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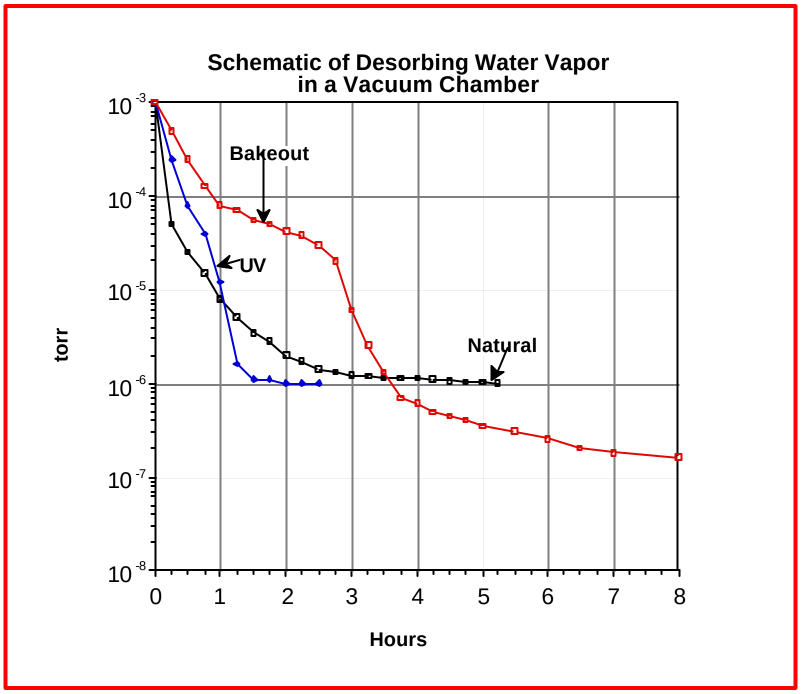
Desorbing Water in Vacuum Systems: Bakeout or UV?

Shorter pumpdown times or lower pressures can be achieved by using either bakeout or UV energy to desorb water vapor. Here's how to make the choice.

Water vapor, desorbing from the internal surfaces of vacuum systems, is a problem that's common to vacuum technology as a whole. Every time a vacuum system is opened to ambient air with its attendant humidity, the internal surfaces become covered with layers upon layers of water molecules. Every item placed within a vacuum system is likewise covered with water molecules. Every pumpdown cycle from atmosphere will go through two overlapping stages. Firstly, the permanent gases making up the air mixture in the system's volume, such as nitrogen (N_2) and oxygen (O_2) will be pumped away. Secondly, the water molecules desorbing from the internal surfaces must be pumped away. Once the pressure is in the low millitorr range, the desorbing water makes up over 99% of the total gas load. If, for example, you turn on a residual gas analyzer (RGA) in the high 10^{-4} torr range during a pumpdown from air, the O_2 and N_2 will drop off-scale almost immediately while the water vapor peak will seem to be fixed. Alternatively, if you pump a system down to its ultimate pressure of, say 10^{-6} or 10^{-7} torr, and then backfill it to atmospheric pressure with dry N_2, it will pump back down to its ultimate pressure again in minutes instead of hours. This is explained by the fact that the pumpdown rate is not controlled by the amount of water vapor within the chamber but by its desorption rate from the internal surfaces. Since a certain amount of sorbed water had already been removed by the first pumpdown, the desorption rate is still at the same lowered level it had been prior to the N_2 backfilling, and the low pressure equilibrium could be quickly re-established. The time vs pressure pumpdown curve, then, will be controlled by the changing desorption rate of the water molecules from the system's internal surfaces.

During a pumpdown, the desorption rate will be initially high due to the fact that the last formed layers will have weaker water-to-water bonds[1,2] than those closer to the chamber wall. With pumping time, the desorption rate will drop lower and lower as the stronger-bonded molecules are exposed. Taking the fundamental vacuum relationship Q=SP as desorption rate = pumping speed x pressure, it is easy to see that the desorption rate needs to fall to some given point before a given pressure can be achieved with a fixed pumping speed. The normal desorption of water vapor requires that the molecules absorb enough energy to overcome their bonds before they can desorb to be pumped away. This is usually due to thermal energy transferred from the chamber itself to the molecules. It follows, then, that if additional energy is applied to the molecules, the desorption rate can be increased while the energy is being applied. If this is done on a temporary basis during a pumpdown, a much lower final desorption rate will result when the energy source is removed, since only the more tightly bonded molecules will remain undesorbed.

There are two efficient and effective methods of energy transfer that are commonly used: heat and ultraviolet (UV) light. Although each method has its own advantages and disadvantages, the choice of which method to use for a given application depends upon the performance goals to be achieved. In general, the goals can be simplified to two options. The goal might be to achieve a lower pressure in a given time, or it might be to achieve a given pressure in a shorter time. Both methods of energy transfer will accomplish either of these goals, but each method needs to be considered in light of the application and the system itself.

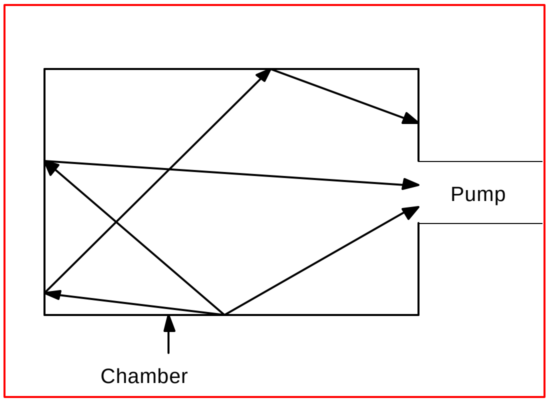


Pumpdown performance of an O-ring sealed system showing the effects of both desorption techniques against no forced desorption.

