Introduction to Vacuum Technology

General

Many surface scientists work with their samples in a vacuum system. The reasons for this are several fold: first, many samples react with the gases in ordinary room air which means they must be kept in a clean environment; second, the experimental probes used to measure sample properties may depend on electron or other beams that simply could not exist outside a vacuum. This guide will introduce you to basic terminology and technology for producing and maintaining vacuum conditions.

Ambient pressure is defined as the current atmospheric pressure which is usually right around 1.0 atm = 1.01×10^5 Pa = 1.01 bar = 760 Torr. The composition of air is approximately 78% nitrogen and 21% oxygen with the remainder consisting of a mix of carbon dioxide, water vapor, etc. At atmospheric pressure there are roughly 10^{19} of these molecules per cubic centimeter which indeed makes maintaining a clean surface a challenge.

According to the American Vacuum Society scientists working with vacuum equipment distinguish between seven different pressure ranges, but for the purposes of this discussion we will limit ourselves to three. Rough vacuum covers a range from 10^2 to 10^{-2} Torr and can be achieved using inexpensive rotary vane pumps. In these pumps oil provides the seal between the vacuum and atmosphere sides of the pump. Because of their construction it is relatively easy for oil vapor to enter the vacuum system leading to contamination of samples with hydrocarbons which is of course undesirable. Even if hydrocarbon contamination did not exist, the density of gas molecules is only reduced to about 10^{15} per cubic centimeter which is insufficient to keep a sample clean or provide a sufficiently long mean free path for particle based probes (*e.g.*, the mean free path of an electron beam at 10^{-2} Torr is only YYY).

High vacuum lies between about 10^{-2} and 10^{-8} Torr which is made possible by two different classes of pumps. Diffusion pumps using hot oil or mercury have the advantage of no moving parts although they must be backed by a rotary vane pump. They can be used in situations where contamination is not critical such as certain molecular beam experiments. Where contamination (or exposure to toxic mercury, for that matter) is an issue, turbomolecular pumps are used. These so-called turbo pumps are mechanical devices that use a stack of rotating vanes with blades pitched at varying angles to knock residual gas molecules preferentially in one direction. Oil based lubricants have been eliminated in recent models which reduces the chances of contaminating the vacuum considerably although care must be taken when using a rotary vane pump to back the turbo pump – more on that later. At the low pressure end of the range the number of gas molecules has been reduced to 10^9 per cubic centimeter which is a significant improvement over rough vacuum conditions.

To give you an idea of what this means, consider that using the kinetic theory of gases and some basic assumptions like a unity sticking coefficient (if a molecule hits a reactive surface it will stick to it and not reflect back into the vacuum) you can show that at 10^{-6} Torr it takes 1 second to cover a surface with one atomic layer (a monolayer) of contaminating gas molecules. So at 10^{-8} Torr it would take roughly 100 seconds to contaminate the surface which is two orders of magnitude longer, but still not long enough to perform most measurements.

Ultrahigh vacuum represents state of the art performance in experimental surface science today. Pressures from 10^{-9} down to 10^{-11} Torr are routinely achieved using mass produced standard parts and ion pump systems. Ion pumps, like diffusion pumps, have no moving parts, but unlike diffusion pumps contain no messy or toxic working substances. Rather they use high voltages to induce what is called cold cathode emission – essentially, electrons are ripped from a negative electrode by extremely high electric fields. These electrons accelerate toward the positive electrode and ionize residual gas atoms which in turn accelerate under the influence of

the electric field and become embedded in chemically reactive plates. This pumping action reduces the number density of molecules down to approximately 10^6 per cubic centimeter and theoretically gives researchers tens of thousands of seconds to perform their measurements before the surface gets dirty.

