# Reproducibility in Optical Thin Film Processing
## Part 3, Temperature Control

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*Contributed Technical Article: Part 3 of 3*

Variations in the temperature during an optical thin film coating process can cause a significant lack of reproducibility in the product of the coating run. In Parts 1 and 2 of this series, the effects of variations in vacuum pressure and deposition rate were discussed. Temperature is perhaps the third important cause of variations, and the subject of this article.

### Introduction

It has been discovered over the decades that many if not most optical coating materials are more dense and durable if they are deposited at elevated temperatures (ZnS, ZnSe, Ge, and possibly others are exceptions to this finding). $MgF_2$, for example, has much better durability and much less shift with change in humidity (because of the higher packing density of the deposit) if deposited at 300ºC rather than at room temperature. Similar things can be said for many materials including $TiO_2$ and $HfO_2$. Germanium, on the other hand, has some adverse effects if deposited above 200˚C as discussed in the production book [1]. In this current article, we will discuss how heat is usually applied to (or removed from) a process, how it is sensed and controlled, and how variations can occur in temperature which cause problems with the deposited films.

### Heating

Some of the heating methods that are most commonly used today will be discussed. However, the expanded use of ion assisted deposition (IAD) and other energetic processes might eventually eliminate the use of added heat in most optical coating processes. It is currently unusual for an optical coating chamber to be operated at much over 300˚C. Special considerations of insulators such as Teflon and bearing behaviors are needed for temperatures above this range.

Conduction and convection are not effective in a vacuum; therefore radiant heating is the normal way to heat substrates. Probably the most common heat sources used now are "quartz halide" heat lamps with fused silica cover glasses to keep the coatings from the lamp elements. The filaments may operate at over 2000 K. The quartz envelopes transmit well from the visible to about 5 µm, beyond that the envelopes are opaque and heat is only radiated from the hot envelope. With infrared transmitting substrates such as Ge, ZnSe, ZnS, etc., which transmit in the 5 micron region and beyond, most of the heating probably comes from the visible spectrum absorption. Glass substrates absorb from about 2.5 µm to longer wavelengths, so that most of the heating there may be due to the 2.5 to 5 µm region which would be near the peak of 700 to 2000 K blackbody sources. The protective cover windows over the lamps become coated and need to be cleaned and sometimes replaced due to breakage. It is often practical to clean these flat window surfaces by scraping the coating off with a razor blade. Bead blasting has also been used successfully. There may be some change in the angular distribution of the heat due to the diffuse scattering in the visible spectrum of a bead blasted surface, but we have not noticed any obvious effects.

Nichrome wire heaters have been used in various forms. We have worked with several Leybold Heraeus A11000 chambers with heaters of nichrome wire tightly coiled on a ceramic core. They operate at a much lower temperature than a quartz lamp, with a color temperature less than an orange glow. These have been surprisingly durable. They can be bead blasted for cleaning and rewound with fresh nichrome wire when necessary. Such heaters are generally positioned like quartz lamps near the base plate of a box coater radiating the substrates from below.

Another approach that we have observed, from Balzers and Leybold, is an array of rod and coil heaters of the general type used in domestic ovens, electric stove-top "burners," and electric hot water heaters. The array is placed above the calotte in a box coater as seen in **Figure 1**, and it radiates downward on the back of the substrates in the calotte. The calotte would need to be open in the back to expose the substrates to the radiation from the heaters. It would seem that the back side heating is desirable in avoiding coating on the heaters, but that might obviate its use with a planetary system.

