Reproducibility in Optical Thin Film Processing

Part 2, Deposition Rate Control

Ronald R. Willey, *Willey Optical Consultants, Charlevoix, Michigan*

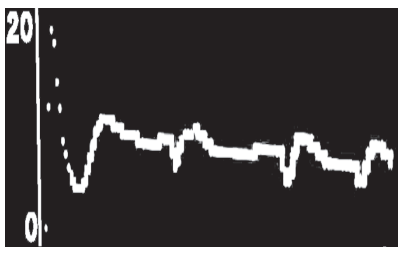
Variations in deposition rate of materials used in optical thin film coating processes can cause a lack of reproducibility of the results and other serious problems, if not properly handled. This is particularly true with vacuum deposition processes that use IAD and/or a reactive background gas pressure such as oxygen, nitrogen, etc.

Introduction

In Part 1 of this series, the pressure in the coating chamber was discussed. Once the chamber pressure is under control and reproducible, it is primarily influenced by gasses which are then intentionally added to the chamber such as oxygen which reacts with the material being deposited, to form TiO_2; or argon, nitrogen, etc., used in Ion Assisted Deposition (IAD). With such processes, an important parameter is the ratio of the arrival rate of the depositing atoms to the reactive gas atoms/ions arriving at the substrate. In the case of IAD, this would be known as the "ion to atom arrival rate" (IAAR)[1].

Deposition processes for optical thin film materials such as TiO_2, MgF_2, etc., may be optimized using Design Of Experiments (DOE) methodology with the three important variables of **pressure** (gas flow, such as oxygen), deposition **rate**, and **temperature.** Part 1 dealt with pressure, and Part 3 is planned to deal with temperature. This present article deals with **RATE**.

The density and thereby the index of most films are a function of the deposition rate. If a process is reactive and/or ion assisted, variations of deposition rate will affect the IAAR and therefore will affect the n- and k-values of the resulting film. The films may therefore be inhomogeneous if the rate varies through the layer. In a typical optical coater, the goal is that, when the shutter opens, the material is depositing at the desired rate and that the rate does not vary until the shutter closes. This goal is **not** often well satisfied in the industry, and therefore can contribute to various problems in the optical and physical properties of the films. Guidance is provided in this article in finding the various settings for a quartz crystal controller which will minimize these problems. Although some of this information may be familiar to many in the field, it is thought to be of enough potential benefit to others that it is reviewed here. **Figure 1** is a photo from the author's typical experience in the industry of an actual rate record for a material deposition as monitored by a quartz crystal oscillator. It can be seen that the rate goes to about 2X the set rate after the start and then to about ½ the set rate before somewhat settling in to a fairly uneven rate. It most cases observed, the operators have little concern in such a case (where they probably **should** be concerned.)



(1)

Figure 1. Actual rate of deposition example from industry.

The Problem

With a reactive gas process, a higher than expected material deposition rates will allow for less reaction time and thereby possibly higher optical absorption in the deposited film, and the opposite with lower rates. With a constant flux of arriving **ions** but a variable rate of arriving **atoms** (material), the properties of index (density), absorption, etc., can vary. Even with no reactive gas or ions supplied, the depositing material is competing for positions on the substrate with whatever the residual gas may be in the chamber (usually water vapor).

It may be possible that strong variations in the initial deposition rate when the shutter opens (as seen in **Figure 1**) can also effect the adhesion of a film to the substrate from one deposition to another. These various effects can cause serious run-to-run differences which may be the causes of the failure of some coating runs to meet specifications.

The Solution

Fortunately, quartz crystal monitors (QCM) are now installed on most optical coating systems, and they can control the deposition rates of most materials when used properly. The QCM normally generates a signal which is proportional to the amount of mass deposited on the sensor head. This information can be processed within the controller with the proper calibration factors to provide a rapid estimate of the deposition rate and thickness of the layer being deposited. This information can then be used to control the power to the deposition source for rate control and to open and close the shutter for that material. One should however be cautioned by the statement of D. Radgowski et al.[2] which said that "Benchmark studies of 30+ production systems revealed that none of the systems are reaching the full potential of their QCM's rate control capabilities".

