Optical Monitoring of VLA Coating Process Using Hyperspectral Imaging

SBIR Topic No. MDA-02-045

Phase I Final Report

Contract Number: F33615-03-M-5005

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Abstract
An optical monitoring system has been developed and implemented which facilitates the repeatability of spectral results in the production of multilayer dielectric films for precision optical coatings. Surface Optics Corporation has integrated the SOC-700 hyperspectral imaging system with an electron beam evaporation vacuum coating chamber for the purpose of in-situ monitoring of reflectance and reflectance variation at any point on the substrate surface. The implementation of this technology enables the ability to (i) collect reflectance data over the entire substrate in-situ over a wavelength range of 0.4 to 0.9 microns, (ii) calculate thickness variation across the substrate for each layer (iii) re-optimize coating layer-by-layer based on measured reflectance data , and (iv) ensure reproducibility.

Introduction
Current High Energy Laser (HEL) mirrors require multi-layer optical coating stacks with a high degree of coating uniformity over apertures up to 2 meters in diameter. These layers can be very time consuming to manufacture, and the inability to achieve the desired thickness across the surface often leads to unfeasible optical properties. The ability to monitor the optical uniformity over the entire surface, layer-by-layer, would reduce the possibility of coating failure do to insufficient thickness, index variation, or non-uniformity. The improvement of optical performance and reduction of risk to rework is of great benefit to the HEL coating industry. In order to achieve the type of control and repeatability that is desired, some method of in-situ control of the deposition is required.

Background
Thickness monitoring is essential to a coating evaporation system. The rate of deposition is typically monitored by an oscillating quartz crystal (Quartz Crystal thickness monitor). This system has a built in quartz crystal oscillator that changes its frequency of oscillation as its mass changes. As material evaporates onto the crystal, the mass increases and the frequency of oscillation decreases. The processor is calibrated according to the material's density and position with respect to the substrate, so that the deposition of the coating material on the surface of the crystal is correlated with that which is being deposited onto the substrate. Essentially, quartz crystal monitoring provides data on the physical thickness of a deposited film, and tends to work best with metals and other samples not prone to outgas surface-absorbed moisture.

Optical monitoring, on the other hand, measures the optical thickness, which is a more accurate measurement of the film (except for highly reflective coatings), particularly when specific optical properties are desired. The reason optical monitoring is more accurate for interference coatings is because the data collected is a response of the thickness *and* the index of the deposited material. Optical responses are proportional to thickness measurements, assuming knowledge and repeatability of the refractive index. However, deposition conditions such as temperature and rate, can have an enormous effect on the refractive index.[2] Hence, the ability to control the optical thickness is crucial where complex precision optical coatings are desired.

Current optical monitoring systems are available that can provide wideband spectral data for in-situ process monitoring. However, these systems are typically limited to observation of a single position within the coating chamber. The SOC-700 is comprised of a (i) high-speed, low-noise visible/near-IR camera, (ii) high-quality spectrometer, (iii) integrated scanning system and (iv) software. This system can obtain and process 100MB hyperspectral data cubes in 6.4 seconds. The data cube contains spectral information on 120 different color bands per pixel over a wavelength range of 0.4 to 0.9 microns. By

using this camera to record color changes of an optical film during deposition, information pertaining to the film thickness and refractive index can be computed in real-time, over the entire coated surface.

Experimental Procedure

Three different multi-layer coating designs were deposited and imaged after each layer. The first coating design considered the simple case of a single dielectric layer over aluminum. The second and third sets consisted of alternating layers of dielectric and semi-conductive films.

Chamber Configuration

SOC's CHA electron-beam evaporation chamber was used for the experiments; the main features of the setup are illustrated in Figure 1. The chamber consists of a substrate fixture, electron-beam evaporation source, illumination source, and a reflectivity standard. The SOC-700 resides outside of the viewport, parallel to the coating plane; hence, a reflective surface is placed in the chamber at an angle to obtain radiance from the coating.

