CERAC™ incorporated
Coating Materials News
Volume 17 / Issue 4
December, 2007

Tutorial on Testing and Analysis of Optical Coatings

Beyond the materials and deposition process selection and development, there is the finished product, the coated optic. Testing standards for optical coatings have been used in practice for at least 45 years; they have been revised in the interim to accommodate improvements in coating and testing technology. This issue discusses how finished thin film optical coatings are examined and evaluated to determine if they meet the requirements imposed on them by the customer.

Quality Testing to Standard Test Protocols

Evaluation of coating physical quality involves testing according to standardized methods. The coating will either pass the optical, mechanical, chemical, and environmental requirements, or be subjected to further analysis to determine the cause for failure. The optical performance is specified to be stable within tolerable limits during or after the coated optic is subjected to various durability tests. The durability properties of coatings have traditionally been tested against US Military Standards (MIL-STD) or European DIN (Deutsches Institute fur Normung) and BSI (British Standards Institution) standards. These standards include requirements specific to interference filters, AR coatings, mirrors, and other optical coatings. For example, DIN series 58197-x includes a -1 part for AR coatings, -2 for mirrors, -3 for neutral beamsplitters, and -4 for laser optic coatings. BSI 07/03157861 DC is the most current for optics. BSI ISO and DIN ISO 9211-2:1994 is a combination spec. Copies of standards can be purchased from www.document-center.com.

The foreign standards have much in common with the US standards, and identical test protocols for abrasion, adhesion, temperature and humidity cycling, salt fog, and solubility persist. In the US, MIL-C-48497 deals with interference coatings as a general class of coatings that are located internal to a system and not subjected to harsh environments. MIL-C-48497 is the most appropriate specification for most optical coatings. The durability requirements of coated surfaces, such as AR coatings, on glass and other substrates that are exposed to external environments are called out in MIL-C-14806. These two standards have replaced the older versions: C-675 (MgF_2 AR on glass), M-13508 (aluminum mirrors), that covered single- and multi-layer coatings. MIL-C-14806 directly replaces MIL-C-675, and includes testing for resistance to the harsh marine exposure to salt fog, to jungle (fungus growth) and to desert (sand erosion) environments.

Special application coatings such as those used in medical instruments that require durability to steam sterilization in the autoclave or high damage thresholds for laser optics have additional requirements attached by the user. Laser coating testing is dictated by the laser wavelength, energy and duration. Coatings for CW lasers have lower damage thresholds than coatings used with pulsed lasers. Pulse width, repetition rate, and power are main considerations.

The entire durability standard, or application-specific sections of a standard, are called out on the procurement requirement documents for the coated surface by the customer's engineers. These are often derived requirements that flow down from system operation requirements, for example, coated windows that are used aboard ships where they are subject to salt-ion corrosion will have different survival and operational lifetime requirements associated with them than coatings used on vehicle windows that are used in an abrasive desert environment. Automobile windshield coatings must pass different tests than coatings used on entertainment display screens or commercial cameras.

Optical Performance Testing and Evaluation

The first test to be performed when the coating run is vented is a visual one that inspects the work and witness parts for stress cracking, spontaneous adhesion failure, color clarity, particulate inclusion and haziness, and general appearance uniformity. Witness test samples dedicated to the verification of optical, mechanical and environmental performances are coated simultaneously and in proximally with the deliverable work pieces. Their compositions, surface preparations and cleaning should closely represent the actual work. Verification of the requirements of spectral properties uses a spectrophotometer, and the spectral and transmittance or reflectance ranges are specified. The instrument accuracy can be established using traceable reference standards (reflectance) or internal calibration (transmittance). Incidence angle and environmental exposure might be conditions of the testing.