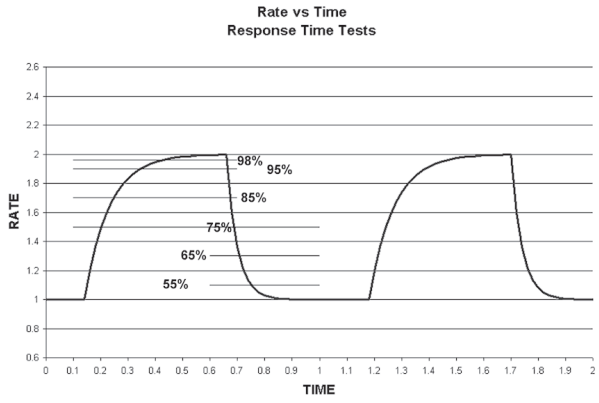
There are three areas of QCM setup and operation that are important to this discussion. First is to get the proper constants set into the controller for each material, and the Second is to get the power to the vaporization source such that the rate is very close to what is desired **just as the shutter opens**. Third is to find what control delay is needed for each material. There are various factors for each material to be entered from appropriate tables, but those which are addressed here are the "PID" control values. PID generally stands for *proportional(Push, gain), integral(Inertia, momentum),* and *derivative(Drag, damping)*. One could imagine an example of pushing a stalled auto on a smooth and level paved road onto a soft sandy shoulder (all on the same level). As a push or force is applied, the sum of that force over time (integral) accelerates the mass or inertia of the auto up to some speed on the smooth pavement. As the sandy shoulder is reached, the friction or drag in the sand increases significantly over the drag of the road, and the auto slows or decelerates in proportion to the speed it was moving (derivative of distance versus time). The **proportional** aspect of the controller is that its correction signal or force applied is proportional to the error between the desired rate or set point (SP) and the rate at any given moment.

Setting Ramp and Soak Times

To get some data on how the source behaves, manually adjust

the power to the source with the shutter open until a small rate is detected. Control the source to keep a rate of 1 Å/second for about one minute, and record that power. Increase the power for a steady rate of 2 Å/second and record that power. Do this also by factors of two (4, 8, 16, etc.) until the maximum expected rate is covered.

It is next desired to get some data on the response time of the material system. It is recommended that the following procedure be used on tuning **resistance sources** initially, since e-gun depositions add new factors which would need to be handled. With the shutter open, set the power for 1/2 the maximum expected power for the deposition rate and wait until the rate is steady. Increase the power quickly to the power for the maximum expected rate and record the time it takes to reach something like 75% the desired rate, 85%, 95%, and 98%, as seen in **Figure 2**. Then turn the power down quickly to the value for 1/2 the rate and record the time it takes to reach a rate of 75%, 65%, 55%, and steady at about 50% of the maximum deposition rate.



(2)

Figure 2. Testing the non-controlled response time for changes between 50% and full rate.

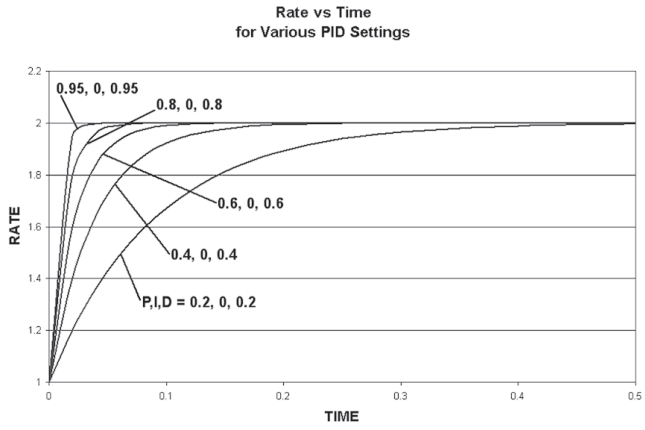
A possible starting setup for ramp and soak times would be as follows, but adjustments may be done as needed. Set the RAMP 1 Time to the time found above to reach about 95% rate, and set the RAMP 1 Power to the level found which just starts to evaporate material at a **very low** rate. Set the SOAK 1 time to about the same as the RAMP 1 Time. Set the RAMP 2 Power to the power determined above for the **desired rate**, and set the SOAK 2 Power to the same as the RAMP 2 Power. Then set the RAMP 2 Time the same as the RAMP 1 Time. Set the SOAK 2 Time and SOAK 2 Power to the same as RAMP 2 Time and Power.

