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The Coating Process: Requirement to Delivery


Introducing New Pre-Melted Oxide Evaporants from CERAC. See insert for details!

In this quarter's CMN, we analyze the general process of producing some specific optical thin-film coatings beginning with the requirements and proceeding to deposition and delivery. The deposition procedure was outlined in a previous publication prepared by this author [1]. The steps involved in proceeding from the required coating performance to delivery of the performing product require the participation of engineering and deposition technology personnel as well as quality inspectors, and are outlined below.

- Evaluate the requirements
- Search for a solution
- Develop a process
- Deposit production run
- Evaluate the product
- Deliver product and test data.

These steps are expanded to greater detail.

Evaluate specification for:

1. Required optical performance:
   a. Spectral reflectance or transmittance,
   b. Incidence angle range,
   c. Substrate material and preparation.
2. Required mechanical and environmental durability:
   a. Temperature ranges, adhesion,
   b. Solubility and cleanibility,
   c. Exposure to moisture, salt spray, abrasive wear, high-energy laser or particulate radiation.

3. Time out:
   Are any requirements not within current experience or capabilities (or require amendments to the laws of optics!)? Are we equipped to properly evaluate and report verifiable data?
4. Decide to proceed or discuss with requester.

Solution search:

1. Resort to a previous successful procedure or recipe that had similar requirements and either modify as required, or,
2. Conceive of a new design / process.
   a. Selection of materials.
   b. Generation of preliminary design by coating design engineer.
   c. Tolerance study evaluating capabilities to control thickness and index variations to the sensitivity of the design.
   d. Generate a layer-by-layer run procedure.

Develop process:

1. Select materials
   a. Forms (tablets, sized pieces, target)
   b. Preparation: chemical and physical states (reduced, pre-melted)
   c. Source and container (e-beam, resistance-heating, sputtering, IAD)
   d. Calibrate materials and thickness-monitoring equipment
   e. Check n & k values for the layer materials
   f. Verify thicknesses and uniformity.
2. Test run of the design
   a. Compare the optical performance with requirements.
   b. Evaluate mechanical durability per MIL or ISO specs.
   c. Document preliminary procedure, including adjustments and iterations.
   d. Design tooling for production lot.

Deposit the production run:

a. Evaluate, document measurements.
b. Report performance compared with requirements.
c. Prepare for delivery.

continued on page 2

General Engineering Notes

It is always helpful if the designer is provided with some idea of the operation of the system and the optical environment in which the coating will be expected to perform. Often reflected or transmitted energy from coatings propagates in directions and to places not initially anticipated. It must be kept in mind that, with interference coatings, what is not transmitted is reflected and vice-versa. Thus, for example a color filter transmits the desired passband but reflects all other wavelengths. The reflected energy path must be controlled to prevent the appearance of stray light in the form of ghost images or contrast-diluting energy.

Knowledge of material behavior is essential for high yield production. Optical coaters have developed in-house processes to deposit a select number of materials. The job of the material supplier is to reduce the effort involved in evaporation or sputtering a material with consistent film properties [2]. The physical properties of some materials, notably the metal oxide compounds, are very sensitive to the oxidation state of the starting material. Similarly, the evaporation properties: rate, deposited packing density, absence of particulate inclusions, etc. are sensitive to the pre-conditioning of the starting material. Materials that melt do not generate particulate spatter; those that sublime from the solid state are more likely to outgas and spatter microparticles unless pre-melted or properly fused. Properly prepared starting materials reduce the in-chamber time required to bring the material to its optimum evaporation condition.

Coating chamber design, especially parameter monitoring equipment, plays an essential role in coating production. For evaporation processes, three important parameters are chamber pressure and gas composition, substrate temperature control, and uniformity of condensing vapor distribution. For sputter deposition, substrate temperature in not as important as ion-plasma energy. During reactive deposition by either process, the partial pressure of the reactive gas is not uniform throughout the chamber because its supply and consumption (source and sink) are localized. The pressure gauge or RGA probe is rarely located near the substrates, therefore the reactive gas pressure is not accurately measured. Substrate temperature is difficult, if not impossible, to accurately measure except when the substrate is stationary. Because the evaporation distribution is not geometrically (and perhaps compositionally) uniform, the properties of the coating can vary spatially over the substrate holder. Applications such as metal mirror production are not affected, but uniformity is critical in the production of demanding broad-band or multiple-band ARs, narrow bandpass filters, polarizing coatings, and hot / cold mirrors.