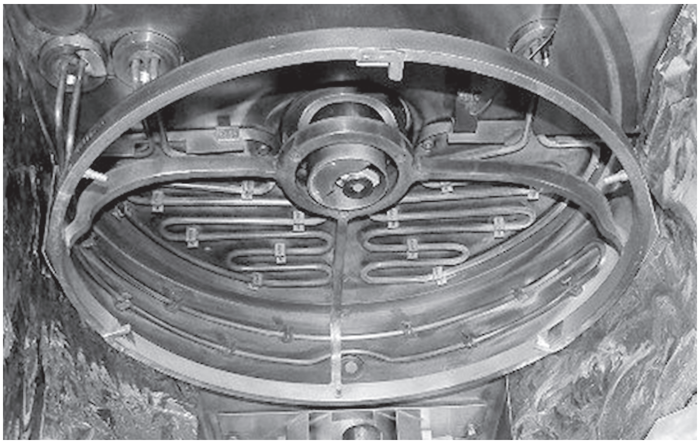

*Figure 1. Chamber heaters similar to domestic oven heat rods or coils.*

**Figure 2** shows a simple application of nichrome wire coils used as heaters in a glass bell jar coater. The goal in the chamber shown in **Figure 2** was to coat $MgF_2$ on lenses heated to 300ºC. The heaters were nichrome wire coils above the substrates. In one batch of parts, it was a surprise to find that the $MgF_2$ coating appeared to have absorption. It was then discovered that too much power had been applied to the nichrome wire and some of it had evaporated unto the back-sides of the lenses.

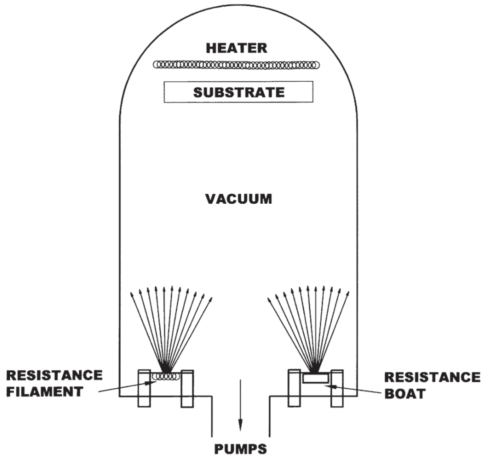


*Figure 2. Nichrome coils used for heating at the top of a glass bell jar.*

A one-meter box coater might require about 10KW of heater power to reach 300°C in a reasonable time. The condition of the chamber walls will have a major effect on the time for heating and the ultimate temperature. An unfoiled stainless steel chamber wall shield that has been bead blasted for cleaning might absorb about 50% of the energy falling on it. If the shields are water-cooled, this absorbed heat will be removed. A chamber wall or shield that has been freshly covered with aluminum foil might reflect about 90% of the heat and only absorb 10%. The foil and/or shield may not make good thermal contact with the wall and cooling, and therefore not even all of the 10% is lost heat to the cooled wall or shields. As the foil becomes coated, its average reflectance will probably drop and the heat balance will change. This may be the source of some process changes noticed from a "dirty" chamber to one that has just been cleaned and foiled.

Some groups recommend changing about 1/3 of the foil (or cleaning 1/3 of the shields) on a rotating schedule in production. In this way, the chamber has a more nearly constant state of "clean/dirty" with each production run. We think that this is a good operational practice to minimize the effects of changing wall reflectance.

## Cooling

The only common source of cooling for box coaters is water cooling, other than cryopumps and Meissner traps with liquid nitrogen or "Polycolds". The chamber walls are usually water cooled for hot processes such as 300°C so that the work area is bearable, etc. The E-guns and high current feedthroughs for the resistance sources need water cooling. Many ion/plasma sources for IAD and sputtering need water cooling.

Historically, quartz crystal monitor (QCM) heads must be kept cool, but more importantly be kept at a constant temperature. Some coating facilities have used a separate small water circuit at 100°F to feed the crystals water at a constant temperature, as illustrated in **Figure 3**. It is easy to thermostat the small reservoir kept at above room temperature and add a little heat to the water as needed to maintain the feed temperature.

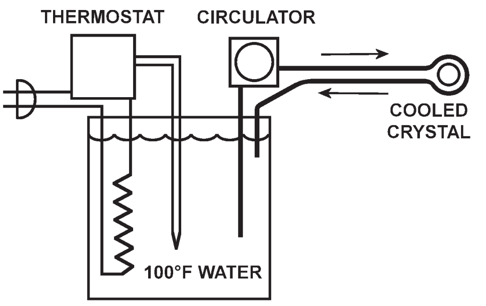


*Figure 3. System for stabilizing the temperature of the cooling water for a crystal monitor.*

The source of cooling water can be critical. If crystal cooling lines which typically have a small bore (1/8" to 3/16") become clogged, the crystal readings become poor to useless and batches of parts can be lost. We were in a facility once where leaves were seen passing through the clear plastic piping from a cooling tower system. This could have caused some very expensive problems if the leaves had plugged the cooling lines to an e-beam source, crystal, and other things. Avoiding obstructions in cooling lines is very important. The production manager quickly remedied this situation!

