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From

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Conditioning Vacuum Chambers

Confronting a high gas load from a new or newly cleaned vacuum chamber can be daunting, but there are some simple techniques for bringing it into normal condition.

Although there are a plethora of data that allow a vacuum practitioner to model a vacuum system's expected performance in terms of pumpdown, ultimate vacuum, and total outgassing rate, there's still a point where practical anarchy exists. Since all the various data sets that have been so carefully measured are only useful within envelopes of specific parameters that can be defined, we still have to consider that every practical vacuum system will have points in its history where those parameters cannot be related clearly to the conditions within the system.

Those points are several: a new system, a newly cleaned system, or a system taken out of storage, and many others. Chambers, under these stages must be considered to be in an "unconditioned" state that will require that they be placed into a "conditioned" state before their performance parameters can hope to match those in the literature.

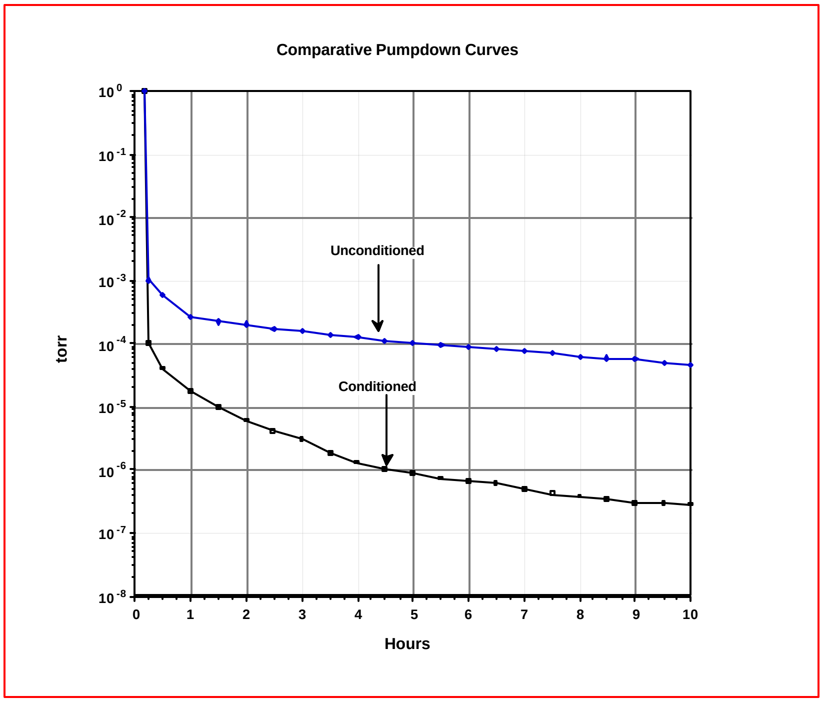
The Unconditioned Chamber

The chamber, in an unconditioned state, will almost certainly present a total gas load that is orders of magnitude higher than the literature's accepted outgassing rates. This condition is traceable to a myriad of possible discreet sources, but taken together, they are all a result of the chamber's history. Whether the chamber is new, newly cleaned, or merely resurrected, the first pumpdown curve will show pressures much higher than what would be expected for a normal working chamber.

A residual gas analyzer (RGA) will usually show that the residual gases contain a much higher than usual predominance of water vapor with various contaminant gas peaks that are from cleaning agent residues and a mix of hydrocarbons.

Every internal surface will be covered with a maximum thickness bed of monolayers of adsorbed water vapor molecules. This thick bed forms a matrix that contains volatile contaminants that encountered the bed as it formed. The trace hydrocarbons found in ambient air will be literally plastered over by the water molecules in the bed.

Additionally, the bed will act as a protective cover over contaminants already on and within the bulk material of the chamber's surfaces. Cleaning agent and degreasing solvent residues fall into the initial contaminant category. These initial contaminants will



Comparing typical pumdown curves of a chamber before and after conditioning shows the difference in gas load when the chamber is conditioned and ready for service.

also potentially be found within every crack and pore of the chamber material, and that includes many non-exposed surfaces such as flange-pair gaps and other virtual leaks.

It's no surprise, then, that the initial pumpdown of a chamber in this condition will be far below expectation in terms of pumpdown time and ultimate pressure. Although extended pumpdown, or pumpdowns, will help in removing some of the contaminant molecules making up the higher than usual gas load, it's usually necessary to subject the chamber to either a single or multiple-stage conditioning process.