Substrate surface cleaning is important for achieving a good adhesive bond with the coating, and can also affect coating morphology and microstructure by influencing nucleation and growth patterns. We discussed cleaning procedures for various substrates including polymer in a previous issue [3]. Chemo-mechanical cleaning might be done by hand or automatically in an ultrasonic bath line. The nature of the substrate dictates the chemistry and mechanical forces applied. Soft and soluble / absorbtive substrates such as polymers are cleaned without mechanical contact and must be dehydrated before being coating. Some high-index glasses must be treated with extra care because they are susceptible to water reaction that will deteriorate their polished surface or change the composition of it. In batch production, enough time might elapse between when the first part was cleaned and loaded on to tooling and subsequent parts that recontamination might begin. In-chamber final cleaning often employs energetic ions generated in a plasma discharge ('glow discharge' in oxygen or nitrogen, depending on the substrate) or argon ion bombardment using an ion (gun) source. The layer contaminating the surface is either eroded away or reactively modified as, for example oxidation of organic molecules. Sometimes, particularly in the case of metals, reactive or non-reactive, on substrates differing between polymers and glasses, it is necessary after extensive cleaning to create a surface layer that promotes adhesion and dense micro-structural growth. Thin precursor or nucleation layers are deposited that might be a reactive metal such as chrome or a layer of the same composition as the substrate. The first approach is used on polymer surfaces; the second on inert IR (non-oxide) materials. These layers are so thin as to be optically absent.

Examples of Coating Processes

Broadband AR for Glass

To illustrate the process, we choose as the first example a requirement for a typical broadband AR coating on glass. Such coatings are in demand for practically every glass and polymer surface in applications from eyewear to display panels to auto and aircraft instrument panels. The typical requirements are:

- R <0.5% averaged over wavelengths 420 to 680 nm.
- Incidence: near perpendicular (<15°)
- Durability (varies widely with application): adhesion, cleaning / solubility, moderate abrasion, temperature cycling, etc.

AR requirements such as these are common, as mentioned, and CMN past issues have discussed these coatings [4]. AR coatings that exhibit ~0.2% R at all wavelengths between 450 nm and 650 nm are in production using in-line sputtering [5]. The thin-film designs and deposition processes are adapted to substrates of different indices and different compositions. For example, polymer substrates require a different surface preparation than glasses and are heat-limited. Many coating companies use e-beam evaporation and commonly TiO_2 and SiO_2 as the material combination in a 4-layer design. Other combinations evaporable by e-beam are: MgF_2 / TiO_2, SiO_2 / Ta_2O_5, etc. Oxides are conveniently sputtered, and other high-n materials such as Nb_2O_5 can be added to the list. Some of the layers are too thin to permit optical monitoring, so crystal thickness monitoring is employed. Achieving minimum average R without excessively high bumps appearing requires thickness and index precisions (optical path) of <10% or approaching ± 10 A for the thin, more critical layers.

continued on page 3

The broad-band AR coating for glass in the visible region is a commonly available coating. Requirements added to the standard specifications that involve more complex design considerations include: large incidence angles, control of the polarization at large angles, polymer substrates, extended wavelength coverage (VIS and a near-IR laser line, for example), UV or IR wavelength ranges, inclusion of a transparent conductive layer, abrasion and wear resistance for harsh environments, and exposure to high-energy laser radiation. To accommodate such requirements, the designs might require more layers, more than two materials, and alternate deposition processes.

"Cold Mirror" or IR-Rejection Dichroic Filter

The spectral requirement is for a coating that transmits the visible range of wavelengths (~420 – 680 nm) and reflects thermal energy of wavelengths >~700 nm. The rejection level of ~99% might be required extend to ~1100 nm in applications using a silicon sensor or where excessive heating can cause optical distortion effects. This cold mirror dichroic filter splits the energy regions typically at 45° incidence so the visible energy beam is folded 90° and the heat energy is transmitted. The opposite is true for "hot mirror" operation. These filters require good mechanical and environmental durability because they are often located near hot light sources, and some heating will occur. The substrate thermal properties also need to be considered with respect to preventing film fracture or de-adhesion or substrate cracking.