Scott Grimshaw [2] of Colnatec LLC has come up with innovations

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in crystal technology which look like they may revolutionize QCM technology and move it ahead by a quantum leap. With respect to heat, he has found that most water cooling of QCMs is very inadequate with surprising temperature variations (see his web site [2]). He has gone to thermocouple (TC) sensing at the QCM head and a heater system to control the head temperature precisely. Some quartz heads are also operated at higher temperatures up to 250˚C to minimize stress cracking of the films and thereby QCM failures. Using other than quartz crystals, they have even gone to 900˚C. High pressure air is also used instead of water cooling. Grimshaw has also invented an "RC" crystal cut which virtually eliminates the spikes in crystal signals when there is a change in the radiant heat falling on the crystal. This can be of particular advantage with thinner films where the former transients due to radiation changes would give great errors at small thicknesses.

Cooling water expense can be a surprisingly large problem. At one time, we knew of two 760 mm chambers running three shifts using city water for cooling and discharging it down the drain. This is not even legal in some areas now, but the cost turned out to be about $2000 per month in the 1980s when dollars were more valuable! This was about $4 per hour. In East Coast Central Florida, the best source of cooling turned out to be deep wells (~100 feet) to water at about 24˚C (76˚F). Wisnewski [3] described how such a cooling system was designed and constructed with a heat exchanger and a closed loop treated water system circulating to four chambers and their supporting PolyCold cryobaffles. This system provided cooling to the chambers at or below 26˚C (79˚F) year round. Lore is propagated by some equipment manufacturers that coolant at lower temperatures than this are required for diffusion pumps (DP). We have operated box coaters with DPs from four different builders at these temperatures without noticeable ill effects. Missimer [4], Gordon [5], and Zahniser [6] reviewed cooling towers which are the most common approach to obtaining water cooling in most parts of the USA. Any of these systems should bring the operational costs of cooling down to a small fraction of the electrical costs.

One practice with the chamber cooling water is to turn it off at the end of a process when the chamber has cooled to some temperature appropriate for venting but not as low as the ambient temperature. When the vented chamber is opened, it is still warm enough to minimize the condensation of atmospheric water vapor inside the open chamber. This practice is oriented toward production where the chamber would not cool too much before being reloaded and pumped again. Some facilities have a separate hot water circuit and valving to heat the chamber at venting. This might be most beneficial when coating runs such as developmental runs are not in a rapid sequence. We have not found this hot water necessary even in the high humidity environment of Florida, as long as rapid run sequencing was done. In cases where the cooling water is well below room temperature, leaving the cooling water on with the chamber open can cause visible water condensation inside the chamber. Keeping the chamber closed as much as practical seems to be a good practice.

### Temperature Sensors and Control

The almost universal temperature sensor used in a box coater is a thermocouple (TC). The electronics used for converting its voltage to temperature and controlling the power to the chamber heaters is generally available as "off-the-shelf" equipment from suppliers such as Omega Engineering of Stamford, Connecticut, etc. We will not belabor the details of the sensor and control, but touch on their application.

The sensor is usually placed close to the substrates since it is the substrate temperature that is most important to control. The TC sensor leads might come through a central hole on the axis of a calotte or planetary drive and have some height adjustability. They might come up from the base plate or through a side port of a box coater to the vicinity of the substrates. The reading at the junction point of a TC is only an approximation of the substrate temperature at best, since it is not imbedded in the surface of the substrate. It is a relative reading, but some effort is needed to have the readings as reproducible as practical because some deposition processes are quite sensitive to temperature.

We have had a problem with an infrared coating using many germanium and fluoride layers. The process was running well until the chamber was cleaned. After cleaning the chamber, the next run of parts exhibited severe absorption. A week's effort of tests and checks was wasted before it was discovered that the TC had been mispositioned by about 10 cm from its normal distance to the center of the chamber and the parts in a calotte. We then learned [1] that the Ge would show absorption losses if it was deposited at above about "190˚C" as indicated on the TC (in its normal position). This mispositioning caused more heat to be applied than normal to get the reading on the TC up to its usual setting, and the product therefore showed severe absorption. This experience convinced us of the need to relocate the TC sensor reproducibly.

