

## How Low Can You Go? Part 2

### Gross Chamber Contamination

In this issue, a continuation of *Lesker Tech Issue 4*, I'm considering an existing leak-free high vacuum system with a base pressure that, in classic Pittsburgh, PA parlance, 'needs fixed'. This is a 'legacy system' and I can do nothing about construction materials or effective pumping speed. And, when I peeked at the system's log book, I found the base pressure had always been lousy. Phew! At least I can blame this mess on the previous operator.

As a further limitation, I'll address only really bad chambers—those with histories as exotic as Madame de Pompadour—that require drastic efforts to bring them back into the high vacuum fold. In the next part of this trilogy, we'll talk about reducing gas loads of less pressure-challenged chambers.

Please note, while I suggest clean-up steps below, don't think for a moment this is the last word of cleaning procedures. At best, this is general guidance to make you aware of approaches. Although its authors have different aims in mind, real procedures for UHV applications are described in the *Red Book* from Daresbury Lab (a synchrotron beam line facility in the UK). Unfortunately, this site's URL changes with every flash from the Lands End lighthouse and the *Red Book's* location gets progressively more obfuscated. But I've had luck using Google and 'red book vacuum' (without the quotes).

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### Donning the Deerstalker

Before we start any evidence gathering, we must first convince ourselves the rotten pressure reading is real. (This isn't the place to discuss pressure gauges but one day, my son, we must have that talk!) Assuming the pitiful pressure reading is valid... get a good flash-light, vent the chamber, take off a couple of large flanges, and spend several minutes staring inside. Note everything worth questioning in your log-book, but don't mess with decisions until the inspection is complete.

- How many bits are no longer used?
- Are there multistranded wires with PVC insulation?

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- Is there more plastic than in the local Toys C Us?
- Were aluminum fixtures anodized after machining?
- Are the deposition shields festooned with guck?
- Do the walls look like Aunt Millie's flaky pastry?
- Are there visible oil droplets on the walls?
- Does the 'white glove test' turn your Kimwipe as black as Newgate's Knocker?
- Is that really a cheese sandwich?  
or
- Does it look perfectly reasonable?

## Decision Time

Since fainting frequently follows fact-finding, put your head between your knees and determine your course of action.

Convert your inventory of 'things to question' into a list of appropriate changes, starting with stuff that should be removed or swapped. For example: replace multistranded coated wires with single wires, insulated by slip-on Teflon tube or strung with fish-spine beads; swap the unknown plastic parts with Teflon or vacuum-compatible machined ceramics; replace deposition shields; reverse engineer the anodized parts and don't anodize them; look for motion devices, electrical feedthroughs, or deposition sources you no longer use and replace them with blank flanges.

Let me point out one action plan that, given its illogicality, I've heard an unbelievable number of times over the years, "Oh, I'll pump the crap away." Really? Weren't you the one just complaining about the base pressure? And haven't you been pumping up to now? Sheez!

OK, we've dispensed with the kid's stuff—what can be removed or swapped. It's time for the hard questions:

- Are the chamber walls oily?
- Are they coated with nasty-looking deposits?
- Worse yet, are there flakes falling to the chamber floor or into the pump?
- Are you going to do something or does the ostrich strategy look like a winner?

Make no mistake, doing something about the chamber walls is the big-leagues in the clean-up stakes. To tackle this type of problem, you must first remove all gauges, valves, pumps, gas inlets, hoses, electrical and motion feedthroughs, manipulators, etc. that are flange-mounted to the chamber. Then, remove all the internally mounted bits. You must leave no screw or bolt un-turned since your

aim is to have a completely bare chamber.

On occasion I'm asked, "Can't I clean the chamber *in-situ* without removing all those parts?" Being of a cruel and sadistic bent, I answer, "Sure! Can I watch?"

## Oily Surfaces

Without twisting the truth too much, I can claim the vapor pressure of a bulk vacuum oil at RT is  $\sim 10^{-7}$  torr. But what about oil visible on a chamber wall? If it is the result of backstreaming from a mechanical pump, then chances are it's the more volatile fraction—stuff that was thermally cracked at mechanical 'hot spots' in the pump. That is, its vapor pressure is probably higher than  $10^{-7}$  torr.

If the oily coat is the result of a diffusion pump venting 'event', when air blew through the boiling oil vapor chemical reactions probably formed two product groups: goopy tarry messes that didn't go far; and high volatility products that blew by the LN2 trap into the chamber. (Yes, we'll talk about trapping, or lack thereof, one day.)

In reality, the implication that higher vapor pressures only arise with visible oil films is untrue. Invisible films of high vapor pressure oils are just as damaging to the base pressure! But, there's a piece of Missouri in all of us and seeing is believing, right?

To remove oil from chamber walls you must find a company offering a vapor degreasing service, preferably one using environmentally safe solvents. An azeotropic mixture of hydro-fluoroethers, n-propyl bromide, and isopropanol is good. This blend dissolves hydrocarbon- and silicon-based oils, removes soils and particulates, fingerprint oils, and 'light ionic residues' which, roughly translated, means salts like sodium chloride from your sweaty palms.