The designs are based on stacks of layers of quarter-wave optical thickness generally using TiO_2 and SiO_2, but the other high-index oxide compounds are often used as dictated by the deposition processes available for production. It is important to control the following spectral features: the cut-off slope and wavelength point and the in-band ripple. For tilted coatings, polarization splitting occurs, and the requirement should consider this expressed either as the mean value or the value for either polarization. A requirement variable affecting the cut-off wavelength point is the range of incidence angles around the nominal tilt angle. The rejection bandwidth is determined by the index ratio between the materials; a higher ratio produces a wider rejection zone. When the rejection region needs to be extended to exclude longer wavelengths, more QW stacks are added, resulting in increased in-band ripple. To smooth the ripple, matching layers, i.e., modified QW layers, are needed. This fact complicates what was otherwise a repetitious deposition routine of repeated thicknesses. The steepness of the cut-off slope and the level rejection both increase as layer number increases; but in-band ripple also increases in turn. Thus a compromise to the 'desired' vs 'required' specification is needed to minimize production costs and maximize lot yield. The thin-film designer would present trade-off options to assist in the final procurement specification.

Two-Band Reflector

Color analyzers or color projection systems might have a requirement to isolate two (or more) distinct narrow bandpasses or to attenuate specific region(s) of the spectrum. Satisfying this unusual requirement will heavily involve the thin-film designer. The designer's variables are refractive indices and layer thicknesses. The choice of materials and the layer configuration determine the locations and widths of the reflected bands. The figure on page 4 illustrates a 41-layer design solution that isolates blue and red bands and attenuates the green band. Attenuation can be varied by varying the layer number. The separation of the bands is affected by the index ratio between the high-and low-index layers, and their relative thicknesses. The coating engineer's variables include achieving the refractive indices and their absolute thicknesses if the bands are to be correctly centered. Iterations will be required to dial in the bandpass centers, and perhaps their widths.

continued on page 4

References

1. Thin-Film Coatings: Applications and Materials, Photonics Handbook, Laurin Publishing, pages H348-H357 (2003).

2. CMN V10 Issue 1 March 2000 and V1 Issues 1 & 2; V3 Issue 2; V9 Issue 4

3. CMN V6 Issue 1, Jan-March 1996.

4. CMN V12 Issue 3, Sept 2002.

5. CMN V13 Issue 1, March 2003.

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2-Band Reflector: Reflectance

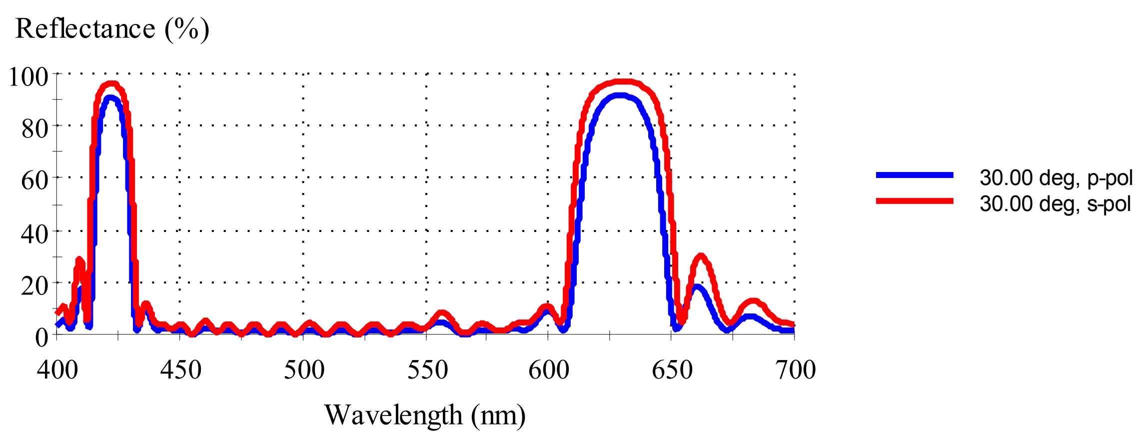

Figure 1. Performance of a design intended to isolate two narrow bandpasses and reject a region in between. Incidence angle is 30°