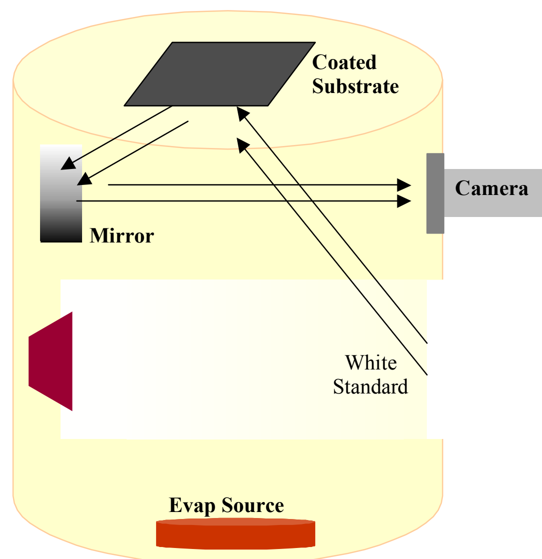

Figure 1. Chamber Configuration.

The angle of the reflective surface is accounted for in the modeling software. After each layer is deposited, the camera scans the surface and collects a three-dimensional cube of spectral information (wavelength, radiance, and position). From this information, one can obtain the energy reflected from the image as a whole, or from a particular point within the image. A halogen bulb with a coated directional backing was selected as the illumination source. This selection was made based on the source's ability to (i) provide sufficient illumination without radiating excessive heat, (ii) provide uniform illumination within the desired region, and (iii) operate under high vacuum.

Deposition and Optical Monitoring

Three sets of data were collected on stacks of incrementing complexity. The first consisted of a single dielectric layer, deposited with three different coating thicknesses, to establish the camera's ability to correctly register the corresponding interference fringes. The second consisted of a metallic/dielectric stack, and the third was a 44-layer dielectric stack. TfCalc™, a thin film optical design code, was used to model each stack. The physical thickness for each layer was guided by an Inficon™ crystal monitor, which had been previously calibrated for the appropriate material density, z-ratio, and chamber tooling factor. Radiance spectra were collected after each layer using the SOC-700 Hyperspectral imager. Physical thickness of the completed stacks were compared with the data using a DEKTAK-6 profilometer. Details of these tests are described in the following paragraphs.

Thickness evaluation: The arrangement of the substrate and step-measurement slide is shown in Figure 2. Three 5kA layers of SiO2 were deposited, and the radiance was collected after deposition.

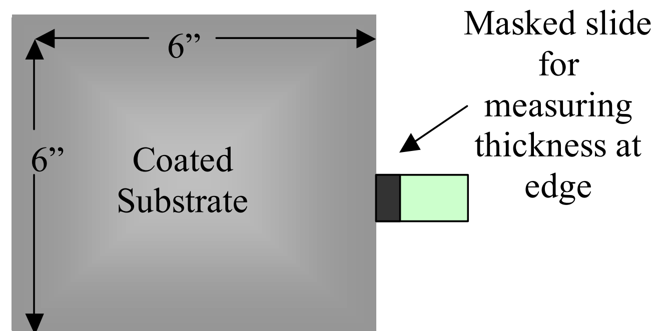

Figure 2. Arrangement of thickness measurement with respect to substrate.

Four Layer Stack: A 4four-layer stack consisting of alternating layers of silicon and SiO_2 was deposited on aluminum, and the data was collected in-between optical layers.

Dielectric Stack: The final run consisted of a 44-layer stack of alternating dielectrics (SiO_2 and Ta_2O_5). For this run, the system was reconfigured such that the camera was mounted to the viewport of the chamber to ensure that its position remained constant throughout the run. The software was augmented to automatically extract a region and convert radiance to reflectance for in-situ monitoring. Layer-by-layer reflectances were modelled using TfCalc™ before the run, so that the operator could compare the reflectance features at given wavelengths from the SOC-700 with the modeled reflectance spectra throughout the coating process.

Results

The following paragraphs present the results from the three coating trials.

Thickness evaluation: Figure 3a shows the reflectivity spectrum measured for a target coating thickness of 5kA of SiO_2 on aluminum. The predicted reflectivity using the optical design code, TfCalc, is also shown in Figure 3a. Figure 3b shows the predicted and measured response for a target coating thickness of 10kA of SiO2 on aluminum. Figure 3c shows the predicted and measured response for a target coating thickness of 15,000A of SiO_2 on aluminum. The profilometer measured a thickness at the edge of 14,580A. The SOC-700 reflectance at the edge is shown in Figure 3d, along with corresponding TfCalc spectra for the thicknesses.

