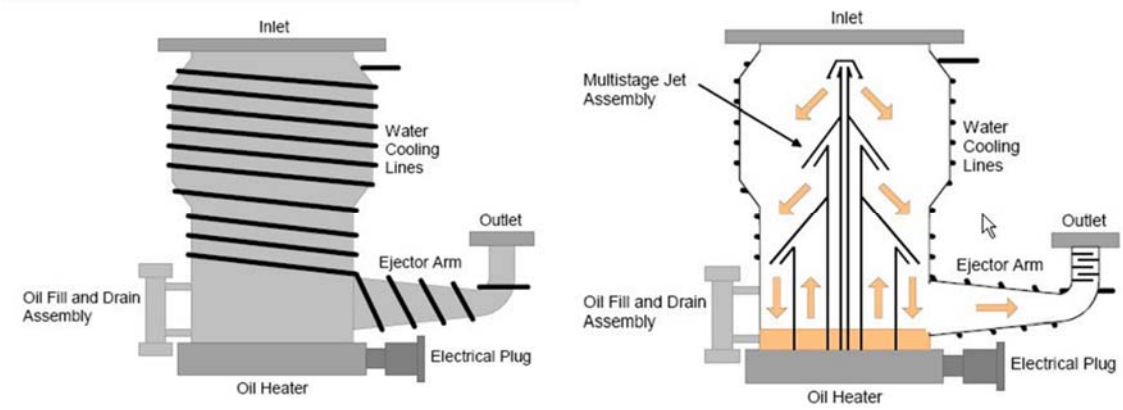


A problem with roughing pumps is backstreaming of the hot oil vapor back into the vacuum system, especially when the pump has been recently run and then shut off. For this reason, the inlet is usually vented after use, or a valve on the inlet line is closed. Another approach is to continuously flow an inert gas through the vacuum system to ensure the chamber pressure is always greater than the pump inlet pressure. Dry roughing pumps are becoming better all the time, but are still expensive.

Oil diffusion pump (ODP)

Oil diffusion pumps are high-vacuum pumps that operate on a surprisingly simple mechanism. The pump is usually suspended from the chamber it is pumping, with the inlet at the top. A central tube with an attached set of downward-pointing "jets" rises vertically within the main cylinder of the pump, and an outlet port extends to the side, near the base. A heating coil, similar to an oven burner is mounted to the bottom.

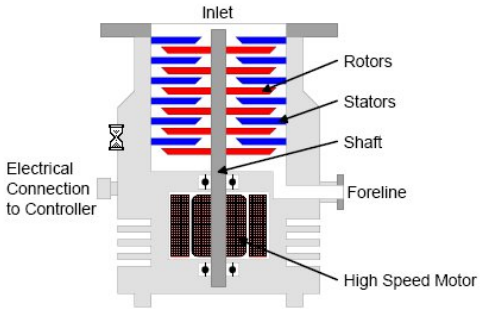
The pump contains a relatively small amount (~ 100 ml) of a special, high vapor pressure oil. The oil is heated, which pushes oil vapor up the central tube and out of the jets towards the pump walls. There, the oil vapor encounters any gas molecules that are to be pumped out of the chamber. The walls must be water cooled, so when the vapor contacts the walls, it condenses, then gravity draws the oil, along with any captured gas molecules, back down to the reservoir at the base of the pump, where the are likely to be expelled out the exhaust. The net effect is an overall flux of gases from the inlet to the outlet.



To actually do any pumping, the ODP has to be backed by a roughing pump. ODPs have no moving parts, so they are desirable when vibration cannot be tolerated. On the other hand, the oil vapor can escape into the vacuum chamber, leaving an oily residue. So, ODPs are often separated from the chamber by a liquid-nitrogen-cooled shroud, where escaping vapor is likely to condense. Nonetheless, ODPs may not be desirable in applications where a very clean vacuum is required.

Turbomolecular pump (TMP)

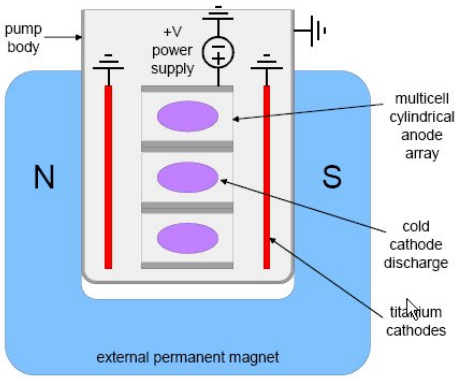
A turbomolecular pump uses many rapidly spinning blades, similar like a turbine engine, to propel gas from the inlet to the outlet. The blades, which are mounted on an axial shaft, comprise the rotors of an electric motor, which are drawn to or repelled from stators affixed to the walls. The motor requires a fairly complicated drive that ramps the input as the pump speed is increased or decreased. There is a great deal of motion involved, so some systems use magnetic levitation to isolate the pump from the system it is evacuating.



