https://doi.org/10.1017/S155192950005690X Published online by Cambridge University Press

Notes On Vacuum Techniques For Microscopists

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This article was originally written to respond to a discussion on the Microscopy Listserver found on the Internet concerning backfilling an electron microscope with nitrogen versus laboratory air. It was expanded to include general vacuum comments, suggestions, and techniques that could be of possible value to the working microscopist.

NITROGEN BACKFILLING

It was suggested by a few that since the film is *outgassing* it is OK to backfill a TEM with lab air which has a high moisture content because the film in the camera chamber has a significant outgassing rate. My general comment is that you want to keep as much water and oxygen out of a vacuum system as possible. There is no need to add more to what may already be there.

In a vacuum system without any leaks or severe outgassing problems and with properly cleaned surfaces, the major factor which limits the ultimate pressure and pumpdown time is the water adsorbed on the exposed surfaces of the system. Depending on the type of surface, water molecules can either adsorb or chemisorb onto the surface. A chemisorbed species is characterized by having an exchange of electrons with the atoms on the surface and will have a higher energy associated with the desorption of the molecule from the surface. In an analytical microscope, the system should be baked above 200°C to get the water to desorb from the surfaces. Here it is important that all of the internal surfaces reach a high enough temperature and remain there sufficiently long so that as much of the water on the surface is desorbed as possible. This is a thermally activated process, so it takes more time at a lower temperature. For UHV systems, it is generally held that 200°C is about the minimally accepted temperature for an adequate bakeout. Once the molecule is desorbed and is in the volume of the vacuum chamber, it is easily pumped by whatever high vacuum pump the system has. The system will not go down below the vapor pressure associated with the adsorbed species until it is gone. For example, the oils and moisture contained in a thumbprint will limit the vacuum pressure to about the 10^{-8} Torr range. The pressure of the system will not go below that until these oils are gone, *i.e.*, pump on it for a very long time, or bakeout the system. That is why it is very important that powder-free gloves be worn when handling samples and items that go into the vacuum system.

To improve vacuum performance after a system is brought up to atmospheric pressure, as dry a nitrogen that can be found should be used for backfilling. Unless ultrapure nitrogen gas is used, regular cylinders of nitrogen can have quite a high moisture content. Even ultrapure gas doesn't guarantee that it is free from moisture. It has been suggested by contributors on the Microscopy Listserver that the gas be dried. This works well by passing the gas through a coil of tubing submerged in liquid nitrogen (LN2). Several loops should be used with a low gas flow rate to insure drying the nitrogen. However, if you have access to liquid nitrogen (LN2), the boiloff is the purist, driest nitrogen that you can get. I like to backfill a system by using an open container of LN2 and inserting a tube into the container and then putting the other end onto the up-to-air valve. When you insert the open tube into the

LN2, the tube is purged of gas because it is warm and the LN2 boils off fast. If you use tygon tubing, you can see liquid being pushed out the other end. It is best to use a longer tube so that the tygon is not cooled too close to the system inlet. Use the gas to purge the inlet valve by holding the tube close to it, but not making the connection. Just before the rapid boiling stops, put the open end of the tube onto the inlet connection. The tube and atmosphere side of the valve is purged and the gas in the tube is at atmospheric pressure. Open the system slowly and it will draw nitrogen off and equalize at atmospheric pressure since the LN2 container is open.

SAFETY CONSIDERATIONS WHEN BACKFILLING

If I'm not going to have a large port open, then the above method is my preferred way of backfilling. It does not overpressure the system. However, a slight overpressure is good because it keeps dust and atmospheric air out of the system. (I always cover open ports with clean aluminum toil or lint free cloths when they are open.) DANGER!!! Too high an over pressure can blow out a window port. (It doesn't have to be very high since it depends on the area of the window.) A glass window is designed to be in compression under vacuum. When there is an overpressure the window can be put in tension which could cause it to break and blow out causing harm to a nearby observer. Don't go above 1 or 2 psi when backfilling a chamber with windows in it. More than that is not needed and it is dangerous. Remember, the viewing window on a TEM is very large, very thick, and very expensive. When I use pressurized nitrogen without a demand regulator to backfill a system, I set the pressure so that the gas coming out of the tube just feels slightly cool after I moisten my lips. Another trick that some people use is a TEE on the tube with a balloon attached to it and the pressure is set so that the balloon is slightly inflated, *i.e.*, it stands up but without stretching the surface.

When I want a slight over pressure with the LN2, I use a percolator consisting of an inverted tunnel attached to the filler tube, a small resistor positioned in the funnel and attached to a small battery outside of the LN2. When the current goes through the resistor, it boils off some nitrogen. The more power going to the resistor, the higher is the flow of nitrogen in the tube. I played around a little to find a resistor that gave me the pressure that I wanted and it works well. A word of caution, an open container of LN2 will condense oxygen which can pool at the bottom of the dewar. My resistor is never near the bottom of the dewar, does not have much power going through it, and is not used long enough so that a large amount of oxygen can accumulate in the dewar.

Additional safety caution: If you use a LN2 dewar that can be pressurized by an external gas source to deliver nitrogen gas or liquid, NEVER use lab air to pressurize the dewar because of the liquid oxygen that can build up in the bottom of it.