The HOLD Power is for use when another material is being evaporated and the one considered here is waiting to be used for another layer. This should be set to the same level as SOAK 1 power. This then should evaporate very little material while on hold under a shutter, but it will be ready to ramp up as fast as practical to deposition rate when needed by the application of RAMP 2 and SOAK 2.

PID Settings

Figure 3 illustrates a set point (SP) of 2 units of rate (perhaps in Å/second) and an initial rate of 1 unit when the shutter opens at time equal zero. The QCM controller then needs to Push (P) the power to the source until the rate is 2. The figure shows that a P-value of 0.2 will cause the rate to come close to 2.0 in about 0.5 time units (perhaps minutes). As the P is increased to 0.4, 0.6, 0.8, and 0.95, the rate more and more quickly reaches the SP. This would be the same as getting the automobile in the example above to the rolling speed which is wanted more quickly by pushing harder on the car to accelerate the process.

The curves in **Figure 3** are all for D-values of the "PID" which provide "critical damping" for a given P-value. This means that the rate gets to the SP as quickly as possible for that amount of gain (P) and stays there without going beyond the SP or overshooting. One would like to have as high a gain (P) as practical to correct any rate error as quickly as possible without a significant overshoot. Too much damping would delay the rate from reaching the SP.



(3)

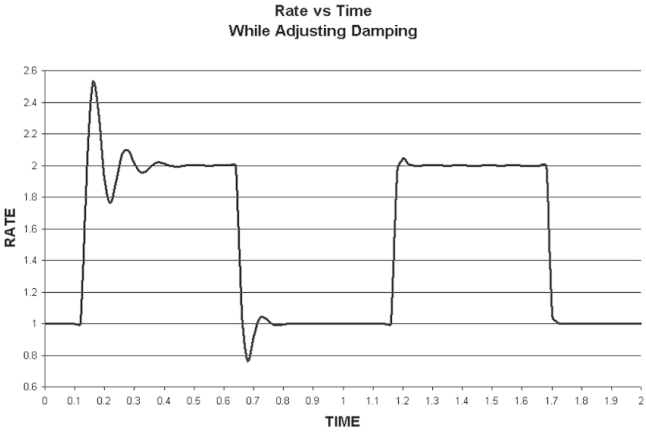
Figure 3. Various PID settings with critical damping showing relative response times.

The I-value may not need to be used, depending on the system. In that case, the tuning procedure or selection of the P- and D-values can be fairly straightforward.

Recommended Setup Procedure

Set up the QCM as needed and manually control the power to start depositing at 1/2 the desired rate until steady. Make the QCM automatically control at that steady rate for perhaps 1/2 minute. Change the SP to the **full** desired rate and record pattern of rate versus time as in **Figure 2**.

If the pattern shows oscillations as at the start of **Figure 4**, increase the D-value by a factor of two. Then cut the rate SP to 1/2 of the desired SP; this will create another transition/change in the other direction which can be observed for its oscillation as in **Figure 4**. If there are still oscillations, increase the D-values again by a factor of two, and set the SP back to the desired (higher) rate. Continue this procedure until the transition is nearly critically damped, as at the right end of **Figure 4**.



(4)

Figure 4. Adjusting damping to higher values to reduce overshoot and "ringing."

