Reproducibility in Optical Thin Film Processing

Part 1, the Vacuum and Pumping

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Variations in vacuum and pumping practices used in optical thin film coating processes can cause a lack of reproducibility and adhesion problems with the product if not properly handled. The quality of vacuum needed, mean free path effects, cross-over pressures, backstreaming, and checks for the "health" of the vacuum system are discussed and practical recommendations are made.

Introduction

In this series of short articles, we discuss ways to promote the reproducibility of processes for optical thin film coatings. One of the first things needed is an adequate vacuum for the process at hand. Variations in the quality of the vacuum can cause a lack of reproducibility in optical thin film results. Film density and index of refraction can vary if the pressure for the process is not reproducible. This could cause the spectral results and uniformity to vary from run to run. Adhesion failures may be due to oil backstreaming from both the mechanical pumps (MP) and diffusion pumps (DP). DPs will be addressed here for high vacuum pumps because they are in wide use around the world and are the most vulnerable to backstreaming.

The pressure in a chamber is a measure of what vapor is still in it and what is competing at the substrate with the materials being deposited as a coating. The mean free path (MFP) is an important factor illustrating what is happening in the chamber on an atomic scale. If there were a small amount of residual gas in the chamber with which to collide, the evaporated molecules which collided with the gas would go in any and all directions and would probably also lose some of their energy (heat). If there were large amounts of gas, the molecules might actually lose so much energy by multiple collisions as to condense to solid particles before reaching any surface. Such high pressure depositions can result in powdery films or even dust which falls to the bottom of the chamber. We would like to assume that the vacuum in the chamber is such that the collisions are few and do not significantly affect the result as compared to a "perfect" vacuum. However, this is not always the case.

How Good a Vacuum is Needed?

The MFP is the statistical average distance that a molecule will travel before colliding with another, at a given pressure and temperature. **Equation 1** can be used to calculate the MFP at room temperature. provided films with the highest packing densities [35]

$$MFP = \frac{5.0 \times 10^{-3}}{P\,Torr}\,cm \qquad (1)$$

$$\frac{N}{N_0} = \exp\left(-\frac{x}{MFP}\right) \qquad (2)$$

In **Equation 2**, N is the number of molecules from a starting number N_0 that have not collided after a distance x. **Figure 1** shows, from **Equation 2**, the fraction of uncollided atoms after a distance x measured in MFP. About 50% have not collided by 0.7 MFP, 36.8% by 1 MFP, and only 13.5% by 2 MFP. This implies that collisions in a one meter size box coater are not significant at 1×10^{-6} Torr, where 98% would not have collided, but could be a factor to consider at 1×10^{-5} Torr, where only 82% are uncollided. However, when the pressure is 1.5×10^{-4} Torr, as might be used in reactive oxygen depositions of SiO_2 and TiO_2, only 5% of the molecules reach the other side of the chamber without collisions. In the case of uniformity masking, small changes in pressure at these levels can have measureable effects on material distributions.

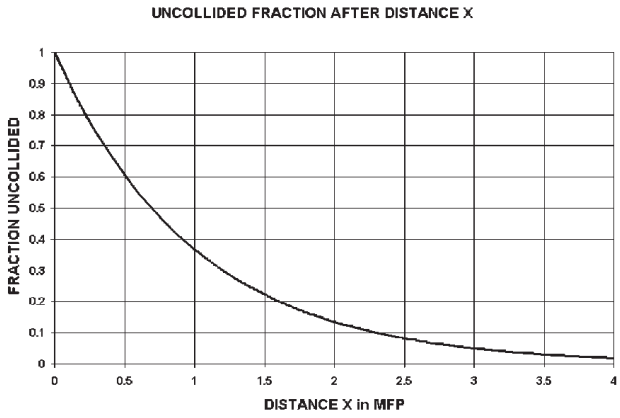

Figure 1. The fraction of uncollided atoms after traveling a distance x measured in MFP.

Typical magnetron sputtering occurs at tens of milliTorr. The MFP for 1.0×10^{-2} Torr would be 0.5 cm. Therefore, at one inch or 2.54cm distance from the sputtering cathode, only 0.6% of the sputtered molecules/atoms would not have collided. Clearly these sputtering processes are not "line of sight" depositions; an atom would typically suffer many collisions before reaching a substrate more than a centimeter away.

It can also be shown that, at 1×10^{-6} Torr, it only takes about a second for a monolayer of whatever gas is in the chamber (usually water vapor) to deposit on a surface. If the material depositing were contamination and it stayed on the surface, we might have some serious adhesion problems.

How Can We Create the Vacuum Needed?

