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How Low Can You Go? Part 3

Some Like it Hot!

You'll remember in parts 1 and 2 of this trilogy, we reached a nodding acquaintance with the term **gas load** and discussed how to recover chambers that were 'beyond the pale' gas load-wise.

Here I'll discuss stainless chambers with base pressures that are, metaphorically, as scruffy as your favorite old boots. Some serious spit 'n polish is needed to recover the former luster of either. But you intend to do this without removing anything (other than gas, of course). So, collect your system log-books and follow me. . .

[Oh, a slight pause–I've been persuaded by others that what's written is much too long for one *Lesker Tech*. So, it's goodbye trilogy and hello . . . well, whatever is next! But since it's all written, LT Volume 2 Issue 2 should follow LT Issue 1 as night follows day.]

Heat

Heating is the first line of attack for all gas load issues. So I'll examine the technique and heaters in some detail.

Temperature

Baking a vacuum vessel, necessarily, heats everything that's inside or attached to it. So, the primary question is: how toasty can you make the chamber before the most

thermally sensitive part of the other gear (gasket, valve, gauge, motion feedthrough, pump, etc., etc.) rolls over and plays permanent possum?

If it's 400°C, you're in great shape to reach low gas loads. If it's 200°C - 250°C, you'll get most of the surface water desorbed but not the hydrogen in the steel's matrix. If it's 100°C, well. . . it will help, but you surely won't set world records for low gas loads. Less than ~70°C and you have to wonder if heating is for you. The parts and labor to install heaters will be pricey, given the pretty small effect they'll have.

For my money, the component that will fail at the lowest temperature gets my attention for three reasons. First, what is it? Second, can I replace it with a less heat sensitive equivalent? And third, if I replace it, how much does the temperature limit rise? Yeah, yeah, I know replacing means I'm removing something, but that won't stop me assessing all angles. I want to get the best out of this system. One of our engineers, on reading a draft said, "Are you going to mention plastics like Delrin?" Actually, I wasn't. It's too close to Valentine's Day to dig into horror stories of heated chambers containing plastic parts. But I'm sure you get the idea.

After you find the maximum bakeout temperature is really quite low, the immediate follow-up question is: does a 100°C bake for a long time remove as much adsorbed water as a 200°C bake for a short time? The short answer is. . . no! There is an old adage in chemistry–the rate of reaction doubles for every 10°C rise. No, it's not accurate (Arrhenius plots come with different slopes) but it's close enough for us to guess-timate the gas release rate from a 200°C surface is ~1000 times (2^{10}) faster than the release rate from a 100°C surface. (See side bar in next issue.)

But here I must point out a problem that plagues all forms of chamber bake-out. What if some parts of the chamber are at a steady 200°C while others are coasting at 100°C? Well, you get the dreaded 'double-whammy' effect: (a) gases won't desorb from the cooler regions anywhere near as quickly as they will from the hotter regions; and (b) heavy vapors desorbing from the hotter regions will, on collision with cooler spots, re-adsorb. So, don't for a moment think you can heat this chunk here then, an hour later, heat that chunk there. Bake-outs don't work like that. Unfortunately, many types of bake-out heaters are particularly well-suited to create double-whammy conditions. Just keep firmly in mind–for a really successful bake–all parts must reach the same temperature more or less simultaneously.

A final, but far from insignificant question is: since we now know the upper limit, how rapidly can we take the chamber to that temperature? This is entirely 'condition-of-system' specific, a trial and error thing. However, the overarching limitation is clear–the gas release rate **must not** cause the pump's inlet to exceed its maximum designed pressure. If it does, nasty surprises await you since a(n):

- **Diffusion pump** will show its displeasure by backstreaming all the oil vapor in creation into your chamber, totally defeating the reason for heating.
- **Ion pump** will just whimper and die, maybe permanently, maybe not. But it won't be pumping when you need it most.
- **Cryo pump** will capture the gas load until the watts released doing so exceeds the cooling capacity of the compressor, at which point the pump's cool surfaces heat up and the pump says "Sayonara" to all the gas it previously captured.
- **Turbo pump** will adiabatically compress the gas, like your bicycle pump in days of yore, until it overheats and shuts down (or screws up, literally. There's a story here–I'll tell you one day).