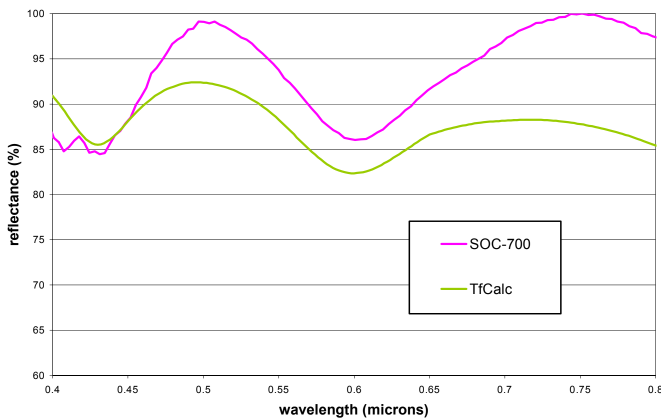

Figure 3a. Percent Reflectance for 5,000 Å SiO_2.

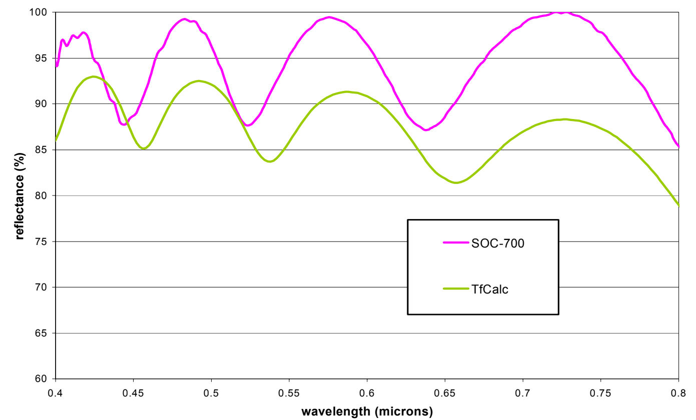

Figure b. Percent Reflectance for 10,000 Å SiO_2.

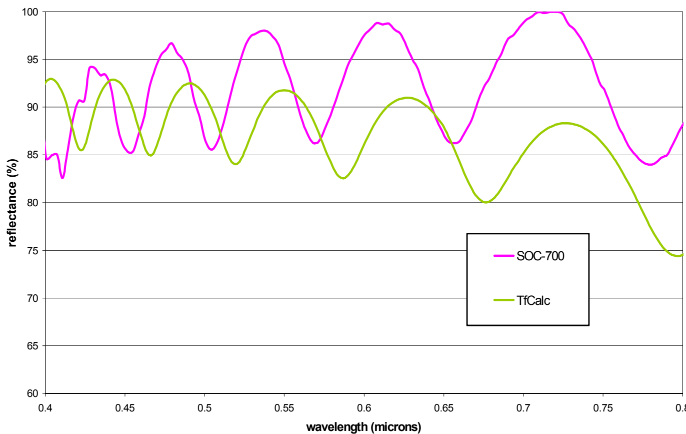

Figure3c. Percent Reflectance for 15,000 Å SiO_2.

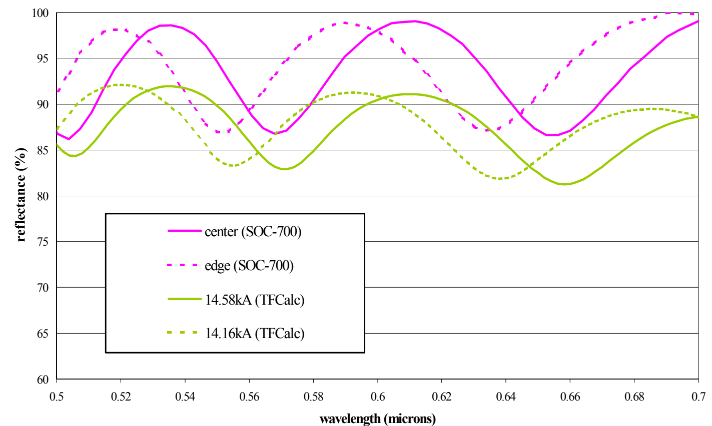

Figure 3d. Measured data at substrate edge and center matched with data modeled by TFCalc to determine thickness.

Four Layer Stack: Figure 4a shows the measured and predicted reflectance for the 3rd layer of a 4-layer stack comprised of SiO_2 and Si on Aluminum. An additional reflectivity curve is included on this figure, which describes the predicted reflective response for a coating thickness error of -2%. Figure 4b shows the measured and predicted reflective response for the 4th layer of this stack, as well as, the predicted

reflective response for a –2% coating thickness error. The coating sequence that generated this reflective response is given in Table I, below.