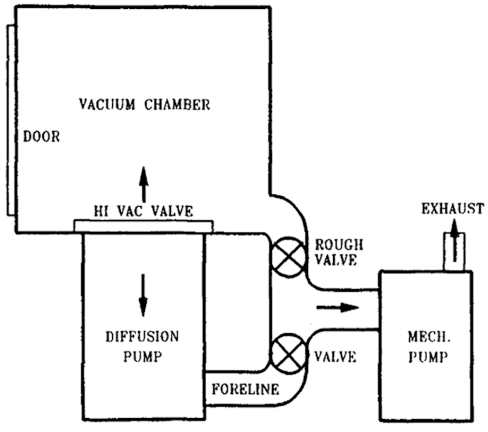

Figure 2. A typical box coater vacuum pumping system.

Figure 2 shows a typical box coater vacuum pumping systems. The chamber has two pumping port valves: the roughing valve and the

high-vacuum valve. When the chamber has been loaded and the door closed, it starts at atmospheric pressure. The types of high vacuum pumps which can achieve the low pressures cannot typically operate at atmospheric pressure. It is therefore necessary to lower the chamber pressure sufficiently before the DP is brought into action. A mechanical rotary vane or piston displacement pump is commonly used to transfer most of the gas from the chamber out to the atmosphere. With the chamber isolated by the roughing and HiVac valves closed, the mechanical pump (MP) is turned on and the foreline valve is opened to the HiVac pump. The HiVac pump might be a fractionating DP as illustrated.

The DP needs to be warmed up to operating temperature while being mechanically pumped. When it is warm enough that you cannot hold your hand on the foreline elbow, and it is pumped down to about 10^{-2} Torr (10 microns), it is ready to use. To shut down a DP, the heaters are turned off while the cooling water is still running. When you can again hold your hand on the elbow of the foreline, the water cooling and MP can then be shut down also.

When the DP is ready to use, the foreline valve can be closed (for a short while) and the roughing valve opened to the chamber. The MP then reduces the chamber pressure to about 10^{-1} Torr (100 microns) where the DP can be used. Then, the rough valve must be closed and the foreline valve opened to clear the DP of any gas which has built up while the chamber was being rough pumped. When the foreline pressure is back to 10^{-2} Torr or less, the HiVac valve is slowly opened to the chamber. If the valve is opened too quickly, the gas load on the DP might be too great and undesirable effects such as oil backstreaming would happen from the DP. To avoid this "breakdown of the top jet," the indicator is that the foreline pressure should stay below 100 microns at all times. This 100 micron for the MP to DP "crossover", and no more than 100 micron foreline pressure is a good rule of thumb ("100 and 100 microns") to avoid oil backstreaming from the MP and the DP.

Note that it undesirable to rough the typical chamber much below 10^{-1} Torr (100 "microns"). This is because at lower pressures the gases flowing down the roughing line to the pump cease to be in viscous flow where they drive the pump oil molecules back into the pump. At the lower pressures, the molecules of MP oil may be in molecular flow, and some can travel back the roughing line into the chamber. Viscous flow would be like a stream which flowed so rapidly that no fish were able to swim upstream against it. However, when the stream was flowing slowly, the fish could swim anywhere. Similarly, in molecular flow, MP oil molecules can flow anywhere, including back into the coating chamber. It will be seen in **Figure 3** that the cross-over pressure to change from MP to DP is a compromise between upper and lower pressure limits, and the pressure at which the transition from viscous to molecular flow regime occurs depends on the mean dimension of the openings involved. The 10^{-1} Torr (100 microns) value mentioned above would be an appropriate lower limit for a 2-inch (50 mm) diameter roughing line and perhaps a 4-inch (100 mm) line. The background for this is discussed in more detail by Willey[1].

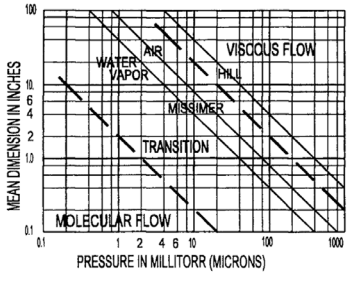

Figure 3. A chart of the pressures and mean gas conduit dimensions where flow changes from viscous to molecular. Dashed lines are typical, see text for other lines.

In past times when pumping cycles were not automated, and even now on some manually operated pumping systems, backstreaming was more of a problem than it typically is now. It was not uncommon for an operator to start a system roughing and to go for a coffee break or lunch. When they returned, the chamber might be pumped down to a few microns, which is big NO-NO as indicated in **Figure 3**. Many operators were unaware of this problem, and many coating runs had adhesion failures due to backstreaming.