All materials seem to have some temperature sensitivity to greater or lesser degrees. These variations are most notably index of refraction, density, hardness, absorption, etc. We can assume that the TC is being heated mostly by radiation and not conduction or convection if it stands in the open in the chamber as in the left-hand image in **Figure 4**. Its heating is coming from the radiation of the heaters plus reflections from the walls, fixturing, and parts in the chamber. Its absorption of the radiation will depend on the absorption versus wavelength of its surface and the coating on it; and it will also depend on the energy versus wavelength from the heaters. Some coaters have used the practice, as seen in the middle of the figure, of placing a glass block over the TC. This is so that the temperature reading in the block will behave more like one of the lenses being coated. The best would be to use one of the actual lenses, but the next best might be a block of the same glass material and the same mass as the lenses that were being coated. We have seen a coating operation that moved partly in this direction by putting small glass test-tubes (available from laboratory supply houses) over the TC as seen in the right-hand image of **Figure 4**.

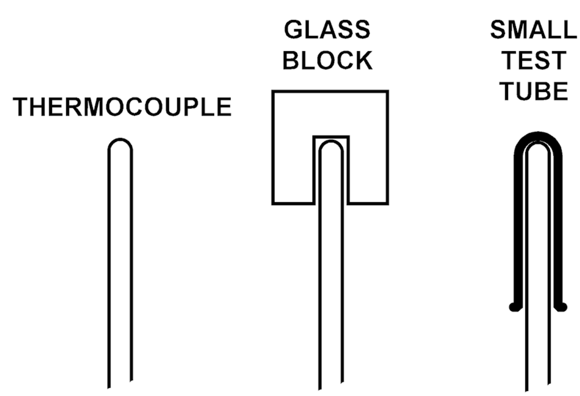


*Figure 4. Bare thermocouple and two with thermal masses.*

We have seen a pyrometer used to sense the temperature of the substrates through an infrared window in a chamber, but even this is not an absolute measure of the temperature. Temperature sensitive paints and other materials can be used to calibrate the substrate-to-thermocouple tooling factor. Small high limit recording mechanical dial "clock" thermometers are sometimes placed in the fixturing for the parts to do this same type of calibration. Within a single process chamber, we believe that the critical element is reproducibility of temperature, it does not matter as much as to what the absolute value is. The process temperature would have been adjusted for the results needed from that chamber. However, when a process is to be transferred to another chamber, the absolute values would be more helpful. Because obtaining the absolute values is not often practical, it is probably necessary to test for optimum/acceptable upper and lower temperature limits of the process in the new chamber.

Temperature controllers are now typically of the "PID" type. (PID concepts were discussed in Part 2.) Here the power applied to the heaters can be proportional (P) to the error between the desired set-point and the actual temperature. The gain and damping or phase lag of the control can be set so that power to the heaters does not oscillate on the one hand or act too sluggishly on the other. Usually the desired settings give a slight overshoot of the temperature which settles to the set value without oscillation. This is "critically damped" and reaches the set-point value to within some tolerance in the minimum time.

When E-guns, resistance sources, and some ion/plasma sources are operated at high power levels, most of the power appears in the chamber as heat and less power is needed from the heaters to maintain a given process temperature. We have had processes at over 200°C that became overheated for the depositions by using 6 to10 KW of E-gun power, and the process had to wait for the chamber to cool down to proceed at the set-point temperature. Controllers may allow an upper and lower limit to be set to the permissible temperature band and inhibit the process until those limits are satisfied. Excess heating due to sources is a common problem when plastics are to be coated. Heat removal may be desirable. In web or roll coaters, there is often a water-cooled drum behind the surface being coated to keep the plastic below a critical temperature. A Meissner trap should also help keep a chamber cooler, even though its principal function is additional water vapor pumping speed.

## Conclustion

Since the properties of the deposited films such as index of refraction, density, hardness, absorption, etc., are usually strong functions of temperature, it is important to have a stable temperature environment during the deposition process. Many of the possible pitfalls to temperature stability and reproducibility in a process have been discussed.

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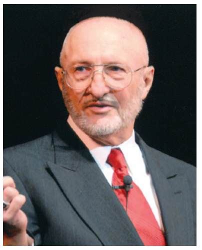


## About the Author

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*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.*

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