Table I. Coating Sequence for Figure 6.

Layer	Material	Target Thickness (A)
1	Aluminum	1,000
2	SiO_2	5,000
3	Silicon	300
4	SiO_2	5,000

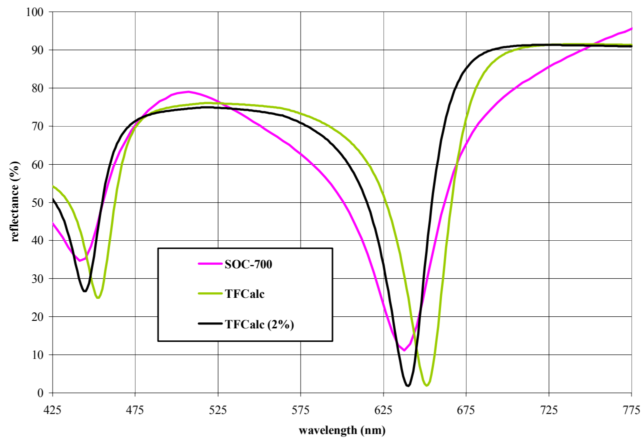

Figure 4a. Reflectance for Layer 3 (4-layer SiO_2, Si stack).

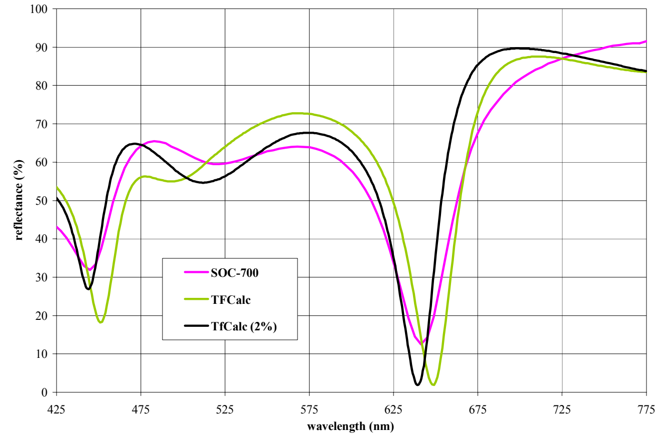

Figure 4b. Reflectance for Layer 4 (4-layer SiO_2, Si stack).

Dielectric Stack: Figure 5a shows the measured reflective response and two predicted responses (target thickness, and 6% coating thickness error), for the 10th layer in a 44-layer dielectric stack. The stack was comprised of SiO_2 and Ta_2O_5. The coating sequence is proprietary and is not presented in this report. A similar set of curves is presented in Figures 5(b-e) for other layers within the 44-layer stack. Radiance data was obtained for all 44 layers during the fabrication of this stack; however, only a fraction of these are presented in this report for illustration purposes.

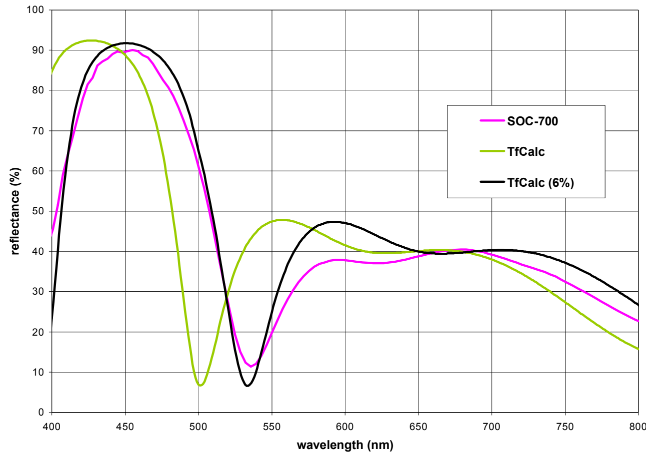

Figure 5a. Reflectance for Layer 10 (44-layer dielectric).