Figure 4 shows a recommendation from Langley and Hincle[2] which was preceded by Hoffman[3]. A valve, which could be as simple as a needle valve, is installed upstream of the MP by about 30 cm. (just to the right of the Roots blower), and it is set to give a pressure in that line of about 100 microns. This insures that this pumping line is always in viscous flow; and this avoids any backstreaming from the MP either to the chamber or to the DP. his is particularly good if there is a Roots blower in the system, because the purge gas will not influence the roughing time of the chamber, as it might in the absence of a Roots blower.

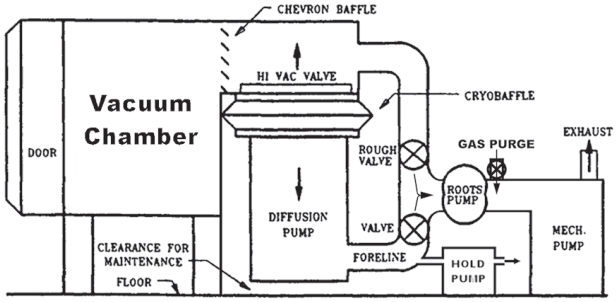

Figure 4. To avoid backstreaming, a gas purge valve (upper right in figure) is set to give a pressure of about 100 microns in the vacuum line leading to the mechanical pump.

It is well to check the "health" of a coating chamber frequently by watching the pump-down versus time and also doing a leak-up test after the chamber is down to a given pressure. In a modern system with a computer display screen, it is easy to have a pressure versus time template of what the chamber does when it is "clean-dry-and-empty" and also an upper bound curve for when it is dirty and needs to be cleaned. **Figure 5** illustrates such a screen. The operator can quickly see if the pumping is within bounds or if there is a problem like a leak, etc.

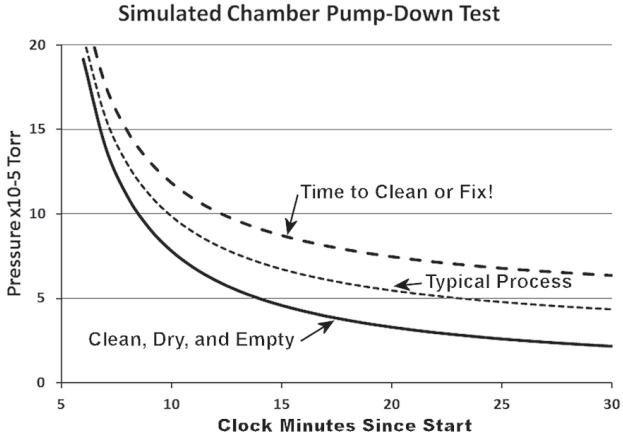

Figure 5. Simulated pump-down screen to observe that conditions are normal during pumping.

Similarly, when the chamber has pumped down normally within bounds, the HiVac valve can be shut and the pressure versus time rise

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observed. References of the rise with time when the seals were tight and the system was "clean-dry-and-empty" can be used, and also an upper bound of a "dirty chamber, but no leaks" rise time. If the latter is violated, it may be time to find and fix a leak.

Conclusion

When the various practices mentioned above are used, the run-to-run variations in the coating results due to vacuum and pumping should be minimal and the processes have a better chance of being the same tomorrow as they were today and yesterday.

References

1. R. R. Willey, Practical Production of Optical Thin Films, Willey Optical (2012).
2. R. Langley and L. Hinkle, "How to Handle Backstreaming in a Diffusion Pumped Process System," Vacuum Technology and Coating, 9, 22-26(2008).
3. D. M. Hoffman, "Operation and maintenance of a diffusion-pumped vacuum system," J. Vac. Sci. Technol. 16, 71-74(1978-9).

About the Author

Ron Willey

graduated from the MIT in optical instrumentation, has an M.S. from Florida Institute of Technology, and over 35 years of experience in optical system and coating development and production. He is very experienced in practical thin films design, process development, and the application of industrial Design Of Experiments methodology. He is the inventor of a robust plasma/ion source for optical coating applications. He worked in optical instrument development and production at Perkin-Elmer, Block Associates, United Aircraft, Martin Marietta, Opto Mechanik, Hughes, and formed Willey Corporation which serves a wide variety of clients with consulting, development, prototypes, and production. He has published many papers on optical coating design and production. His recent books are Practical Design of Optical Thin Films, 4th Ed. (2014) and Practical Production of Optical Thin Films, 2nd Ed. (2012). He is a fellow of the Optical Society of America and SPIE and a past Director of the Society of Vacuum Coaters.

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