Lesker Tech "...vacuum science is our business."

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How Low Can You Go? Part 4

Others Like a 'Lite'

This is the continuation of *Lesker Tech, Volume 2 Issue 1,* so it needs no introduction.

Light

UV Lamps

Up front I admit this segment is somewhat bo-oh-oh-gus! But I want to introduce a point I'll raise again later. To desorb a gas molecule you must add sufficient energy to the molecule-surface bond to break it. IR adds energy to the bond's rotational and vibrational modes—the spinning, rolling, wiggling, wagging, bending, and stretching modes. (Sounds like my morning calisthenics!) While this energy input helps, what really puts zing into the process is energy added straight to bonding electrons. Get those puppies excited and Bingo! Off pops the molecule. And what causes electronic excitations? Yup, light in the UV part of the spectrum.

Since quartz is transparent some distance into the UV range, the halogen lamps discussed above (in *Lesker Tech, Volume 2, Issue 1*) double as both IR and UV sources. My suspicion is, however, the UV photons produced by a hot filament are both few in number and long in wavelength. That is, they are neither copious nor particularly energetic. Presumably, if you installed a fluorescent (discharge) lamp filtched from your local *Exotic Tropic Tanning Salon* (with its blocked alpha rays and its bunched beta rays or whatever rubbish they promulgate these days), you'd do better. But I have a hard time believing *ETTS* would buy fluorescent lamps with silica envelopes.

Plasmas

Let's all get on the same page, plasmatically speaking. Essentially, a plasma is an overall neutral enchilada of: positively and negatively charged atoms, molecules, and fragments; electrons; radicals; excited atoms; metastable molecules; and regular old neutrals. If those terms don't mean anything to you, think of Murphy's Law; Sod's Law; or "Ha valami elromolhat, az el is romlik." As the last phrase, kindly translated by our Hungarian office, states "If something can go wrong, it will." Well, that's a plasma—a once-normal gas in which everything has gone wrong.

Plasmas are formed in many different ways: DC electrodes, RF electrodes, microwave and RF-power inductive coupling, lightning discharge, solar wind, and probably others that only Nicola Tesla knew about. They can either be tenuous, with only few electrically charged species per cubic centimeter, (as in the auroras—borealis or australis) or dense with charged species up the kazoo (like a tokamak fusion reactor). A medium density plasma, however, is ideal for cleaning vacuum chambers and their contents.

1. Argon Plasmas

A plasma struck by putting several hundred volts RF on a plate electrode in a grounded chamber pressurized with 0.1 to 1 torr argon flowing at 1 to 100 sccm, removes adsorbed water and gives the surfaces a good general etch clean.

But, just like a recent caller, I hear you asking, "How can an argon plasma do much good? Argon's an inert gas, right?" Sure is, but remove an electron and argon becomes chemically equivalent to chlorine—that voracious reaction PacMan. (And for those who know about thin film technology, I can't think of a single material, insulating or metallic, that isn't sputtered by Ar^+. Can you?)

Interestingly, argon has a long-lived electronically excited (neutral) state, often noted as Ar*, that occurs in abundance in plasmas. Its potential energy is ~11.5eV, so when it collides with organic materials, it transfers bond-breaking amounts of energy. Better yet, if it 'decays' in a single step, it radiates in the UV portion of the spectrum. (Since I know little about radiative energy losses, take this with a large pinch of salt.) An energy of 11.5 eV translates into a wavelength of 1080Å. Since, for our eyes, blue light ends at 4000Å anything less is in the UV range. The radiative decay of Ar* is classed as VUV (Very Ultra Violet?)

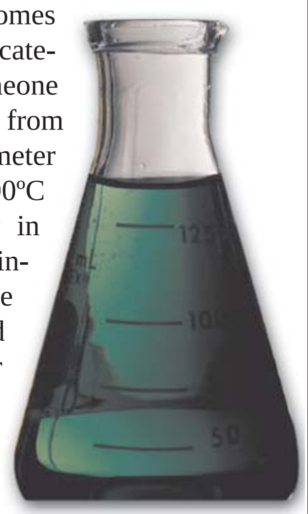
And what's so wonderful about radiation rather than collision? Do you recall the last time someone ended a lame story with "You had to be there!" Well. . . with radiation you <u>don't</u> have to be there. The Ar* loses its energy over here and the radiation interacts with a molecule over there. It's probably something to do with the wave function collapsing but who cares. . . Great stuff this Ar*!