In reality, if your system was designed by someone who knew his/her job, you don't want to exceed an even lower pressure–the **interlock** pressure. To do so will either shut off power to the high vacuum pump or close the main valve. Just when you need the max-most-ult throughput, the valve closes or the pump shuts off? (My editor keeps questioning that "?" but I won't back down. Read the sentence with rising volume, rising pitch, and rising indignation. I'll tell you, when this has happened to me, my first thought was hardly, "Oh, bother." I quickly found out how to disable the interlock. In my defense, I starred the logbook entry so I'd remember to correct it, post-bake.)

It's appropriate to comment on one thing you already know about heating and one thing you may not. There are heaters specifically designed for substrates, crystal growing, pumps, gas crackers, etc. and other devices such as RGAs, B-A ion gauges, e-beam guns, thermal boats, etc. that get hot in normal operation. Unless the chamber walls are deliberately cooled, these devices will contribute to chamber heating during normal operation and help desorb gases.

The more obscure, but critical, point concerns **cryo pumps**. Even if you avoid the Sayonara effect noted above, the cryo pump will still lose its cookies if the first stage baffles face a temperature much higher than 80°C. You have some choices, the first two of which reduce the effective pumping speed: (a) mount the pump around an elbow; (b) mostly close the gate valve in front of it; or (c) take the opportunity to regenerate the cryo and use an ancillary high vacuum pump that's less squeamish about heat.

Types of Heaters

Chamber heaters divide into **external** and **internal** types. The main characteristics are obvious: the former can be made from any materials since outgassing doesn't matter. Their disadvantages are, the thermal energy reaching the chamber walls is much less than 100% of the power input and temperature uniformity is up for grabs. The latter are highly efficient, dissipating all the thermal energy in the chamber walls. However, they are in vacuum, so materials selection is critical.

External Heaters

1. Blankets/Panels

Although an integral part of many external heater arrangements, I don't recall ever seeing a blanket or panel that actually included heater elements. Typically, they simply insulate an air volume that is heated by separate electrical heaters. Blankets are often mineral wool fiber pads, flexible enough to drape, somewhat, over a chamber. Panels are semi-rigid mineral wool pads, sometimes mounted in light-weight frames, with a thermally reflective coating on the side facing the chamber. Blankets are installed as easily as making a bed. Panels, while not difficult to install, require more design thought since you have to form a box-like structure surrounding the chamber. Like your backyard barbeque unit that came in a box, "Some assembly is necessary."

The heaters are either finned metal, ceramic coated, or fan-assisted radiant devices, mounted on a level with the chamber's base-plate. If there is a stainless work top at this level, so much the better. Mount the heaters on it. Naturally, if there are electronic bits-and-bobs or the odd packet of Swedish Fish candy also on this work top, ya godda gettum outta there! The air volume confined by the blanket or panel box is heated and the thermal energy transferred to the chamber by air conduction/convection and maybe a little radiation.

Chamber temperatures for blankets are probably limited to 150°C to 180°C while panel box heaters range between 150°C and 400°C depending on wattage and heat leakage.

2. Mats/Pads

A number of manufacturers make silicone rubber mats or pads with an embedded heater element. Often they are molded to fit a particular tube diameter or chamber shape with cut-outs for side-ports. Custom shapes are available.

The thermal contact between pad and chamber wall is fairly good and presumably the heater element dissipates ~50% of its power in the chamber surface. Aesthetically, they are so much better than other external heaters I'm tempted to call them 'neater heaters'. But their temperature limit is ~200°C.

3. Tapes

Tape heaters are, essentially, very long, narrow width pads that wrap around the outside of tubes or chambers. Two types are available: (a) woven glass or mineral wool fiber insulating a resistance wire heating element and; (b) flexible silicone rubber covering the element.

Some *woven tapes* have top temperatures way up there, like >700°C. Power enters from either end of the tape, presumably to put distance between the parts at full line voltage ('mains' in the UK). In the dim past, some tapes had very open mesh weaves. I once reached either side of a pipe, taped with a 240VAC heater, to hold something behind it and managed to touch the tape's ends simultaneously with my wrists. The combined electrical and thermal shock gave me opportunity to post a 'personal best' long jump. *Silicone rubber* tapes are good for temperatures of ~230°C (and have no open weave concerns). And as with silicone rubber pads, if you're into glamor, they look nicer.