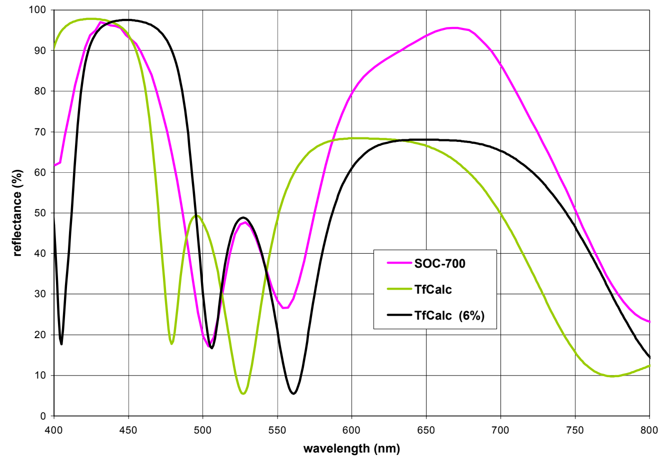

Figure 5b. Reflectance for Layer 14 (44-layer dielectric).

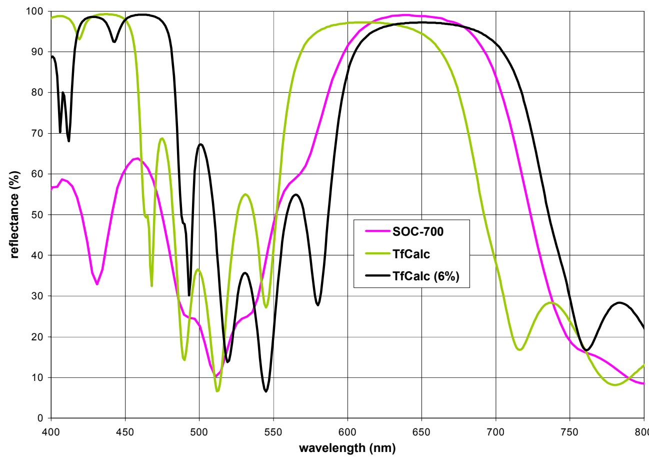

Figure 5c. Reflectance for Layer 24 (44-layer dielectric).

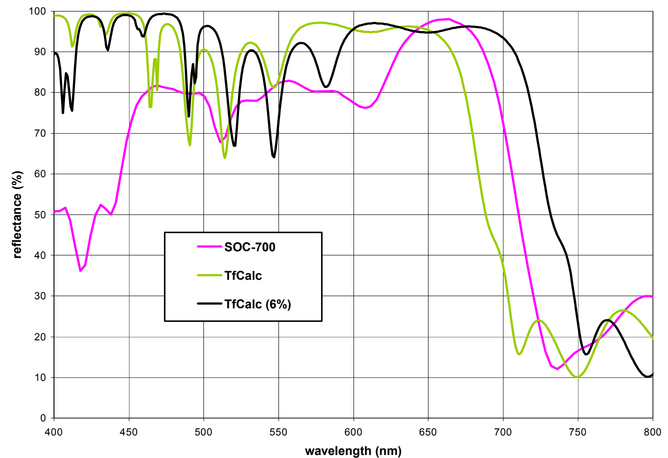

Figure 5d. Reflectance for Layer 32 (44-layer dielectric).

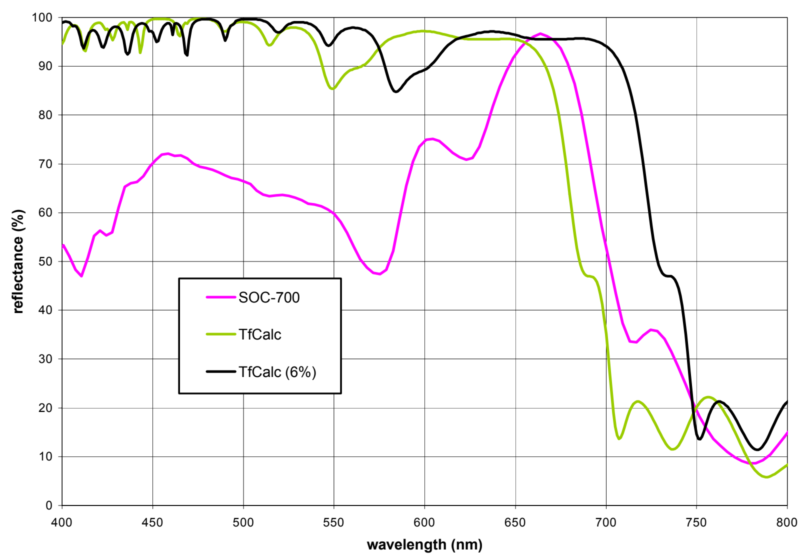

Figure 5e. Reflectance for Layer 44 (44-layer dielectric stack).