The Conditioned Chamber

The chamber, in a conditioned state, will be mostly free of the maximum amount of adsorbed water molecules and included contaminants found in an unconditioned chamber. This will extend to not only surface conditions, but to such contaminant sources as absorbed water and solvent molecules within the thin oxide film covering the metal chamber. Beyond the chamber itself, such other potential contaminant sources as elastomer O-ring materials will be conditioned to a point where absorbed contaminants from within the O-ring's bulk will be greatly reduced as well.

When the entire system is conditioned, the gas load encountered by the high vacuum pump will be within the parameters found in the literature for the materials' outgassing rates. This, then, is the goal that needs to be achieved in order to place a system into operation for its intended application. There are many techniques that can help us achieve that goal.

Conditioning Techniques

If we define conditioning as getting rid of the contamination that provides the high gas loads, it's obvious that we have to focus on the root sources of the contaminants. Sometimes one-by-one.

Elastomer O-rings are a good example. New Viton O-rings are loaded with water that was synthesized internally during manufacture and other components of varying degrees of volatility such as solvents, mold release agents, and unreacted monomers.

These can be removed from the O-rings before installation by the simple expedient of a 150^o C vacuum bake. This should be a standard procedure for new O-rings and older ones that have been exposed to contaminants. Even though they will acquire further water vapor during subsequent air exposure, you can consider them to be conditioned to a standard performance state.

To Bake or Not To Bake

Methods of dealing with the surface and subsurface contamination of the chamber material will depend upon the ultimate vacuum required, the application, and the system itself. Bakeout of the chamber to volatilize water and and hydrocarbon residues would be an obvious expectation. If the chamber is a bakeable, metal-gasketed ultrahigh vacuum (UHV) system, baking the system, especially into the roughing pump, would be standard procedure.

O-ring-sealed systems present bakeout problems. The bakeout temperature, and its effectiveness, will be limited by the temperature limits of the O-ring material. Viton, for example shouldn't be baked much over 160^o C since it begins to slowly break down at this temperature. More importantly, any bakeout must be fairly isothermal throughout the whole system since cold spots will collect the very contaminants that we need to remove.

Flushing the chamber with hot gas, such as nitrogen (N_2) can be effective in removing surface contaminants. If the N_2 is passed through a clean gas heater (commercially available) and allowed to flow slowly through the chamber for maybe 1/4 -1/2 hour, contaminants will be entrained in the flow and carried out through a small exhaust port into the air. Time and temperature parameters need to be worked out for each system. This technique, though effective to some extent, is only a partial solution for high vacuum systems.

Internal Light Bulb Techniques

Considering the problems in baking a system from the outside, it's worth considering baking from the inside. This can be done fairly efficiently by mounting infrared (IR) bulbs within the chamber. The IR radiation will heat the internal surfaces and desorb the adsorbed contaminants, especially water vapor, and provide enough heat to drive contaminants out of the surface oxide layer, pores, cracks, and virtual leak traps.

Drawbacks to this technique are avoiding cold spots and driving volatilized hydrocarbons into the high vacuum pump along with temperature limitations in O-ring systems.

Another approach is to use UV bulbs that emit both 185 and 254 nm radiation. The UV radiation will couple to adsorbed water molecules and raise their energy level sufficient to cause desorption. Additionally, the UV, at these wavelengths, will break carbon-to-carbon bonds so that heavy hydrocarbon contamination can be turned into short chain volatile gases. Tiny amounts of heat will radiate, but this can be considered a non-thermal technique. The UV radiation will also convert large amounts of water vapor to highly reactive OH^* free radicals that will react with hydrocarbons. Energy transfer continues even after desorption so the desorbed molecules remain highly mobile until pumped away.

The main drawback to the UV technique is that it only deals with surface or desorbed gas, and it has little effect upon absorbed contaminants or virtual leak traps.

Combining both IR and UV can result in a very effective and efficient method of conditioning. The heat from the IR will free contaminants that will be dealt with by the UV when they are on the surface or after desorption. This is a classic 1+1=3 situation where their complimentary effects work together.

Glow Discharge

Glow discharge cleaning is very effective in conditioning. Various gas mixtures can be used for particular problems, but inert argon (Ar) can be the most effective for many applications. Ar^* free radicals are amazingly reactive, and they can remove heavy contaminants during glow discharge bombardment.

Summary

Since any new or newly cleaned vacuum chamber will require conditioning before being put into routine operation, there are a number of choices of effective techniques to speed the process. The technique or techniques chosen will depend upon the system, its condition, and the process to be carried out, so careful consideration of technique and needs is required for a successful result.