Two generalizations about tape heaters: (a) don't double-wrap a tape (unless the manufacturer specifically states it's OK); (b) although the maximum tape temperature looks good, you are unlikely to achieve a chamber temperature anywhere close to it. There are too many bits you just can't tape. Even if half the total watts ends up in the steel, chances are the chamber will still reach only a modest top temperature.

4. Hot-Air Guns

If your budget is less than 100 krona for the next 12-months, all is not lost. Try bidding on a used hot-air gun on eBay. What's a hot-air gun? Well, it looks like a hair-dryer for an unstylish lady. And trust me, any lady who used a hot air gun as a hair-dryer, would be quickly rendered unstylish. . . maybe even bald. Plug one of these goodies into a suitable outlet (one with oodles of amps) and the air flowing from the sharp end may be 500°C-ish.

Naturally, you must be careful where you point this thing, but in the general direction of the chamber walls, ports, and blank flanges is fine. Just don't try cooking a PVC electrical connection, an o-ring sealed flange, a viewport, or some such wimpy device.

Can you control the wall temperature? Hardly. But you'll learn to wag the heater around only a short time in the area with temperature sensitive bits. Further away, you can go full bore for a little longer, keeping one eye on the pressure. All of which means, of course, this method gives temperature non-uniformities in spades, re-doubled!

Internal Heaters

Now you're cooking with gas (figuratively speaking, of course). These devices are particularly efficient since all the power dissipated must intersect the chamber walls on its way to the great outdoors. The overall challenge of internal heaters is two-fold: (a) getting sufficient watts inside; (b) preventing localized over-heating of thermally sensitive components.

The 'sufficient watts' part is difficult to quantify. The surface area, shape, mass, heat capacity, chamber wall's emissivity, room temperature, air flow patterns, external insulation, etc., will all contribute to the final wall temperature. But our System Division's guys tell me, for an un-insulated, 40 - 50L chamber, a distributed 2kW will heat the walls to, *very roughly*, 180°C.

Preventing local over-heating has practical difficulties. You can direct thermal radiation from the heat source with reflectors. . . to a certain extent. But when it re-reflects from the chamber walls, chances are it will shine on the very part you are trying to keep cool. Solutions are system specific. If you need comments on your proposals to stop heat reaching some part, contact techinfo@lesker.com.

1. Infra-Red Lamps

Lamps, particularly halogen lamps, are the devices of choice. Single ended, 500W quartz iodine lamps, with their sockets built on standoffs from electrical feedthroughs on small 2-3/4" OD CF flanges (like our **Stab-In Heaters**) are popular add-ons. You should all know by now what happens if you touch these lamps (hot or cold) with your bare pinkies, so I won't stir that can of worms.

Tubular, double-ended lamps up to 1500W housed in stainless reflectors require more space and are more often original equipment than later add-ons. The reflectors give some initial directionality to the thermal radiation, allowing you to 'point' the heat toward a surface. But tubular lamps have a couple of curious drawbacks that prevent you from getting out all 1500W. Since a number of our competitors are *Lesker Tech* subscribers, I'll just drop that hint and delicately exit left. Got to leave them something to work out, right?

But hey! Why take any notice of my nod, nod, wink, wink innuendo? This looks dead easy–and cheap too! Pop down to your local hardware store, pick up a couple of halogen lamps and. . . well, that's when you discover all lamps are **not** created equal. Use an identical looking lamp to the ones we use, with the same part number, built by a major manufacturer and suddenly you'll have a vacuum chamber coated with metal film. Cadmium. . .? Zinc. . .? Who cares! All the electrical insulators are shorted and the base pressure's gone even further down the tubes. Just think how irritated you'll be when I ask, with obvious glee, "And how do you like them apples?" (In anticipation I've already slipped on my Kevlar vest.)

2. Other Methods

Some of our customers have taken some pretty wild steps to internally heat their chambers. I've heard about, or seen:

- nichrome wire elements–exactly like those you've replaced if you're the family's technical expert and your spouse insisted on an electric-heated clothes dryer.
- graphite serpentines machined to fit some particularly tight space.
- silicon carbide ignitors–which you've also replaced if you're the family's technical expert and your spouse insisted on a gas-heated clothes dryer.

To be continued in *Lesker Tech*, Volume 2 Issue 2.

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