Our System

The schematic diagram in Figure 1 shows the pumping elements used to maintain vacuum in the experimental chamber in our lab. To pump down from atmospheric pressure make sure all ports have been sealed and all valves with the exceptions of the one between the turbo pump and the main chamber and the one between the rotary pump and the turbo pump are in their closed positions. Plug the rotary vane pump into a wall outlet to start pumping. The thermocouple gauge on the top of the equipment rack indicates the pressure behind the turbo pump (the so-called backing pressure).

A rotary vane pump works by sweeping a large volume of gas into a small volume thereby increasing its pressure. If the increase in pressure is large enough a discharge port opens to atmosphere and the gas is expelled from the pump. A simplified diagram of such a pump is shown below in

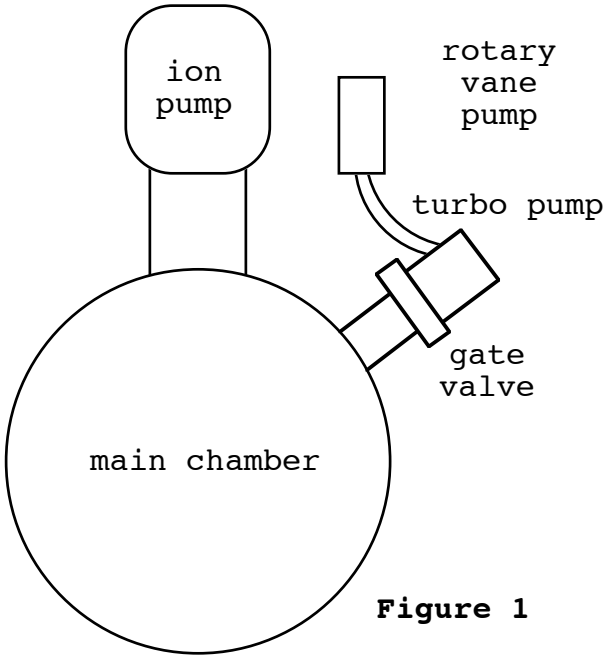

Figure 1

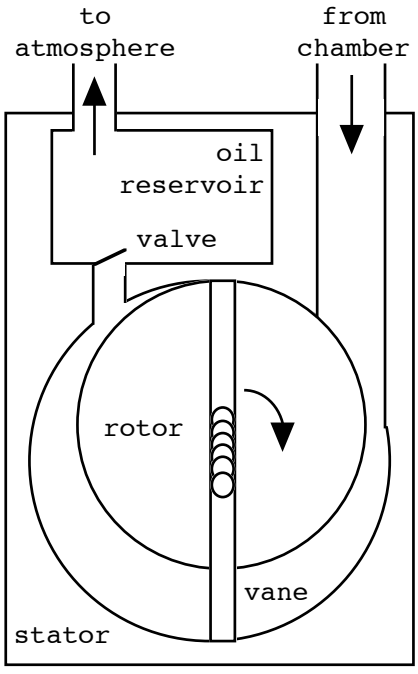

Figure 2

Figure 2. A rotor that is off center with respect to an internal chamber called the stator has vanes attached that rotate with the rotor about its axis. The vanes can move in and out perpendicular to the axis of rotation so they can maintain contact with the stator and provide a seal. Note that as the rotor spins in the sense shown the volume of the gas trapped between the vane and the valve gets smaller so the pressure of the gas rises. Some rotary pumps have three to five vanes although only one is shown here for clarity.

The rotary pump should be run until the thermocouple gauge registers approximately 300 micron pressure. Turn on the cooling water to the turbo pump and then press the start button on the turbo pump controller. The controller brings the pump up to its fully operational 56,000 rotations per minute via a series of intermediate speeds. As the turbo ramps up you will notice the backing pressure rise as much of the gas

remaining in the chamber is forced out. This is normal and should be followed in short order by a drop to the 30 micron range by the time the turbo reaches full speed. At this point it is safe to turn on the ion gauge to monitor the pressure in the chamber. The pressure should drop steadily through the 10^{-4} and 10^{-5} Torr ranges and into the 10^{-6} Torr range. (A quick way of referring to different pressure ranges is to refer to the order of magnitude only. So, for example, if the chamber pressure is 4.5×10^{-5} Torr you would say "4.5 in the fives" or simply that the pressure is "in the fives".)