Prepare the chamber for shipping by putting aluminum foil over the ports and wrapping the whole thing in some plastic film (such as Mylar or polyethylene) that is not coated with a 'slip release agent' (see sidebar p.3). Tape the overlapping seams, crate it, and ship it to the vapor degreaser, complete with a box of vacuum-service compatible gloves. Insist that after degreasing, no-one handles it without wearing gloves. No, cancel that! Take the chamber to the degreaser, mount guard from start to finish, and ensure the help doesn't touch its surfaces with their bare pinkies. As soon as it's dry, re-wrap (with new foil/film you took with you) and re-tape for the journey home.

## Side Bar

I have a theory about plastic films and slip release agents—if the film unrolls ‘noisily’, the sound generated is a succession of continuous tiny thunder claps as static charges are parted. One property of a slip release agent is to neutralize this static cling. Therefore, a ‘noisy’ film means no slip release agent. Would any real chemist care to comment? We’ll gladly publish the best confirmation and/or refutation sent in to [techinfo@lesker.com](mailto:techinfo@lesker.com).

But, I hear you mutter, I can do this back at the farm with Kimwipes and acetone. **No you can’t!** Not if you want to live to a ripe old age; avoid visits from OSHA and NIOSH (or your country’s equivalent); and, incidentally, end with a clean chamber. We’re talking about removing the final mono-molecular layer here and the most prolonged and diligent ‘wax on - wax off’ Karate Kid motions cannot possibly equal the efficacy of one good vapor degreasing.

## Dirty/Flaky Surfaces

Here, safety is a primary concern. You don’t want yourself, your maintenance staff, or a cleaning service company unwittingly fooling with highly toxic or carcinogenic crap. So, from the log book determine the likely chemical composition of the dirt or flakes. If it is very nasty, you’ll need to find a company specializing in hazmat clean-up. If the flakes are only the ‘Usual Suspects’ then your action plan should take into account: (a) their chemical composition; (b) the required chamber base pressure; (c) degree of clean-up you need to achieve that pressure.

The point is, getting rid of the Usual Suspects means removing something from the surface by either chemical or physical means. You just want the cheapest, simplest way consistent with getting the chamber back to an acceptable condition. So, armed with answers to (a), (b), and (c) above, you choose an action plan from a list that looks something like this.

### 1. Required Base Pressure: mid $10^{-5}$ torr range.

a. As long as the system has a half-way decent pumping stack, the clean-up for this base pressure level should not be too problematic. With suitable protection for the guy doing the work (industrial rubber gloves, splash proof clothing, goggles, breathing apparatus if he’s sticking

his head in the chamber, etc.), scrub the surfaces with Alconox detergent, a fine abrasive powder, and lots of water. Give it many final rinses with DI water. A high pressure steam jet used after the abrasive stage doesn’t hurt.

b. If the walls are particularly well-endowed with junk, perhaps try Alconox, water, coarse *stainless* steel wool, and elbow grease. (Note: there is anecdotal evidence that scratching stainless steel surfaces with *normal* steel wool causes corrosion. I don’t know if it’s true, but why risk it?) When the major coating has been removed, try cleaning as described in (a) until the surface is bright stainless steel color.

### 2. Required Base Pressure: high $10^{-7}$ torr range

a. If the dirt is soluble in an acid or alkali you can handle, then clean it in-house observing *all* appropriate precautions, particularly those applying to the safety of you or your help. Mechanical agitation with stainless steel wool (in suitably gloved hands) assists dissolution. Rinse away all the acid/alkali (appropriately capturing and neutralizing—don’t dump raw acids or alkalis in the sewer system) and finally rinse with lots of DI water.

b. If the chamber is aluminum, hold the phone! Aluminum is amphoteric, reacting with any strong acid, or alkali.

c. If there is no known or acceptable solvent but coverage and depth is not extensive then try the Alconox, tap water, and a fine abrasive powder scrub treatment. Rinse with lots of DI water. **Do not** use products like a jeweler’s rouge dispersed in yak fat. That rubbish will make the outgassing worse, not better.

d. Coverage is extensive and deep—have the chamber bead blasted by specialists. Of course, you duct tape the sealing surfaces. You can’t expect ‘Bead Blasting Is Us’ to know where your o-rings or copper gaskets sit. After they have done their thing, use a steam cleaner first and then vigorously scrub the chamber with Alconox and tap water. Rinse with lots of DI water.

### 3. Required Base Pressure: $\sim 10^{-9}$ torr

a. If the degree and depth of crud coverage is not too bad, tape all the flange sealing surfaces and send it to a electro-polishing service company that knows its way around vacuum chambers. I also suggest you send a roll of that plastic film with no slip release agent and ask the electro-polisher to be generous with its application after the chamber has dried.

b. If there is crud enough to choke a horse and you really want this chamber to reach  $\sim 10^{-9}$  torr, then you need a double whammy. Send it first to a bead blaster and then to an electropolisher. Why the second step? Gas load is a function of adsorbed materials and surface area covered by those materials. The effective (atomic level) area of a bead-blasted surface is many, many times larger than the dimensional area. The electropolishing step reduces the area disparity and, therefore, reduces the gas load.

