IMPROVING THE PRODUCTION EFFICIENCY AND YIELD OF OPTICAL COATINGS

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Materion Coating Materials News

Introduction

There are a number of challenges associated with achieving high yield in the coating of many small or large substrates. These include making the most efficient use of both materials and the substrate area, which often has a non-flat surface, as well as considering the amount of time spent in coating production. In addition to these challenges, there are three major issues related to the volume production of thin film optical coatings. Following, we discuss the problems in detail and describe current technological solutions.

The key issues that limit efficient and consistent production of optical coatings are:

- Source material preparation time
- Material usage and consistency over time
- Efficient coating thickness distribution

Coating System Considerations

Complete, self-sufficient coating systems that provide programmable controlled deposition of the individual layer thickness for complex coating designs are manufactured by several firms. However, not everyone adequately addresses all three of the issues previously mentioned. For example, the traditional method for preparing high-evaporation temperature oxide compounds such as Titania, Tantala, Hafnia, Zirconia, and Alumina is to load an evaporation vessel (e-gun crucible or resistance-heated container) with pieces or tablets of the source material. Then the evaporation technician proceeds to exercise repeated melting/venting cycles to attempt to prepare a dense mass of starting material. After the first deposition cycle, fresh material is added to the used charge and another melting/venting cycle is required before production can resume. This procedure is time and resource-consuming, and does not produce a consistently homogeneous starting charge.

Source Material and Preparation Time

A solution to this source preparation and conditioning problem has been available for a few years in the form of **pre-melted forms** made by Materion (CMN: Vol 15 no. 1 (2005); V14 no. 2 (2004). The forms are created by thoroughly melting large quantities of the material at closely controlled temperatures and pressures. Table 1 lists evaporation-ready charges of the most frequently used and preparation-intensive materials that succeed in reducing the labor and vacuum resources required for coating production. Process-ready cone forms of specific oxide compounds that are most frequently used between UV and IR, including high laser damage threshold (LDT) applications, are also described in the following Table 1.

Table 1. Suggested Deposition Parameters of Pre-Melted Forms of Starting Materials

Material	N (550nm)	Sub Temp (°C)	Rate (Å/S)	O2 Pressure (e-05 Torr)	Useable Transm.(nm)	Notes: Liner?
Al_2O_3	1.65	>200	~5	~1	220 - 10000	Sweep, no
HfO_2	2.06	150-250	2-4	~5	230 - 10000	Sweep, no
Ta_2O_5	2.2	200-250	2-4	~8	350 - 7000	Graphite
TiO_2	2.3	<100-200	2-5	~5	450 - 10000	Graphite
ZrO_2	2.1	>200	2-4	~3	250 - 10000	Sublimes

While Zirconia and Hafnia compounds do not melt to a great depth, a uniform evaporation surface created by beam-sweeping provides a constant rate. The other materials evaporate from a more deeply melted surface. When high-energy assist techniques are added, such as IAD or PIAD (plasma ion assisted deposition), pressure and temperature parameters will be appropriately adjusted.

Advantages of Pre-Melted Forms: Consistency

Experience has taught that the preparation and conditioning of the starting material, in particular for refractory materials, has a significant influence on the optical, mechanical, LDT, and environmental properties of thin film layers. These high-evaporation temperature materials require an e-beam to vaporize, and if the material is not melted to a homogeneous consistency, localized erosion will occur and evaporation will proceed from a small area of low-density material, often a cavity. Charges prepared by repeated melt / vent cycles suffer from density stratification and inhomogeneity, issues that lead to inconsistent rates and film composition.

This low packing density geometry is contrary to the more desired uniformly-melted extended evaporation surface that provides a consistent vapor stream flux density. Hole drilling or uneven erosion is reduced with a swept beam. If the evaporating surface changes in breadth or depth, the vapor stream distribution pattern will change, and this produces a non-uniform thickness distribution at the work substrates.

Some of the refractory oxides can grow films with multiple crystalline phases, polycrystalline sizes, and inhomogeneous packing densities. To some degree, these departures from desired amorphous and compact morphology are influenced by the starting material's condition. As new material is added and melted down after a production run, the new upper material layer might not have the same condition as the previous material. The possibility of depositing inconsistent layer-to-layer properties is therefore large. For critical coating designs, such as bandpass and edge filters, as well as wide-band AR coatings, small deviations in refractive index can mean the difference between a failed or a successful coating run. The pre-melted forms act to reduce potentially inconsistent influences on film optical quality and repeatiblity. By sweeping the uniform surface, the creation of high-temperature crystalline morphology typical of a focused beam is avoided. Constant composition and deposition rates are thus achieved.

Reduction of Particulate Emanation

Another advantage of pre-melted forms related to yield and process time is the reduction of particulate emanation (spatting and spatter) that is typically present when the source material is in the form of small individual pieces or tablets. When a beam strikes these source material forms and encounters a void, a trapped pocket of moisture, or a pocket of trapped volatile cleaning agents, particulates can be emitted. Coating technicians attempt to avoid these emission problems by cyclically filling a crucible and melting the charge down, then repeating this as many times as required to build a dense evaporation cone. This process is eliminated with the use of pre-formed cones.