POOR MAN'S (WOMAN'S) BAKEOUT—
AN ALTERNATIVE TO BAKE OUT

There is a method for cleaning water off of the internal surfaces of vacuum systems without the time or the bother of a prolonged bakeout. It uses two vacuum principles, a hot gas will transfer energy to the internal surfaces, and viscous flow will pull molecules along. Here's how to do it. Heat your dry nitrogen. You can do this with heating tape wrapped around a coiled tube of sufficient length to heat the gas. Then set up a steady state flow by opening the gas inlet valve and balancing the pressure with the

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valve to the vacuum pump to keep the flow in the viscous regime The viscous range can be calculated for the size of tubing you have, but about 100 millitorr will probably satisfy the condition for most systems Let it go for several hours and you will get rid of most of the water in the system from the internal walls. I once got a UHV system that had a 5 inch chevron assembly in the 10^{-10} Torr range without baking the system by using this method.

SPUTTER ION PUMPS - IMPROVED STARTUP PROCEDURE

In the new TEM's and SEM's the higher vacuums in the gun area and sample area are achieved with ion pumps. The ion pump is a capture pump, meaning that the gas that is pumped is captured in the pump and is retained. A characteristic of the pumpdown of a system with an ion pump is the "burping' of some of the gas out of the pump which is seen as a sudden pressure burst on the vacuum gauge. There are several pumping mechanisms for an ion pump, but in general, active gasses are gettered and inert gases are buried. There is a limit to the amount of gas that an ion pump can have in it. The more gas that it does not have to pump and contain, the lower the ultimate pressure and the faster is the pump-down down. For microscopes, the vacuum is typically pumped down with a high throughput pump such as a diffusion pump or turbopump prior to the ion pump being turned on. Before the system is brought up to atmosphere, the ion pump surface has freshly sputtered titanium available for gettering the active gases. When the gas is introduced, the titanium getters as much as it can. Since titanium doesn't bind nitrogen as tightly as it does water and oxygen, it is again very important to backfill with dry nitrogen. HERE COMES THE TRICK. You want to get as much of the adsorbed, buried, and chemisorbed gas out of the ion pump as you can with your high throughput pump before using the ion pump to pull the system down to its ultimate pressure. When you first turn the ion pump on, all the stuff that was gettered by the pump is blown off and then will be either repumped by the ion pump or drawn out by the diffusion pump. To get this gas out of the ion pump, manually flick the pump on then off very quickly and then let the other pump take the gas out of the system by waiting a few moments. Do this several more times until you do not see the big pressure burst in the system. After that, let the ion pump start normally. You will see that the ion pump in your system starts faster and there are fewer "burps," leading to a faster pumpdown time. You may also see a lower vacuum pressure achieved. I've taught this to several microscope service engineers and they are usually impressed with the results.

PRESSURE MEASUREMENT - WHAT'S THE PRESSURE AT THE SAMPLE?

For an analytical microscope, it is desirable to have the lowest vacuum pressure possible to eliminate contamination buildup under the electron beam. Manufacturers have gone to great lengths to improve the vacuum systems in the modern analytical microscope. They have used clean, oil-free vacuum pumps in the critical areas such as the sample and the electron gun areas and they have improved their vacuum design and practices for high vacuum and ultrahigh vacuum pressure ranges. The ultimate pressure on the microscope is a specification of the microscope which is often a consideration in the purchase of the instrument. It is important that you view the pressure of the microscope with a skeptical eye.

Typically, the high vacuum gauge is mounted just in front of

the pump, the place where the vacuum pressure will be the lowest. Sometimes the high vacuum gauge is the high vacuum pump itself. For an ion pump, the log of the ion current is linearly related to the pressure of the system. Some ion pump controllers will have a logarithmic pressure scale on the current meter that is calibrated for the size of the ion pump. However, the sample is located in a region of the microscope (TEM) which is some distance away from the pump and the tubing connecting the pump to the sample region will limit the action of the pump at that point. The property of the tube which limits the pumping is called conductance. Conductance has the same units as pump speed (liters/sec). The effective pump speed at a point in the vacuum system considers the conductance of the pathway to the pump. The formula for the effective pump speed for a pump rated at a speed of S and a tubing with a conductance, C, is given by S(eff) = SC/(S+C).

If the conductance of the tubing is equal to the pump speed, the effective pump speed will be S/2. If the conductance is less than the pump speed, the effective pump speed is said to be conductance limited. This is the case for the analytical TEM. Practically, this means that the pressure at the sample will be higher than the pressure where the gauge is located. There is a very simple equation which relates the pressure to the effective pump speed and is given by Q (Torr-liters/sec) = S(liters/sec) P(Torr), S is the pump speed, P is the pressure, and Q is the throughput. The throughput is the amount of gas that is being handled by the pump. The contributions to the total throughput include outgassing from the walls, leaks, virtual leaks, permeation through seals, etc. At steady state, the throughput at the sample will approximately equal the throughput at the pump, leading to S(pump) P(pump) = S(eff)P(sample).

Since S(eff) is less than S(pump), the pressure at the sample will be higher than the vacuum gauge reading.

In the TEM, there is a further complication when the anticontamination device (ACD) is used. The ACD is a LN2 cooled surface near the sample which condenses gases which might otherwise find their way to the sample and contaminate it when the electron beam hits it. Effectively, this is a mini cryopump around the sample which will reduce the pressure. Because of these considerations, there is no way of precisely knowing what the pressure is around the sample. The best estimate would be arrived at by calculating the conductance of the tubing leading tho the sample area, using this value to calculate the effective pump speed, and then use the measured pressure and the rated pump speed to calculate the pressure. This would then give you an upper limit to the pressure, especially if the ACD is in use. ■