No oil is involved, so the result is a very clean vacuum. But TMPs are somewhat fragile and must be ramped up and down very carefully. Sudden venting will essentially crash a large amount of air onto the blades, which are necessarily light and therefore fragile, and can damage their shape, or destroy the pump bearings. The TMP is a high-vacuum pump, so it is almost always backed by a roughing pump.

Ion getter pump

Ion pumps are sometimes called ion getter pumps. The use a magnetic field, usually from a permanent magnet, and high-voltage electrodes to achieve ultra-high vacuum. They are both oil-free and motion-free. Some systems use many small ion pumps, while others use one large ion pump.



The high voltage separates a cylindrical anode from titanium cathodes. Gas molecules entering the electric field generated by the high voltage are ionized, which causes them to accelerate, colliding with other gas molecules and ionizing them. The magnetic field increases the path length by causing the ions to deflect into cyclotron orbits, generating even more plasma. Ultimately, ions are embedded into the Ti cathodes permanently. The pump should only be turned on in high vacuum to extend the life of the pump. The electrical current generated naturally allows the pump to serve as a vacuum gauge, while it pumps.

Vacuum gauges

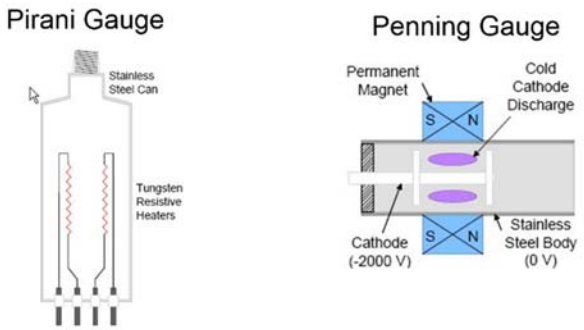
Here we will consider two types of vacuum gauges:

1) Pirani gauge: This contains one or more resistive filaments in a cylinder. A voltage is applied to the filament leads, generating a current. In steady-state, the heat generated by the resistive losses is transferred to gas in the cylinder by conduction. As the gas pressure drops, heat cannot be conducted away as quickly, so the filament will get hotter. The resistance of a metal wire increases with temperature, so the current will drop. By relating the measured current, the pressure in the cylinder can be calculated.

This method works through the low-vacuum range, down to pressures of around 10^{-4} Torr.

2) Penning gauge: This type of gauge works in high vacuum, down to around 10^{-8} Torr. They are often called cold-cathode gauges. As in an ion pump, the cathode can be a metal pin, sometimes with plates, extending into a cylinder. The cathode is maintained at around -2 KV, and the outer body (the anode) is at ground. The cylinder is mounted inside a permanent magnet. As explained for the ion pump, the high voltage ionizes any gas atoms, and the magnetic field promotes avalanches of ions. Again, we measure the current to compute the pressure.

Sometimes, a penning gauge contains a small radioactive source, such as americium. If you first turn on the gauge with the chamber already at very high vacuum, it may be difficult to form a plasma, without creating a few ions first. Without the source, the gauge may indicate a perfect vacuum, at least for a few minutes. The source decay products ionize some gas atoms, which gives the gauge enough boost to get started reading the pressure.



TEM specimen holders

This seems like an OK time to take about some of our TEM specimen holders. Almost all holders for commercial TEMs are configured for mounting 3.0-mm diameter, circular samples, usually grids. The mechanisms to hold these, while maintaining good access and maneuverability of the sample, differ greatly, though.

JEOL uses two o-rings. They have to be kept clean and slightly moist. We usually use a fluorine-based grease, which has a low vapor pressure and won't contaminate samples. In the TEM column, the end holding the sample freely extends inside the objective lens pole piece.

The pole piece is the main limitation on the tilt range. The main tilt axis (parallel to the holder shaft), is erroneously called the "x" -axis (I say this because x translation is perpendicular to this axis); a better name is the "alpha "axis. All TEM holders have some range of alpha tilt. To increase the tilt range, the part of the holder extending into the pole piece has to be narrower, which often limits the possibility of adding other controls.