A problem often encountered with dielectric starting materials such as oxides is local charge build-up on their surface. Charging can deflect the electron beam, or arcing, causing inconsistent evaporation rates. The pre-melted forms prepared as sub-oxide compositions are slightly conducting (as well as being a uniform surface) thereby preventing electrical charging. Alternatively, for plasma-assisted processes, the cones can be created in a more fully oxidized state. For example, silica and alumina can be deposited in a low oxygen background pressure, whereas the high-index materials require a high oxygen background pressure to produce low absorption films.

Depending on the volume of material evaporated from a pre-melted charge, and the frequency of refilling and melting the added material, there is a use-limited lifetime beyond which the added material does not restore the consistent rate and composition of the new charge. This can also introduce the possibility of spitting for the reasons described above. The lifetime of reuse is different for each material, dependent on the volume consumed and application, and is learned through experience.

Coating Thickness Efficiency

A further problem is the introduction of thickness errors on the monitoring sensor, which is typically located away from the work pieces, as the vapor plume changes shape. To illustrate this phenomenon, we modeled the thickness distribution over a substrate area from a centered source whose distribution pattern is changed according to $(\cos \Theta)^m$. Computations for Figure 1 were made using Dr. Bill Southwell's Uniformity-Pro software program (Laser Focus World, July 2012). A value of $m = 1.5$ represents a typical e-beam plume. The fall-off in thickness increases as the beam plume diameter collapses from hole drilling. This model demonstrates the importance of establishing and maintaining a constant evaporation source surface such as provided by a pre-melted form using a swept beam.

See following Figure 1 related to distribution uniformity.

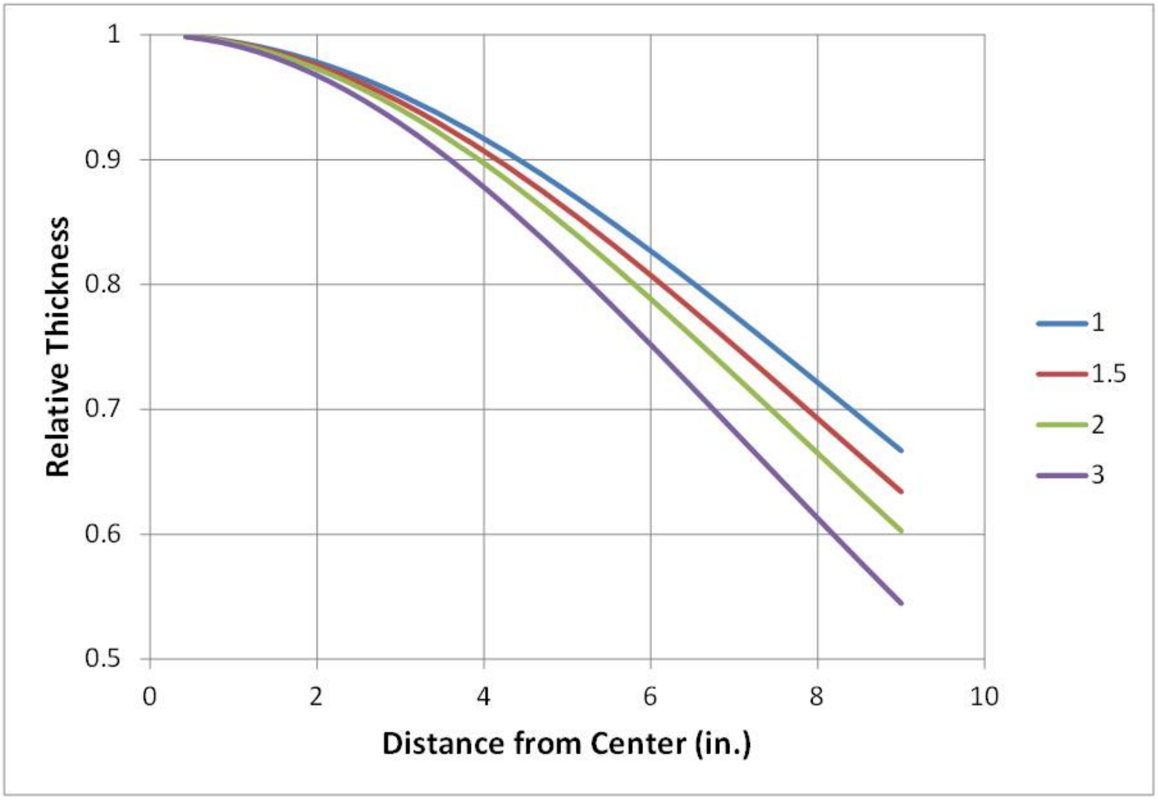

Figure 1. Distribution uniformity over a 0.5 m diameter flat surface created by a centered source at 0.5 m distance. Plotted is distribution function, $(\cos \Theta)^m$, where m values are 1.0, 1.5, 2.0, and 3.0.

Conclusion

Operationally, the benefits of material pre-forms is that they save technician time, improve yield, produce more consistent high quality films and reduce maintenance. Materion produces pre-melted products in virtually any oxide or ceramic evaporation material in early any size or shape, achieving the highest quality and purity in the industry. Click for more [Materion pre-melted evaporant](#) information...

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