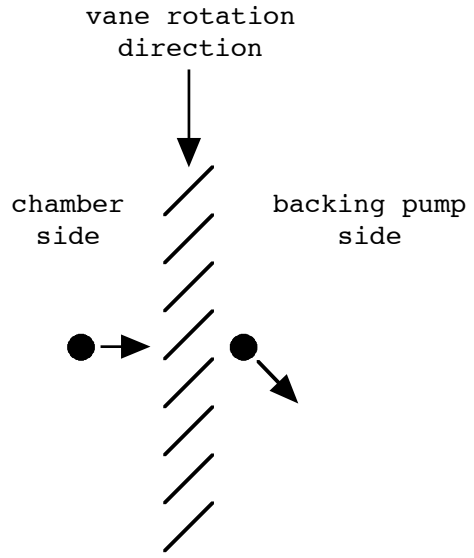

Figure 3

The turbo pump works by giving gas molecules a push in a particular direction by the action of rotating vanes. The sketch in Figure 3 shows a small section of one stage in a turbo pump. Imagine you are looking at a disk edge-on that is rotating about a horizontal axis somewhere behind the page. The diagonal lines represent the vanes which would be moving essentially vertically on the page. A gas molecule is shown impinging from the chamber side on the left, and as it enters the vanes it is given a velocity component down-and-to-the-right. In other words, the molecule is pushed toward the backing pump. On the other hand, if a molecule were incident from the right it would be pushed back in the direction from which it came when it encountered the vanes. In this way, a turbo pump maintains a pressure difference across itself by creating a preferred gas flow direction. In a typical turbo pump there are several rotating disks separated by stators with the vane angle decreasing at each stage.

Depending on how long the chamber was open and what sort of work was done, the chamber pressure may drop into the sevens relatively quickly. At this point it is safe to turn on the ion pump. Set the ion pump controller to the 3 kV supply range and make sure the "run" mode is selected. This mode monitors the total pump power over a period of seconds to ensure that the safe maximum current has not been exceeded – it is possible for the pump to develop a short between its anodes and cathodes either through the creation of a plasma or a piece of material making direct contact. (There is a "start" mode available on most controllers that bypasses the detection circuit, but it is not recommended that you use it until you have more experience with vacuum techniques. In any case, the pump should never be left unattended unless it is in the safe mode.)

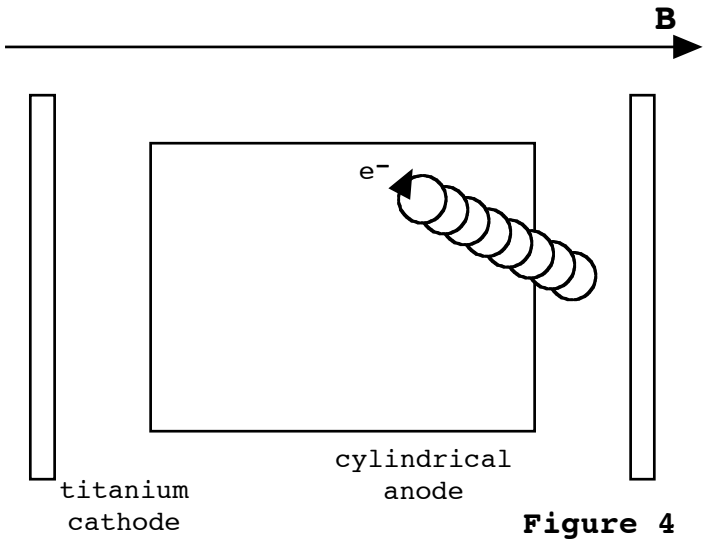

Figure 4

The basic construction of an ion pump cell, shown in Figure 4, consists of titanium plates held in close proximity to but electrical isolation from stainless steel cylinders with an external magnetic field

