SVC Topics

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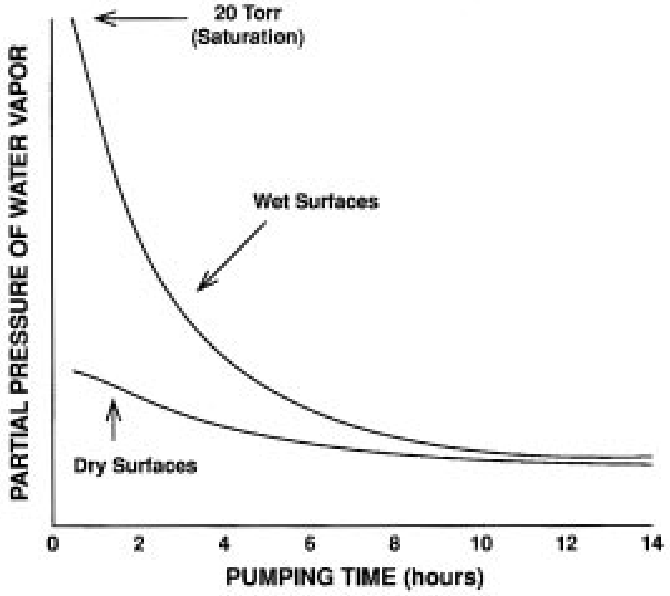
PVD Processes: Water Vapor

Water vapor often presents a major variable in PVD processing, and is generally the most common contaminant in a good vacuum processing system. Water and water vapor in the vacuum system increase the pump-down time and, during the deposition process, can increase contamination to an unacceptable level. In the extreme, gas cooling during rapid pumping can actually cause condensation and “rain” in the vacuum chamber.

Removing Water Vapor

Water vapor is much more difficult to pump away than a gas, because the water vapor molecule has a longer “residence time” on a surface than a gas molecule. If many adsorption-desorption collisions are necessary for the water molecules to be removed, then it will take a long time to reduce the partial pressure of water vapor in the chamber. The figure shows the pump-down time for water vapor in a vacuum chamber, starting with dry surfaces and with wet surfaces. Note that it can take hours to reduce the level of water vapor in the system if the surfaces start out wet. If there is standing water in the system, rapid evaporation (evaporative cooling) can form ice, which will further increase the time to rid the system of water.

The surfaces of materials in the vacuum chamber also affect the removal rate of the water. If the surfaces are porous, water is retained in the pores and capillaries much longer than it would be on a smooth surface. A common source of this surface porosity in PVD processing is film buildup on chamber surfaces and on fixturing. Removal of water vapor becomes more difficult depending on how long ago the surfaces were cleaned. Roughening the surfaces, such as through abrasive cleaning, will also increase the surface water retention.



Examples of pump-down curves for water vapor, starting with dry surfaces and wet surfaces. The saturation (100% relative humidity) vapor pressure of water at room temperature is about 20 Torr.

If the partial pressure of the water vapor in the system is high, compression-type vacuum pumps, such as the oil-sealed mechanical vane pump, will compress the water vapor above its saturation vapor pressure and condense water in the pump. This water will mix with the oil and cause the oil to “froth,” reducing pump efficiency and causing vane wear. Removing the water from the oil can take appreciable pumping time. Mechanical pumps are usually fitted with “ballast valves” that allow the dilution of the moist air from the vacuum chamber with dry gas. This increases the pumping load and extends the pump-down time. It is important that the gas into the ballast valve be dry—moist gas will only exacerbate the problem.

Turbopumps have a low pumping speed for water vapor because of the many surface adsorption-desorption events necessary for the water molecule to pass through the pump. Some turbopumps have heated stages to increase their pumping speed for water vapor. Some hybrid turbopumps have a cryo-stage to pump water vapor rapidly. Although cryopumps have a high pumping speed for water vapor, if there is a lot of water vapor, the pumps have to be “regenerated” often to remove the accumulated water. Diffusion pumps more rapidly pump water vapor.

Crowding of fixturing in the chamber reduces the conductance of water vapor to the vacuum pumps. Large-area, in-chamber cryocondensation surfaces (Meissner traps), which freeze-out the water in the form of a frost layer, are good for pumping water vapor because the conductance of the water vapor to the pumping surface can be made high.