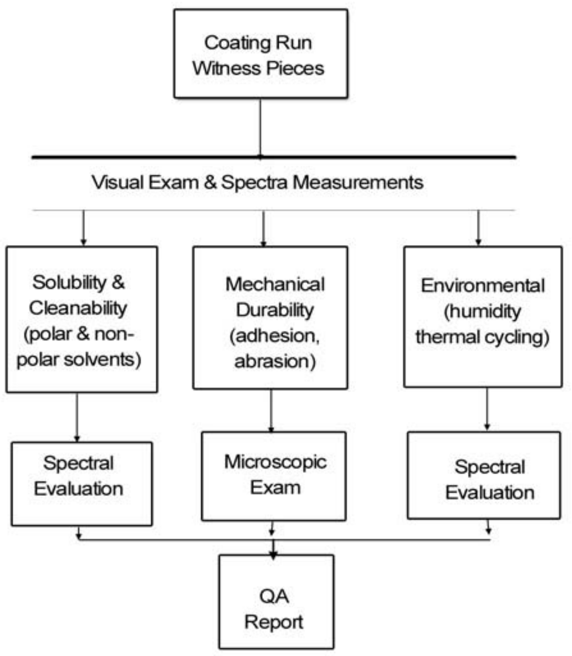
continued on page 2

Environmental Durability

Because optical films are thin (measured in µm), not dense, and are often deposited on chemically unrelated surfaces, they are vulnerable to degradation from chemical, thermal, and mechanical interactions. One purpose of the quality standards is to evaluate and project durability by subjecting coated surfaces to accelerated exposure to humidity, high-and low temperature, mechanical abrasion, and salt-ion exposure under carefully controlled conditions. Coated components used in scientific instruments either for commercial use or in high-value space applications must survive the MIL-C-48497 requirements.

A typical test regime is the following: 24 hours soak in 95% relative humidity (no condensation permitted) at 49° C followed by cleaning and then abrasion and adhesion tests. An unintended problem that has lead to declaring a coating "failed humidity" has frequently occurred when the humidity water bath has not been refreshed frequently enough to avoid the concentration of carbonic acid from the absorption of atmospheric CO_2. Coated surfaces must be cleanable and thus insoluble in alcohol, acetone, or water, depending on their composition. For example, fluoride coatings should not be soaked in water-based cleaners for an extended time. Evaporation of acetone caused rapid surface cooling and either thermal shock or water condensation either of which might damage some coatings. Moderate abrasion consisting of 50 rubs of a cheesecloth pad at 1 lb force. The force value specified will be influenced by the hardness of the substrate. In the severe abrasion test, a specially designated eraser loaded with clay particles is rubbed for 20 strokes at 2-2.5 lb force. Examination for density and depth of scratches under magnification is specified as a pass / fail criterion. In the case of optical windows, humidity or abrasion testing might increase surface scatter, leading to degradation of imagery through the window, or resistance might increase in the case of electronic / RF shielding windows. To determine adhesion to the substrate, a special adhesive tape is pressed in contact with and then pulled off either at an angle or perpendicular to the coated surface. The maximum acceptable removed area (spots) and location within the optic's clear aperture are defined. For example adhesion failure area below a specified value that occurs outside the used optical area might be acceptable. The tape-pull test is qualitative in nature, and does not represent any real-life condition; it only provides some degree of assurance of coating bond strength if no areas of coating are removed. A quantitative adhesion test consists of epoxying a stud to the coating and recording the force required to pull the coating from the substrate. This test is not covered in the Mil-standards. Abrasion and adhesion might be tested (no sooner than a specified time) after humidity exposure. This is intended to simulate real-life conditions where a coating's adhesion and cohesion might be compromised after exposure to humidity.

Figure 1



The inclusion within a coating of microparticulates that are emitted during evaporation can be revealed by the abrasion and adhesion tests. Such particulates cause light scatter, and their removal leaves pinhole voids that can admit corrosive agents (including water) that can initiate mechanical failure or transmit and scatter light. Such points concentrate stress, and thermal / humidity cycling often accelerates their reactivity.

