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Commonly Used Deposition Processes and Materials

Keeping Pace with Advancing Technology

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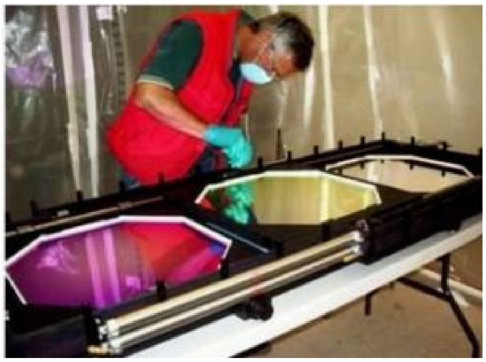

The evolution of optical coatings applications is limitless, and requires continuing development of deposition processes and materials. Both technologies have responded to new requirements for environmental stability, better optical quality and greater durability which were not previously required nor anticipated. Following is a review of a few preferred PVD processes and current applications, some of which were reported at the *Optical Interference Conference* (OIC), Tucson, June 2016.

Photo: Large area coatings are produced to rigid specifications for astronomical and other applications. Pictured are 50 cm bandpass filters produced by Materion Precision Optics.

Sputtering Replaces E-beam in Critical Applications

The introduction of electron-beam evaporation more than 50 years ago enabled high rates of deposition of high-temperature compounds such as the transition metal oxides. Those oxide materials are used in coatings for UV through Mid-IR regions, and commonly include SiO_2, Al_2O_3, HfO_2, Ta_2O_5, TiO_2, Y_2O_3, and others. However, thermal evaporation by a beam of electrons is an inherently unstable thermodynamic process. To help mitigate the instability, starting materials have been specially processed to provide greater compositional and physical uniformity. That processing consists of: deliberate production of conductive sub-oxide compositions, pre-melting and forming shaped sources, controlling density, and additive mixing. Starting material preparation and deposition process parameters work in concert to influence the optical and physical (mechanical) properties of the deposited thin-film layers.

Some degree of control over growth morphology is provided by simultaneous energetic ion bombardment during growth. E-beam evaporation with ion assist (IAD) is applied to increase packing density in growing film layers at lower substrate temperatures and to promote amorphous micro/nano-structural morphology. Both properties produce coatings with high consistency and improved environmental stability.

However, while better control of rate and composition is possible today compared to 10-20 years ago, E-beam deposition is rarely the process of choice for producing precision optical coatings such as narrow bandpass filters, dichroic edge filters, and beam-dividing coatings for near-UV through short-IR wavelengths. Those wavelength regions use the above-mentioned metal oxide materials that are produced by reactive sputtering. Longer wavelength IR, and coatings for wavelengths shorter than

~250 nm, require the use of non-oxide materials which are still best deposited by thermal evaporation sources, including E-beam. These materials include fluorides such as YF_3, YbF_3, HfF3, LaF_3, MgF_2, ZnS, ZnSe, Si, and Ge.

The requirement for the highest quality coatings in astronomy, biotech, NASA, military, medical, commercial, consumer, and entertainment applications has driven the development of a various techniques alternate to E-beam. (View archived issues of Coating Materials News that have previously compared and contrasted E-beam and sputter deposition processes). Stringent demands have been placed on wavelength placement and environmental stability properties. Sputter processes have supplanted E-beam evaporation for the production of many of those precision optical applications in the past 10 years. With magnetron sputter deposition, for example, the accuracy and precision of layer optical properties and thickness, as well as refractive index, are highly reproducible. The improved process control reproducibility has surpassed the advantage of the higher deposition speed that E-beam offered.

A primary reason for this is that sputter processes are stable, and therefore can be controlled through active monitoring, feedback procedures and even time. The sputter process has been adapted and scaled to produce large uniform bandpass filters for astronomy projects such as Subaru (600 mm diameter) and LSST (800 mm diam.).

Photo: 600 cm diameter Narrow Bandpass filter for the Subaru HSC observatory being transferred from the coating chamber at Materion Presision Optics.