The table shows the vapor pressure of water as a function of temperature. In order to reduce the vapor pressure of the water to a low level, it must be

Equilibrium Vapor Pressure Of Water

Temp (°C)	Vapor pressure (Torr)
-183	1.4 x 10^{-22}
-100	1.1 x 10^{-5}
0	4.58
20	17.54
50	92.5
100	760
250	29,817

cooled well below freezing. Cryocondensation surfaces operate at about -150 °C and are cooled by hydrocarbon refrigerants. At that temperature, the vapor pressure above the frost is an inconsequential 10^{-15} Torr. The thermal conductivity of ice is rather poor, so as the frost layer thickens, the ability to cool the surface decreases to the point that the cryocondensation surface must be regenerated by heating and "defrosting" the surface.

Care must be taken that heat produced during processing does not heat the surface of the frost and cause water vapor to be returned to the vacuum environment. This often means that the cold surface must be shielded from the source of heat.

Controlling Water Vapor

The best procedure for minimizing and controlling water vapor in the vacuum chamber is to minimize its introduction in the first place. This can be done by:

1. Backfilling the vacuum system with a dry gas,
2. Minimizing the time the system is open to the ambient,
3. Maintaining a flow of dry gas through the chamber while it is open,
4. Keeping the chamber walls and surfaces warm to prevent condensation,
5. Wiping down the vacuum surfaces with anhydrous alcohol,
6. Drying and warming the fixtures and substrates before they are introduced into the deposition chamber, and
7. Controlling the humidity in the processing environment.

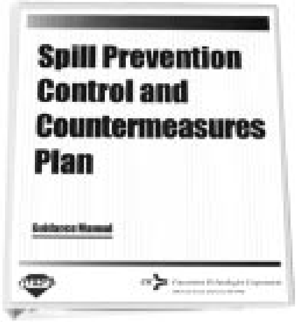
Large volumes of dry gas can be obtained from the vaporization of liquid nitrogen (LN2), usually from above the LN2 in a tank (1 liter of LN2 gives about 650 liters [stp] of dry gas), by compression and expansion of air or by using high-volume air dryers. The relative humidity in the processing environment can be regulated by drying the air through refrigeration, heating to the desired temperature, and introducing water vapor to the desired level by using nebulizers that spray a fine mist into the air. It is useful to keep a record of the relative humidity in the processing environment. High humidity can often be correlated with abnormal pump-down times or other processing problems.

When introducing substrate materials that can absorb moisture (*e.g.*, polymers or porous materials), the history of the material can be an important variable in the amount of water vapor released in the deposition chamber. If the material has been processed or stored in a moist environment, it may contain appreciably more water than if processed and stored in a dry environment. In order to have a reproducible process, the history of these materials must be controlled, and perhaps the materials outgassed (vacuum-dried) before they are introduced into the deposition chamber. In some applications, water vapor and other volatile materials may be sealed in by using a dense basecoat.

In some cases the design of the system or chamber can be optimized for handling water vapor. In some web coaters, for example, the web material is unwound in a separately pumped vacuum chamber before it is introduced into the deposition chamber. This isolates the deposition chamber from most of the water vapor released during the unrolling operation.

Removing water vapor from surfaces during pump-down can best be done using a plasma. Surfaces in contact with the plasma are subjected to low-energy ion bombardment (ion scrubbing), which desorbs the water from the surface. It is important that the desorbed water vapor be flushed away by the gas flow during this plasma cleaning. ***P&SF***

CTC Guidance Manuals For Environmental Plans Available from AESF



Spill Prevention Control and Countermeasures Plan

Guidance Manual

Federal regulations require industrial facilities, such as finishing jobshops, to develop and document environmental plans. Concurrent Technologies Corporation (CTC) has prepared and owns a variety of guidance manuals that provided step-by-step instructions to assist industrial facilities through the process of developing and implementing these required environmental plans. The CTC manuals may be ordered from AESF's Publications Sales Department. The guidance manuals available are:

Stormwater Pollution Prevention Plan (SWPPP)—Virtually all NPDES stormwater permit holders must develop and implement a SWPPP.

Spill Prevention Control & Countermeasures (SPCC) Plan—Under the Clean Water Act, facilities that store certain quantities of oil and oil products must prepare and maintain a SPCC Plan.

Pollution Prevention Plan (PPP)—Federal facilities must develop and implement a PPP. Privately owned facilities, pending the applicable state regulations, may be required to do likewise.

RCRA Contingency Plan—Most facilities that handle and store hazardous waste must develop a Contingency Plan.

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