Coatings might be subjected to temperature cycling over their intended survival / operational range. The rate of temperature change must be consistent with avoiding thermally shocking either the substrate or the coating, and is generally <5°C / min. Coatings used in a marine environment must resist corrosion by salt fog / salt spray exposure or by immersion in salt water. Exposure time, temperature, and salt concentration are dictated by the appropriate spec paragraph or system requirement. Windows and canopies used on high velocity aircraft need to withstand the following very harsh environmental events: high-velocity rain drop impact and dust erosion. Rain drops impacting at 450 km/hr can shatter a brittle coating and pit the substrate. Sand impact can abrade soft coatings and repeated exposure can erode them to the point that their useful functional life is severely shortened. Some exposed coated optical windows used in military vehicles must tolerate lubricant and fuel contamination and be cleanable to restore their optical and mechanical properties. An example of severe environmental testing applied to consumer ophthalmic polymer lenses is that the AR coatings must survive cycling between 2 minutes in boiling salt water to 1 minute in cool water without developing crazing or adhesion loss.

We have summarized the tests most often required for coated surfaces. Following these tests, either individually or sequentially, is the optical and other performance comparison with the "before" conditions. Spectral transmission / reflection measurements are used to quantify changes caused by testing. The system engineer will specify the permitted degradation as % loss of transmittance or reflectance averaged over a spectral region. Cosmetic appearance changes that do not affect the spectral performance are to be given low weight in the evaluation of coating durability.

Figure 1 graphically summarizes the durability testing and evaluation procedure appropriate for most optical coatings. The Mil-Standards dictate the sequences at which the individual tests are to be performed.

Analysis and Diagnosis

Failure analysis to determine the nature and consequently the causes of coating failures following the above durability testing often involves sophisticated surface analyses techniques. It is important in failure diagnosis and correction to establish the location of the specific failure. Failure at the coating-substrate interface might be the result of contamination, weak chemi-physical bond, or high intrinsic or thermal mismatch stresses. If the failure occurred after environmental testing, such as humidity or thermal cycling, there might be an unanticipated chemical reactivity at an interface, or pinholes that admit moisture or other gases.

The failed area might be examined by nondestructive microscopic imagery, elemental / compositional analysis, or a combination of techniques. Scanning electron microscopy (SEM) combined with Electron Spectroscopy for Chemical Analysis (ESCA or XPS) might be used to reveal the spatial distribution of materials, defects or contaminants, for example, from the quantitative analysis of elements having an atomic number greater than 8 (oxygen). Energy dispersive X-ray analysis (EDX or EDAX) is can be used to identify elements in a coating. There are many more high-energy laboratory techniques that can be applied by specialized surface analysis labs. The analysis depth is limited to <2 µm, but compositional depth profiling through thicker coating layers can be drilled by sputtering with energetic Ar ions between XPS sampling.

Atomic force microscopy (AFM) can be used to map the smoothness of areas several ìm in size. Imaging techniques using Nomarski microscopy at magnification below 1000x or SEM up to 20,000x is used to detect and analyze stress features to distinguish compressive (film buckling) from tensive (contraction cracking), and often one can associate a point of stress-related failure initiation with a microparticulate or surface contaminant. Nomarski interference microscopy is used to detect layer and substrate adhesion loss by detecting phase differences.

Summary Comment

Property measurement, evaluation, testing, and failure analysis are essential components in the successful development and production of optical (and other) thin film coatings. Coating houses contain a department equipped with quality-testing resources devoted to these components to provide not only reliable reports to be delivered with production, but also to maintain control on internal production processes. It is as easy to over test a coated optic beyond its requirements and thereby report a "failure" as it is to incompletely characterize its physical properties. Proper training in the execution of evaluation tests such as the Mil-standards and the proper use of measuring tools avoids false and misleading interpretations of coating quality, and the potential consequences.

CERAC Coating Materials News is a quarterly publication of CERAC, inc.
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Coating Materials News

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