

SOURCES OF WATER VAPOR IN VACUUM SYSTEMS

It's a well known and generally accepted fact that the residual gases during a pumpdown are dominated by water vapor when pumping down to a process dictated ultimate pressure in the high vacuum region. It often seems as though this condition is merely accepted by vacuum practitioners as an irritating fact of nature that has to be tolerated. The irritation is traceable to the amount of time it takes to achieve a low enough partial pressure of water vapor to meet process parameter requirements. The time delay is caused by the fact that the water vapor is clinging to the inner surfaces of the vacuum system with weak bonds. These bonds have energies that lie between the very weak Van der Waals mutual attraction bonds and true chemical bonds. This energy level means that the bonds can be broken fairly easily by applying small amounts of energy; usually thermal heat transfer from the chamber walls at ambient temperature. In a practical sense, the water can't be pumped away until it leaves the surface, so the pumpdown curve is controlled by the desorption rate of the water molecules. Figure 1 shows the desorption rate of water as a function of time. Note that the shape of the curve closely resembles a normal pumpdown curve. Since time is money, it's worthwhile to consider ways to speed a system's pumpdown. As in most vacuum problems, there's no single "silver bullet" solution. It's necessary to make a number of small solutions that will

add up to one large one. A better understanding of the sources and behavior of water vapor is the key to solving the pumpdown time problem.

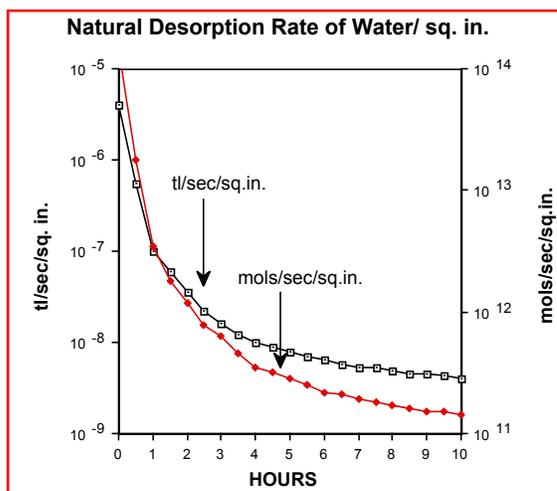


Figure 1

Initial Sources

At some point, all water vapor that is found in a vacuum system came from the atmosphere. Consider that air at 25⁰ C and 50% relative humidity contains 12 torr of water vapor. Since the molecules that make up the gas mixture that we call air are in constant motion, it's certain that any material exposed to air will be continually bombarded with water molecules. Any material that is capable of absorbing water into the bulk, such as plastics or elastomers, will become saturated if they are

exposed long enough. Additionally, any surface will adsorb a certain number of monolayers of water molecules. In many cases, the inner surface of a vacuum chamber can have several hundred monolayers of water molecules adsorbed on the surface at the start of pumpdown. The total amount of water, either adsorbed or absorbed, is proportional to the amount of water in the air during exposure and the duration of exposure. It's not uncommon to have a system go into operation with an initial condition of saturation of both surfaces and bulk.

Tracking the Sources

One source of water vapor that is often overlooked is the continual introduction of water saturated work. A practical process system requires fresh work to be introduced into the chamber from the atmosphere, and any water vapor present will be then transferred into the chamber. This applies, in varying degree, to both batch and loadlock systems. Although it's impossible to totally avoid introducing water along with the work, it can often be minimized. In some cases, selection of tooling to avoid highly hygroscopic materials is possible. In general, though, removal of as much water as is possible and practicable just prior to introduction into the system helps greatly. The application of heat, vacuum, or both will remove large amounts of water even though it's necessary to expose the work to the atmosphere at some point. If the exposure period is kept short, the total amount of water introduced will be kept to a minimum. Any water that's not introduced will not have to be pumped away prior to initiating the process. In some cases, it's easily possible to store pre-treated work and tooling in a protective environment such as a desiccator or a evacuable storage container.