2. Oxygen Plasmas

Oxygen plasmas are recommended for 'hydrocarbon' contamination since the organic material converts to CO_2 and water vapor. And before you complain that you already have enough water in your chamber, note the plasma is energetic enough (hot - if you wish a simple term) to retain the products in the gas phase. And, since there is an O_2 gas flow, they are shoo-ed towards the pumps.

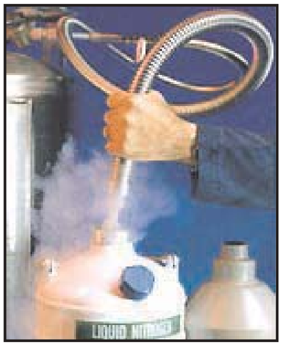
But it's caveat time. Remember the tongue-in-cheek intro that claimed a plasma is everything gone wrong? If the plasma products reach the pump, you'll quicky learn just how un-tongue-in-cheek that remark was. One rotary vane pump sent to our Pump Repair Dept. had been appropriately drained, or so the assigned technician thought since nothing ran out the drain plug. Imagine his surprise when, on removing the outer case, he found a large greenish Jello-like blob surrounding the immoveable pumping mechanism. Yep, this very ordinary pump had been filled with hydrocarbon oil, connected to a plasma process, and had promptly seized. When it comes to most pumps and pump oils, plasma products are unrelentingly destructive! And you **must** expect plasma products to reach the mechanical pump. What would I do if I wanted to plasma clean my system? Oh, I'd buy a plasma-qualified pump, fill it with Fomblin fluid, and have it sitting in the wings until I felt the urge to spring clean. But noting Pennsylvania's weather for early March 2003, I ain't never gonna get that urge.

Chemical



This chamber cleaning method comes under the 'Strange But True' category. In *J. Vac. Sci. Tech* someone reported cleaning surface water from an aluminum tube (9.2 cm diameter x 1.4 m long) by heating it to 100ºC and pressurizing it to 40 torr in 2,2-dichloro-propane for 10 minutes a total of three times. The 'true' part is, the tube pumped from atmosphere to 1 x 10^{-8} torr in 2.9 minutes or 1/300 of the time an untreated tube took. The 'strange' part is, this reaction produces HCl gas. So, fast re-wind to the 'caveat' paragraph of the **Plasmas** section above and add the appropriate words to cover **Chemical** cleaning too.

Cooling



Freezing out vapors on a cold surface is often used in very large vacuum chambers built, for budgetary reasons, from mild steel. Think of freezing as increasing the effective pumping speed rather than reducing the gas load. Huge space simulation chambers use an all-enveloping internal LN2 cooled shroud to simulate the temperature of space. But the shroud traps any water desorbing from the mild steel outer shell or oil vapor molecules from the massive diffusion pumps providing the pumping oomph.

There are two ways of adapting this technique to more regular vacuum chambers. (a) If the chamber is brought to atmosphere infrequently, find a (large) spare port and mount to it a side-arm with an LN2 reservoir in it. Such a trap can dramatically affect the chamber's base pressure. (b) For chambers that are regularly opened (many times a day) to the atmosphere, install one of Polycold's Meissner traps. Their pumping speed on water vapor can work wonders on base pressure and pump-down speed.

Getters

Another method of boosting the effective pumping speed is with an evaporable or non-evaporable getter (NEG). (Note: SAES is a primary supplier of getters worldwide and has produced, some wonderfully informative literature. **See:** ***www.saesgetters.com***)

1. One-Shot Evaporable Getters

The most commonly used material is barium, packaged with a little heater/reflector boat. Once the chamber is under vacuum, the barium is flashed and selected areas of the chamber walls are coated with a thin film that reacts with most active gases/vapors to give low vapor pressure compounds. Of course, the number of people who feel comfortable having a metal film evaporated inside their vacuum chamber can be counted on one finger. Turns out, however, the number of people who own such a vacuum chamber probably numbers several hundred million. An evaporable getter is used in every old fashioned TV or computer monitor. After sealing the glass envelop the barium is flashed as the final, and ongoing, pumping stage.