Surface Optics Corporation proprietary information – do not disclose.

Figure 6 shows the in-situ measured data after layer 44, as well as, reflectance measurements made by a Cary model 2 photospectrometer, before and after bake out. The predicted response based on TfCalc is also presented.

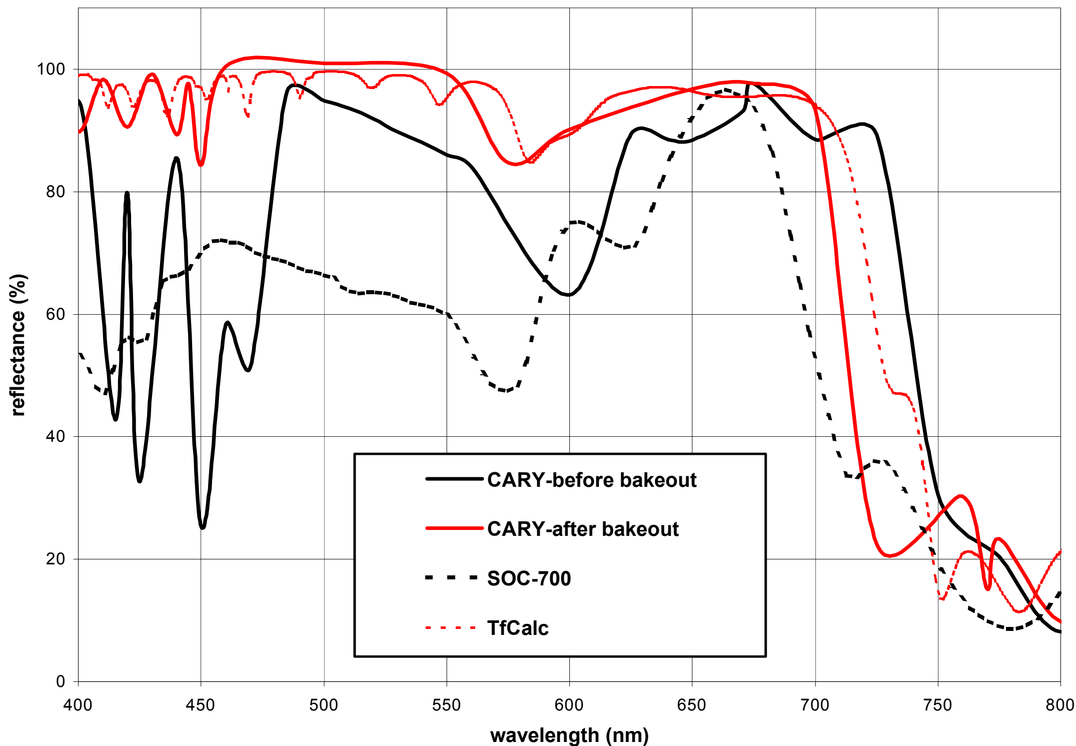

Figure 6. Comparison of 44-layer stack before baking and after with that measured by the hyperspectral imager and modeled using TfCalc.

Discussion of Results

Thickness evaluation: Figures 3(a-c) illustrate the use of interference minimums and maximums to determine coating thicknesses. In each case, TfCalc was used to estimate the actual coating thickness of the film based on the position of these minimums and maximums.

In Figure 3a, the peaks and valleys of the measured curve are slightly shifted from those of the modeled curve, indicating that the coating thickness was not quite 5kA. Since the same tooling factor was used for each layer, the shifts for 10kA- and 15kA- SiO_2 increased with increasing thickness. This increase is evident in Figures 6 and 7, respectively. The profilometry measurement the edge resulted in 14,160A at edge, and the reflectance from optical measurement for 14,160A matched well with that produced by TfCalc for the same thickness (Figure 3d). From here, the thickness in the center could be matched by TfCalc, which amounted to 14,580A. Hence, the hyperspectral camera showed that the crystal monitor tooling was off by 3%. The ability to monitor thickness within 1% should be highly beneficial for coating HEL mirrors, which require a coating accuracy of ~2% across a 2-m diameter part.