LIGO Coatings: A Case in Point

The tendency to favor sputter processes for the production of precision optical coatings is evident in the recent OIC meeting related to optical coatings. A case in point relative to evolving coating process demands is the coating of the Laser Interferometer Gravity-Wave Observatory (LIGO) mirrors. LIGO senses gravitational waves that are the result of disturbances in space-time, and can detect a separation change as small 10 e^{-18} m between the arms of the interferometer. The tantala – silica high-reflector mirror coatings that are currently used must have an amorphous morphology and be optically and mechanically very uniform. The high reflectors of the 1064 nm line require absorptance <0.5 ppm. LIGO mirrors are fused silica cylinders, currently with a diameter of 20 cm, but will increase to 34 cm for advanced LIGO.

In spite of the application of the advanced deposition processes and materials that currently produce the highest quality films, the limit in detectability of gravity waves has been attributed to the coating itself. It has been discovered that atomic (Brownian) motion in coating layers contributes a noise level that currently imposes the smallest detection limit. This is new knowledge in the continuing discovery process in optical coating physics and deposition technology. The deposition techniques that are being investigated for the production of the LIGO mirrors, in addition to magnetron sputtering, are atomic layer deposition (ALD) and ion-beam sputtering (IBS).

One LIGO coating process uses atomic layer deposition (ALD). This process deposits film layers with high packing density and uniformity, as well low defects, low scatter, no pinholes, and low stress. These properties are ideal for minimizing coating “noise” sources and also for high LDT applications. In recent history, only Al_2O_3 was available for low-index layers. Precursors have been developed to permit the growth of SiO_2 layers. An advantage of ALD is the ability to uniformly coat curved and interior surfaces. It operates at a slow deposition rate, and substrate materials are limited to temperatures >200^o C.

Ion-beam sputter (IBS) is a higher energy process than magnetron sputtering or ALD. While it also has low deposition rates, it produces very low intrinsic absorption and high packing density with low defect density and very low absorption. This makes it ideal for high-energy laser and cavity ring-down laser applications. High transmission filters with steep edges and high out-of-band rejection are being manufactured for fluorescence excitation and detection (Semrock.com) by this process. IBS is being scaled to coat mirrors with sizes of the LIGO mirrors.

A parallel approach for producing low noise LIGO coatings involves material research and improvements. It appears that the high-index partner in QW HL mirror design contributes the bulk of the noise (L are the SiO2 layers). In addition to optimizing the deposition process, mixtures of materials may be employed to discourage crystalline growth and to increase stability. Post-deposition annealing is being studied in an effort to reduce thermal and stress-induced noise generation due to structural effects. Undoubtedly, deposition process and materials improvements flowing from the LIGO research will favorably impact and advance optical coating technology, just as DWDM developments did in the 1990’s.

Other Deposition Processes in Use or in Development

A significant improvement in film quality has been generated by IAD of E-beam film morphology and higher refractive index. The introduction of an energetic plasma environment as provided by Plasma Ion Assisted Deposition (PIAD) has been shown to produce amorphous dense metal-oxide films with complete oxidation, low stress, high indices, high damage threshold and environmental stability. The energetic plasma fills the coating chamber volume and encourages complete oxidation. Similar technology has been applied to magnetron sputtering to produce (what might be considered a hybrid variety) Plasma Assisted Reactive Magnetron Sputtering (PARMS). In this variation, higher energies are supplied to the sputter plasma to result in higher rates of oxidation and denser films.

There is interest in engineering the nano-structure of coatings for use in bio-sensing, photo-sensitized chemical activation, and polarization-sensitive coatings for medical and other applications. Such films are produced by using a tilted deposition geometry and rotation of the substrate during film growth. (See Coating Materials News V12/Issue 4, 2001 and other archived CMN issues that discuss structured nano-engineered film growth). Chiral glancing angle deposition (GLAD) column growth is accomplished by specifically programmed geometry to produce film layers with new optical properties.

Nano-structured multi-layer films are now in development that may possess unique optical and mechanical properties. They may have novel applications in optical components with different functions that planar-stratified layered coating film structures cannot achieve.

In Conclusion

This brief review has introduced evolving deposition processes and high-lighted some of the demanding applications that are being served. The advances open the door for future innovative utilization of processes and materials. Materion has been active in exploring new deposition and materials technologies while offering a full range of deposition processes.

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