The secondary tilt axis (called "y", should be "beta") is only available on some holders, because it requires the holder to have its own motor and drive mechanism. A cable extending from the holder has to be plugged into a port on the TEM for communication. Some models use rotation about the axis normal to

the specimen, which has basically unlimited range, but is not very convenient for crystallographic work, because the region of interest generally moves rapidly away from the eucentric point (the point we have agreed is the center for alignment of the TEM) every time the tilt is changed.

1) Low-background, double-tilt holder: This is specially designed for EDX analysis. The region immediately around the sample is made of Be, which is nearly transparent to x-rays, and does not contribute any noticeable emission peak, either. The samples is sandwiched below a Be clip, which is held down by two tiny brass brackets with tiny screws.

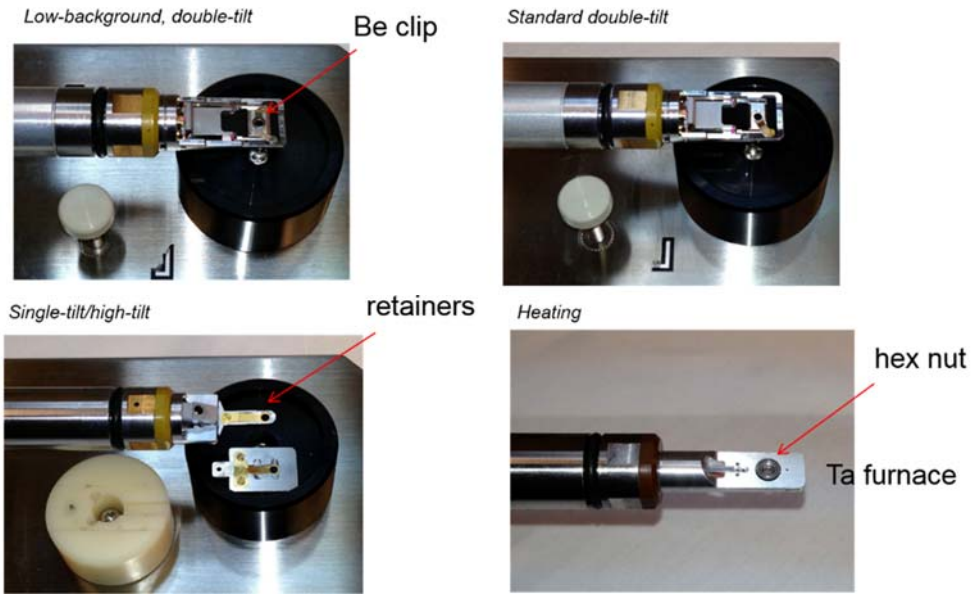
2) Standard double-tilt holder: This has the easiest loading mechanism of the JEOL holders, because it uses only one tiny screw. Otherwise it is very similar to the low-background holder.

3) Single-tilt/high-tilt holder: Most people use this holder. The standard retainer has a brass clamp that has to be swiveled out of the way by loosening the two screws (not all the way!), with one screw as the axis. The sample sits at the "T" intersection near the end. Then the clamp is swiveled back into place and tightened down.

To load your sample, the retainer actually has to be released from the holder using the tool stored on the back of the holder stage as a lever. Just let the retainer drop off, then set it on the white stand. This turns out to be a nice feature, because we can swap out the retainer with a different one, maybe designed for some particular nano-manipulation experiment. We have a high-tilt retainer for tomography that is nearly as good as the standard retainer for just about any other application, too. And it doesn't even have to be taken off the holder to change samples. But it is a bit less stable than the standard retainer, if that is a concern.

4) Heating holder: The Gatan heating holder has a Ta furnace to hold the sample and Ta parts all around the sample, including a hex nut that holds the sample in. Most people like the hex nut better than some alternatives, because you don't have to deal with tiny screws. The hex tool is in a slot on the holder stage.

In the vacuum of the TEM, the holder can heat up to 1100 °C. You should only load samples that can withstand the high temperatures of your experiment. (A Cu grid may not be the best choice.) Put a Ta washer above and below your sample, to make sure it doesn't melt onto the holder itself. The holder should be connected to the SmartStart controller. If you plan to heat above 500 °C, you should also connect the inlet/outlet distilled water lines from the reservoir. (Be sure the reservoir is around half full.) The controller turns on the water when needed to stabilize the temperature of the furnace.


Low-background, double-tilt
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retainers
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hex nut
Ta furnace