Bakeout is the term most commonly used to refer to the application of heat to stimulate desorption. In many minds it's associated only with ultrahigh vacuum (UHV) systems. Granted, it's pretty nigh impossible to achieve UHV without bakeout, but UHV systems have many more considerations beyond mere bakeout. The proper pumps, seals, and materials are just a few examples. Heat can be applied to almost any vacuum system to temporarily increase desorption. Heat, applied externally to the chamber, is the most common method, with heaters mechanically clamped or bolted to the outer surfaces for good thermal contact. For UHV systems, 250^0 C is usually the required/recommended temperature. This temperature is high enough that Viton can't be used because it degrades rapidly at temperatures above 180^0 C, and perfluoro elastomers that can withstand the temperature have too high a permeation rate[3], as does Viton, to allow UHV pressures to be attained. This leaves metal gaskets as the only reasonable choice for bakeout at these temperatures.

Bakeout, however, can be useful for O-ring-sealed systems that are used in the high vacuum region. The water vapor desorption rate is a function of both time and temperature. A lower temperature will just require a longer time at that temperature to provide whatever degree of diminution of the desorption rate is required by the pumpdown specifications. This means that a temperature of, say, only 100^0 C will increase the desorption rate during the temperature cycle to reduce the pressure achieved in a given time than would be possible if no means of additional energy transfer were to be provided. The problem here is time. If a stainless steel chamber is heated by external heaters, the low thermal conductivity and high heat capacity of the stainless steel will greatly lengthen the time between application of heat to the thermal energy reaching the sorbed water vapor inside the chamber. Conversely, the time to cool will be long as well. If, as in most practical vacuum systems, there are internal arrays, the heat has to be transferred through some solid connection with the heated wall. This can require very long time periods. The time lag problem disappears, in a practical sense, if aluminum chambers are used since aluminum has a very high thermal conductivity. One way around the time lag problem is to heat the inside of the chamber with infrared (IR) bulbs mounted inside the chamber. UHV compatible IR bakeout systems are commercially available.

UV energy avoids some of the problems of thermal energy. A UV bulb(s), mounted within a chamber will transfer energy to the sorbed water molecules if the bulb emits the correct wavelength[4] of UV light. If you think of spectroscopy, the mechanism for energy transfer is fairly simple. Specific molecules will absorb specific wavelengths of light. The sorbed water molecules will absorb the UV light until they become sufficiently excited to overcome the water-to-water bonds and desorb. The UV light is reflected internally enough that line-of-sight exposure is not required. UV excitation is a very efficient method of energy transfer since the energy is absorbed directly by the water molecule and only negligible amounts of heat are radiated. A pumpdown time can usually be cut in half if only a total UV energy 2.5 mw/in.^{2} of chamber surface area is provided while doubling the energy to 5 mw/in.^{2} can usually cut the time to one-third.



Behavior possibilities of a desorbing water vapor molecule showing the statistical chance of re-sorbing or entering the pump.

Choosing the best method for a given application requires an understanding of the behavior of desorbed water molecules. A water molecule, once desorbed, will not necessarily be pumped away. Although a desorbed molecule might enter the pump, it's more likely that it will impinge upon another spot in the chamber. When it impinges, it might resorb or it might merely bounce. This results in a chaotic condition of molecules desorbing, re-sorbing, and generally moving around within the chamber until they finally wander into the pump. If thermal desorption is being used, the higher temperature of the chamber will reduce the amount of re-sorption and increase the chances of a molecule entering the pump. If, though, the chamber is not heated isothermally where the temperature is not constant throughout, the molecules will tend to selectively re-sorb on colder spots. Upon cooling the chamber, the previously colder spots will become an enhanced source of water vapor. Although this problem is avoided with UV energy which will flood the whole chamber, the UV energy will not penetrate into the trapped volume of virtual leaks or reach the surfaces of O-rings recessed between flanges. Thermal energy, though, will tend to drive water vapor out of these sources. Another major difference to consider is the fact that UV energy is only useable on surfaces. If a "gassy" material needs to be placed within a chamber that's either hygroscopic or porous, UV energy will only desorb water from the material's surface while thermal energy will drive it out of the material's bulk. This means that UV energy will effectively desorb the water from the surface, but that water will then diffuse out of the bulk to the surface and cause continual desorption.

The specific application will then help make the choice between the two methods of energy transfer. A batch system that is continually cycled from air-to-high vacuum will probably be better served by UV desorption since the pumpdown time can be shortened enough to provide many more product runs per day. A system, sealed with Viton O-rings, could have the pumpdown time reduced by 1/2 to 2/3 of the time usually required to achieve a given pressure. The same amount of UV energy could also achieve about a decade lower pressure in the same amount of time as is usually required. The resultant pressure could be even lower if vacuum pre-baked O-rings were used. Systems and applications that require UHV pressures will require thermal energy at temperatures too high for O-rings so metal gaskets will be required, and the inability of UV to penetrate to the O-rings is no longer a concern. The time penalty for heat-up and cool-down cycles becomes less important when the need is to reduce the desorption rate of the water vapor to almost nothing. In many applications, a combination of both methods used together can utilize the main advantages of both to achieve UHV or even just lower high vacuum pressures in a shorter time.

References

[1] *See #15, “Sources of Water Vapor in Vacuum Systems"*

[2] *See #16, “How To Assess Gas Loads in Vacuum System Design”*

[3] *See #14, “Gas Loads and O-Rings"*

[4] US Patent Number 4660297

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