Further exposure to atmospheric water vapor occurs whenever a system is backfilled to atmospheric pressure for changing work or maintenance. If the chamber is backfilled with air, the water molecules in the air will instantly begin to sorb onto all the fresh internal surfaces. The first layer down will bond relatively tightly to the surfaces, but additional layers of water-to-water bonds will form. The polar nature of the water molecule sets the bonding energies, but, as the water piles up, the bonds will become continually weaker as the sorbed bed becomes thicker and more disordered. This means that the last molecules down will desorb much more quickly and easily than the molecules deeper in the bed. This helps explain the decreasing desorption rate with time shown in Figure 1. In fact, most processes are initiated while there is still coverage of the surfaces but where the desorption rate has reached a low enough level for the process to tolerate the partial pressure of water still in the system.

The obvious answer to the sorption of water during air exposure is to avoid exposure. To some extent, this is solved by loadlock systems

where the process chamber is kept under vacuum while the loadlock is exposed. In this case, the only water vapor exposure of the process chamber is from the residual partial pressure in the loadlock during cycling into the chamber and whatever water might still be on or in the work.

In a batch system, though, air exposure cannot be avoided. Backfilling with N_2 or inert gas is useful, but can also lead to a fool's paradise situation. It is often assumed that the backfill gas will bar water vapor from surfaces, and there is some effect to bear this out. Once the chamber is exposed to air for loading of work, the water vapor in the air will instantly diffuse throughout the volume of backfill gas due to the constant molecular motion. Improvements in pumpdown time following replacing the backfilling gas with inert gases or N_2 instead of air is more a function of pre-filling the virtual leak voids with water-free gas. Desorbing water vapor has the property of desorbing and then resorbing on the first surface it encounters before re-desorbing, etc. until it is finally pumped away. A water molecule can easily spend an extended period of time within the surfaces of the virtual leak void before finally escaping into the main chamber.

Avoiding air as a backfill gas will reduce the amount of water vapor to be pumped away during desorption of surfaces, and it will also help in the case of elastomer O-ring outgassing. The outgassing of O-rings can be a major source of water vapor in a system. In fact, the outgassing rate of 1 linear inch of Viton¹ is about equal to the water desorption rate of 18 square inches of internal surface. The desorption of water from Viton is from the surface, but the bulk near the surface is a major source of water that diffuses to the surface during pumpdown. Backfilling with inert gas or N_2 will load this volume and help bar water vapor in much the same fashion as with a virtual leak.

Getting It Out

Once any water vapor is introduced into the system, it has to be removed to some specified residual concentration. Pumping for a long enough time and accepting the "natural" desorption rate will work, but this discussion started with the need to reduce the pumpdown time. The only solution is to desorb the water faster and to provide enough pumping speed to meet the higher desorption rate gas load. This means that energy has to be applied to the sorbed water bed.

Mechanical energy can be applied during the roughing cycle by bleeding in a dry gas while pumping. The bleed gas impacts the sorbed water molecules and essentially knocks them loose. If the pressure is held high enough, the desorbed water will be entrained within the gas flow into the roughing pump.

Each system is different enough that some experimentation is required for maximum effect, but holding the pressure for a number of minutes at a few torr is a good starting point. This technique is not as good, in terms of total water removal, as the following techniques, but it will remove a lot of water and might be sufficient for many processes.

Applying thermal energy is a common technique. Heating the sorbed water bed will add enough energy to desorb the water at a higher rate. You get less desorption in the long run by temporarily raising it. The trick is to raise and lower the system's temperature quickly enough to have a low enough desorption rate at a specified time. Thermal energy can be imparted through the chamber walls by using external heaters, or heat can be transferred directly to the water molecules by using internally mounted heat lamps.

UV lamps, internally mounted, can be used to transfer energy directly to the water molecules without heat. This technique doesn't require a heat-up and cool-down period. It also reduces the problem of elastomer damage by heat. The UV, of the appropriate wavelength, is absorbed by the water molecules in much the same way as is found in spectroscopy where a given molecule absorbs a given wavelength of light.

Conclusion

Although water vapor desorption can be a major time-wasting problem in production vacuum systems, there are a number of techniques to reduce the amount of water vapor that gets into a system and to speed its desorption once it does get in. As usual, in vacuum technology, attention to small details is important. You don't really have to accept and live with an extended pumpdown time if you pay attention to those details.

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