If the initial pattern shows **no** oscillations, increase the P-value by a factor of two and cut the rate SP to 1/2. This will create another transition/change in the other direction which can be observed for oscillation. If there are still no oscillations, increase the P-value again

continued on page XX

Reproducibility in Optical Thin Film Processing

Part 2, Deposition Rate Control

continued from page XX

by a factor of two, and set the SP back to the desired rate. Continue this procedure until the transition has shown some oscillation but is nearly critically damped.

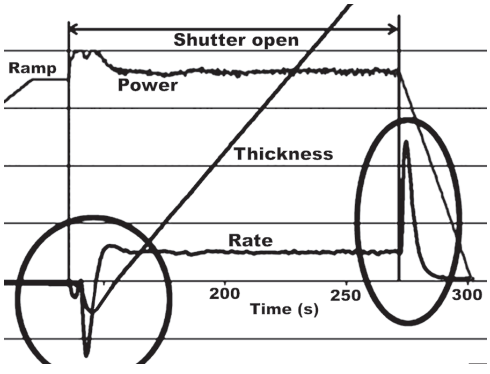
The above procedure should provide good constant rate for an otherwise stable source. However, it may be possible to obtain an even faster response. This should be explored by increasing to P-value and D-value in steps to keep near critically damped at higher and higher values of P. These would be expected to reach some practical limit which would not be beneficial to exceed. At that point, it is advisable to back down a small amount for a margin and use that as the operating PID values for that particular material source.

These results may also have some sensitivity to the size of the load of material in the source. That might require some further tests with a near-full and near-empty source, and program the subsequent layers starting with less material in the source with values adjusted to work well for those conditions.

Soak Level and Control Delay

When the shutter opens, it is intended to have the source soaking at a power such that the deposition rate will be **exactly** that desired. Set the crystal controller to display the rate but to NOT control the rate. Set the SOAK 2 power to give the SP rate. When the warm-up cycle is complete, open the shutter and record the behavior with the power constant to determine when the steady rate is reached after the shutter opens. The radiant energy falling on the crystal can cause a temperature rise in the crystal which gives a false indication (higher or lower) of the rate until the transient passes as seen in **Figure 5** for a short time. **Figure 5** shows real data with this transient effect for a standard "AT" cut quartz crystal. This data is from Colnatec[3] who have a solution to this problem which will be discussed in Part 3. If the final rate is too high or too low, adjust the SOAK 2 power until the rate is correct. Record the time of the transient behavior with each such test.

The above tests will determine the value needed for the Control Delay in the Crystal controller. Enter this value in the controller settings for this material. The Control Delay prevents the controller from acting until it has information which is expected to be correct. Otherwise, the transient is likely to further aggravate and defeat the purpose of having a perfectly constant rate while the shutter is open.



(5)

Figure 5. Real data with the transient effect for a standard "AT" cut quartz crystal..

Conclusions

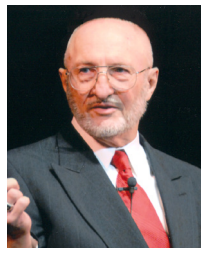
Steady/Constant deposition rates during deposition of thin films are important to the reproducibility of the index values and other physical properties, but this goal may not be often achieved in the industry. The behavior of the control functions of a quartz crystal monitor have been described and procedures have been suggested to optimize the related (17) control values.

References

1. R. R. Willey, Practical Production of Optical Thin Films, Sec. 2.5, Willey Optical, Consultants, Charlevoix, MI, 2008.
2. D. Radgowski, G. Reimann, M. Gevelber, "Critical measurement and control issues in selecting a quartz crystal monitor," 51st Annual Technical Conference Proceedings of the Society of Vacuum Coaters, pp. 31-37, 2008.
3. http://colnatec.com/

About the Author

Ronald R. Willey



Ron Willey graduated from the MIT in optical instrumentation, has an M.S. from Florida Institute of Technology, and over 40 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.

For further information, contact Ron Willey, Willey Optical Consultants, Michigan at ron@willeyoptical.com.