Four Layer: The x-axis data was used to locate sharp interference features, which were then used to compute the actual coating thickness of the deposit. In this Phase I research, the thickness values were determined by imputing various coating thickness values into TfCalc until a close match was made with the measured interference positions. This trial and error process was easy enough to carry out manually,

however, a primary goal of the Phase II program will be to automate this iterative solution process and integrate it with Schafer's THINFILM code. In Figures 4a and 4b, this manual iterative process was used to determine the coating thickness, which was approximately 2% less than the target value at the observed position (pixel).

Dielectric Stack: Figures 5a through 5e document the reflective response observed at various times during the deposition of the 44-layer dielectric coating stack. Figures 5a shows that the actual coating thickness is approximately 6% greater than the target value. With this 6% correction, a very good correlation between the absolute reflectance and the interference positions is made for the measured and predicted response curves.

In Figure 5b (after layer #14 was deposited), the sharp interference minimums match very closely with the predicted response for a +6% coating thickness error, however, the absolute reflectance deviates substantially in the red portion of the spectrum. The measured reflectance value in the red end of the spectrum is significantly greater than the predicted values. The reason for this is unclear at this point.

In Figure 5c (after layer 24) the absolute reflectance values match the predicted values in the red portion of the spectrum, but the blue end of the spectrum is no longer in agreement. This is exactly the opposite of Figure 5b. There are two possible reasons for the measured loss of energy in the blue portion of the spectrum. First, Ta_2O_5 is moderately absorbing through the visible spectrum until it is post-baked at 250°C. Therefore, as the thickness of Ta_2O_5 increases (as the stack progresses), one would expect to see significant deviations from the predicted response. Secondly, when the coating run was complete, it was observed that the white standard being used to illuminate the substrate had yellowed due to the high temperatures from the illumination source, which resulted in a loss of reflective response.

Figure 5d (after layer 32) shows a greater loss of energy measured in the blue end of the spectrum; as more Ta_2O_5 is deposited, and as the white standard was degrading, this is not surprising.

Figure 5e shows the completed 44-layer stack. A considerable amount of energy was lost below 600nm, and there is little agreement between the measured and predicted response curves.

Figure 5f clearly illustrates the effect of both the bake-out and the degradation of the white standard. The coated sample removed from the coating chamber was measured using a Cary model 2 photospectrometer before and after baking at 250°C. Before baking, the sample sustains little agreement with the predicted reflective response, and a significant amount of absorbed energy is observed in the blue end of the spectrum. After baking, however, the same sample agrees very well with predictions. By comparing the Cary measurement before baking with the final SOC700 measurement, the severe degradation of the white standard is observed between 425 nm and 600 nm.

Future Efforts

From the data presented in Figures 5(a-e), it is clear that a more heat resistant material is needed for use as a white standard such as Teflon sheet or thermal-control white paint. In addition, some coating was observed on the standard when the coated substrate was removed from the chamber, therefore, subsequent experiments will include better shielding of the standard from the evaporating material.

Since some optical materials, such as Ta_2O_5, develop their optical properties after they are removed from the coating chamber, predicted responses based on standard n,k data may not be particularly useful. In this case the recorded response curves may be used for process control of subsequent identical manufacturing operations (such as when the 44 layer coating is made a second or third time), or, n,k data may be generated for unbaked films for use in the predictive thin film code.

Conclusions

An optical monitoring system has been developed and implemented which facilitates the repeatability of spectral results in the production of multi-layer dielectric stacks for precision optical coatings. Surface Optics Corporation has integrated the SOC-700 hyperspectral imaging system with an electron beam evaporation vacuum coating chamber for the purpose of in-situ monitoring of reflectance and reflectance variation at any point on the substrate surface, during the deposition process.

Phase I research showed that the integration of the SOC-700 with an e-beam evaporation coating process is capable of:

1. Collecting reflectance data over the entire substrate in-situ over a wavelength range of 0.4 to 0.9 microns.
2. Calculating thickness variation across the substrate for each layer.
3. Optically "steering" a film to a desired thickness.
4. Providing process documentation to ensure reproducibility for complex, multi-layer dielectric stacks.

Many technical challenges were encountered during the course of this SBIR; however, there were no major technical issues that would prevent the end goal of this research to yield the accuracy and uniformity required for HEL coatings and other precision optical coating applications.

References

1. M. Ohring, *The Materials Science of Thin Films*, San Diego, CA (1992), pg 522.
2. Ibid, pg 522.
3. R.R. Willey, *Society of Vacuum Coaters 36th Annual Technical Conference Proceedings* (1993), pp 156-159.