parallel to the cylinder axis supplied by permanent magnets mounted outside the pump body. There are a variety of biasing options that depend on the kind of pump, but the common principle is that the anode is held at a high voltage relative to cathode, usually from 3 to 7 kV. Because of the electrode's close proximity the electric fields within the pump cells are high enough to rip electrons out of the cathode (this is called cold cathode emission to distinguish it from electron emission from hot filaments such as in the RHEED gun). As the electrons accelerate toward the anode they travel in a helix due to the external magnetic field which substantially increases their path length through the vacuum. This enhances the probability that the electrons will collide with a neutral gas molecule and ionize it because of its large kinetic energy. These positively charged ions will then accelerate toward the titanium cathodes and impact with sufficient momentum to sputter titanium into the vacuum which coats all surfaces in the vicinity. Pumping occurs via several mechanisms. First, titanium will form stable compounds with many gas molecules through chemical reactions. Second, the ions incident on the cathodes can bury themselves quite deeply in the metal. Third, neutral molecules and nonreactive noble gases can be buried by the sputtered titanium as it coats surfaces.

When first started an ion pump will typically "burp" gases into the vacuum because the pump surfaces need to be coated with a fresh supply of titanium. For this reason it is a good idea to keep the turbo pump running in parallel with the ion pump when it first starts. As the pressure drops into the low sevens or high eights you can try closing the gate valve that separates the turbo pump from the main chamber. Monitor the chamber pressure to ensure that the ion pump can hold the vacuum on its own – the pressure may rise a bit when the valve is closed but it should plateau and begin to decrease before you decide to shut off the turbo pump.

To turn off the turbo pump use the following procedure: 1) Make sure the gate valve is closed. 2) Press the start/stop button on the turbo pump controller. 3) Close the inline valve between the rotary pump and the turbo. 4) Unplug the rotary pump and open the flange between the pump and the valve to vent it – this is an important step because it prevents oil from the pump from contaminating the line. 5) Allow the turbo to spin down – this takes at least 15 minutes and in that time the cooling water should be continue to run. 6) Turn off the cooling water when the turbo has stopped completely.

When the chamber pressure has reached the low- to mid-eights the controller can be switched to the 5 kV supply setting. Again, there may be a "burp" as shallowly buried gases are blown out of the anodes by the more energetic ions. Make sure the controller can handle this initial extra load without shutting down the pump. Over the period of several days the pressure in the chamber will reach the low nines without any further work on your part. Lower pressures are not necessary for the experiments you will be performing in lab – the transmission diffraction patterns from metal foils and the surface diffraction and microscopy data are not adversely affected by a layer of contamination at the surface.

To achieve lower pressures requires a two day process called baking. The reason the pressure in the chamber is relatively high (remember the ultimate pressure that can be achieved with this system is the 10^{-11} Torr range) is because an effectively infinite supply of junk is adhered to the surfaces within the vacuum chamber. Thermal agitation causes these molecules to desorb from the surfaces and into the vacuum. To speed the process along you can wrap the chamber in electric heating tapes and cover it with aluminum foil to distribute the thermal energy evenly. By heating the chamber to 150 °C or so for about a day the material adhered to the interior surfaces can be blown off and pumped away. With sufficiently careful vacuum practices (wearing gloves when handling samples or working on vacuum apparatus, using materials approved for vacuum systems) and attention to proper bolt tightening on flanges, the chamber will reach the low tens or high elevens when it cools down to room temperature.

References

1. G.L.Weissler & R.W. Carlson editors, *Methods of Experimental Physics – Volume 14: Vacuum Physics and Technology,* Academic Press (1979).

Acknowledgments

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