## OK, So It's Clean (kinda!)

At last, the chamber's internal surfaces look more like a real vacuum chamber. But with all that talk of rinsing, they must have more than their share of adsorbed water molecules, right? While I'll deal with removing adsorbed gases/vapors in detail in the next issue of *Lesker Tech*, just to round out this 'preparation' phase of chamber rehabilitation, while the chamber is still empty, let's talk about three issues.

### 1. You and Your Mates

Recognize that you and your colleagues are to a high vacuum what rap is to Ravel or "Nightmare on Elm Street" is to "Mid-Summer Night's Dream"... incompatible.

- You each shed  $\sim 400,000$  skin cells every day
- Your eyebrows contain thousands of dust mites
- You lose a head hair every hour or so
- Your favorite wooly cardigan is a worse shedder than your Sheepdog
- When you speak, you project tiny balls of spit
- Each fingerprint probably transfers several micrograms of oil (perhaps  $10^{19}$  molecules)

Inside a vacuum chamber these "human components" give rise to the very base pressure conditions you are trying hard to correct. So, when you are not working on it, a clean empty chamber must have all its ports covered with aluminum foil and should be in as dust-free environment as you can find. When you are working on it, you should be covered with aluminum foil (or its textile/plastic equivalent) to prevent skin cells, mites, hairs, spit, and fingerprints from reaching the chamber surfaces.

### 2. Those D\*\*\* Components

Before disassembly, every square centimeter of every surface was contaminated to roughly the same extent. If you simply re-mount all the components into the nice clean chamber, when you pull a vacuum, contamination will

transfer back to the chamber walls. Like Typhoid Mary, vacuum surfaces are into sharing. The only sensible thing to do is to clean the components too.

### 3. Water, Water Everywhere

While we can't do much about removing the last few monolayers of adsorbed molecules until the chamber is buttoned up and pumping, at this stage we can remove the 'excess' with the application of heat. In increasing order of effectiveness we can:

- a. Heat the (small) chamber with a hand-held, industrial hot air blower.
- b. Heat the (big) chamber with a series of industrial radiant element heaters or a NFL sideline blower heater. (Choose an electric powered sideline blower. I'm suspicious propane blowers will give off unburnt hydrocarbons or microscopic soot particles.)
- c. Put in a vacuum oven and bake.

Here, I'll digress a moment to generalize about chamber baking. Ignoring limits imposed by the temperature sensitivity of other materials, we bake chambers to some given temperature to achieve certain results:

- Aluminum—baked to  $\sim 200^{\circ}\text{C}$  (max) to reduce water
- Stainless—baked  $\sim 100\text{-}200^{\circ}\text{C}$  to reduce water
- Stainless—baked to  $\sim 450^{\circ}\text{C}$  to reduce hydrogen
- Stainless—baked to  $\sim 1000^{\circ}\text{C}$  to degas bulk metal

The most common stainless chamber bakeout—with the system pumped with its own pumping station, top temperature between  $100^{\circ}\text{C}$  and  $450^{\circ}\text{C}$ —is discussed in the next *Lesker Tech*.

But for open, empty stainless chambers?

Well, it's pretty obvious what we're trying to do with proposals (a) and (b) above—heat the metal for many minutes with the expectation that, the higher surface temperature leads to lower adsorption of vapors. So, while the chamber is still warm/hot, re-assemble and get it under vacuum. If you bake, wait a week, and then assemble, you've wasted the bake time.

Proposal (c) is a whole different animal. Baking an empty chamber or component (destined for UHV service) at  $200\text{-}250^{\circ}\text{C}$  for many hours at high vacuum is what the *Red Book* writers and others do. And very effective it is, too! But with a vacuum furnace capable of  $1000^{\circ}\text{C}$ , you get even more spectacular gas load results.

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Various authors, referenced in O'Hanlon's book *User's Guide to Vacuum Technology*, have measured outgassing rates (in T.L/sec/cm<sup>2</sup>) for various baking treatments as:

- |                                   |                         |
|-----------------------------------|-------------------------|
| A. Unbaked                        | 1 x 10 <sup>-9</sup>    |
| B. 30 hrs @250°C                  | 3 x 10 <sup>-12</sup>   |
| C. 2 hrs @850/900°C               | 2 x 10 <sup>-13</sup>   |
| D. 3 hrs @1000°C + 25 hrs @ 360°C | 1.6 x 10 <sup>-14</sup> |

This means, with an appropriate pumping stack, treatment D gives a chamber pressure almost 5 decades lower than treatment A. And if that doesn't impress you, throw away the traditional whip and chair and take up training tigers using a tooth pick and paper cup because, clearly, nothing in the vacuum world's gonna knock yer sox off!

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