2. Multi-Shot Evaporable Getters

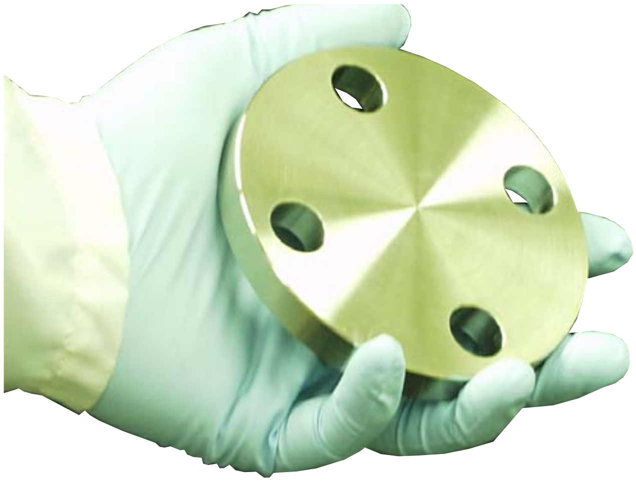
Yes, you'll think this is another bogus description because I'm talking about the titanium sublimation pump. But what is a Ti sub pump if not an evaporable getter? For those of you unfamiliar with these devices, titanium is evaporated from a filament, sometimes in an ion pump, sometimes in a separate side volume with LN2 cooled walls. The evaporation power supply is switched on for only a few minutes. The fresh Ti film that is created is very reactive and an excellent pump of water, oxygen, nitrogen and hydrogen. When the film has mostly reacted and its 'pumping speed' slows, another layer of Ti is evaporated.

Combined with an LN2 cooled surface, Ti sub pumps are very effective aides to ion pumps pulling into the UHV range. But a little caution is appropriate when using them. They can spray Ti from the attachment port into the chamber and coat insulators. But it's a line-of-sight spray and easy to stop with a baffle.

3. Non-Evaporable Getters (NEGs)

The idea of using NEGs is perhaps less daunting than using evaporable getters. Some zirconium alloys have the peculiar property of providing surface reaction with active gases. When the surface is 'full', heating the NEG to a higher temperature causes the reacted alloy to diffuse into the bulk metal and provide a clean alloy surface ready to adsorb more gas. NEGs can be installed as free standing devices or as flanged mounts connected at a spare port. Just note that neither the evaporable nor non-evaporable getters do a good job on hydrocarbons.

Prevention



Maybe I've left it too late to say this, but an ounce of prevention—blah, blah, blah. It is imperative to remove machining oils or sweat from any components or equipment added to the chamber. Once cleaned, no vacuum surface should ever be touched with your dirty pinkies, or even your clean pinkies. **Always** wear gloves. And a note about gloves: they **must** be barriers to sweat and body oils; they **must not** contain softening plasticizers; they **must not** be coated with slip release agent; they **must not** be made tacky with some stickum; they **must not** be made from a material that will shed lint; and they **must not** be coated with talcum powder. Apart from that, they can be anything you want.

Conclusion

With most cleaning methods designed to produce a lower base pressure, the line between success and screw-up is thin. Take your time deciding which is best for your situation, install the equipment with care, and watch it like a hawk while it's working. The idea of slapping on some tape heaters, firing them up at 5:00 pm and pushing off for a night of disco dancing is daft. Cleaning has to be important to you, or why bother!

Bakeout Temperatures

For this *highly idealized* example, assume the desorption rate at 200°C is only 10 times faster than at 100°C. Consider two identical 30cm diameter spheres, attached to identical pumping stations and continuously pumped, each with one monolayer of water molecules covering the interior surface. At 1 x 10^{-6} torr there is ~6000 times as many molecules on the surface than in the gas phase.

When one sphere is heated to 100°C, say it reaches 1 x 10^{-5} torr. The pressure change tells us there are now 10 times more molecules in the gas phase than there were at room temperature. This means the surface/gas phase ratio is reduced to 600:1. The other sphere is heated to 200°C. Since the desorption rate is 10 times faster than the 100°C sphere, it reaches 1 x 10^{-4} torr and its surface/gas phase ratio is 60:1.

At constant pressure, the rate at which molecules leave the surface equals the rate they enter the pump and are removed from the system. That is, the 200°C sphere is removing gas permanently from the system at 10 times the rate of the 100°C sphere. In other words, if both spheres are pumped for equal times, the 100°C sphere will retain 10 times as much gas. Which means, to remove an equal amount of gas, the 100°C sphere must be heated and pumped 10 times longer.

Recall, however, I assumed the ratio of release rates was 10:1 rather than the more realistic 1000:1. So, the conclusion is—get that temperature as high as you can.

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May 5-6	Society of Vacuum Coaters	San Francisco, CA	902
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