Quality control of beam splitters and quarter-wave-mirrors using the Agilent Cary 7000 Universal Measurement Spectrophotometer (UMS)

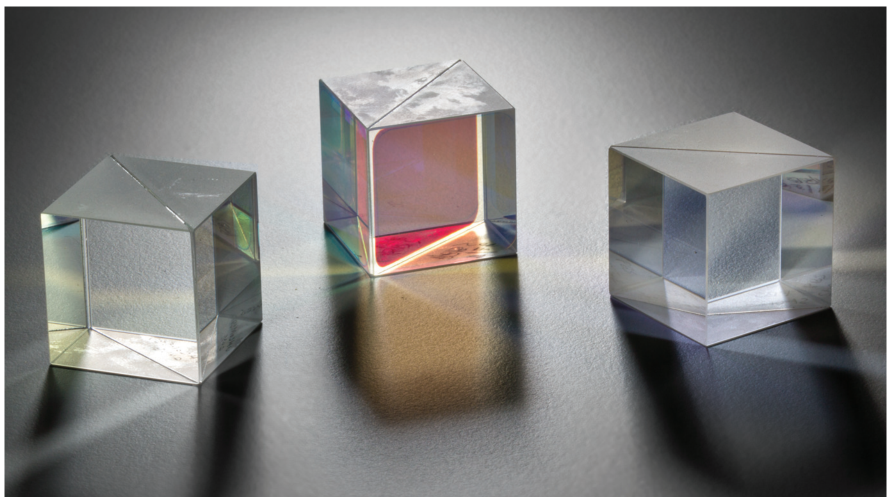
Application note

Materials testing and research

Authors

David Death
Farinaz Haq

Agilent Technologies, Australia



Introduction

Optical coatings and coating technologies have matured over many years in terms of the design, production and characterization processes. Today optical coatings are ubiquitous and can be found in applications from research and space optics to consumer items and industry. The variety of applications include eye wear, architectural and automotive glass, illumination and lighting systems, displays, optical filters, specialty mirrors, fiber optics and communications and medical optics. The performance of optical coatings depends on the specifications of the coating and substrate materials.

The design and manufacture of high quality multilayer optical coatings require accurate measurements of not only the final production component but also the optical constants of the materials in the thin film layers. These measurements enable the detailed design of sometimes very complex multilayer coatings. Measurements made at the end and during production can also be used to reverse engineer optical coatings to provide feedback on the design manufacturing process [1]. A primary aim of reverse engineering is to detect systematic and random errors in individual layer parameters. This helps to improve layer control and optimize the optical coating deposition.

Reliable reverse engineering of an optical coating depends critically on accurate measurements of reflection and transmission. Historically these measurements have been limited to normal incidence transmission (T) and/or near normal incidence reflection (R) data. As may be expected the ambiguity of multilayer reverse engineering grows as the number of coating layers increases. In general it is possible to minimize the ambiguity in reverse engineering by using more measured data. Both T and R measurements made at a range of angles of incidence (AOI) are valuable for the characterization of thin film materials and the reverse engineering of multilayer coatings. Most typical reverse engineering involves the detailed numerical analysis of normal or quasi-normal incidence T and R data related to the coating under study. While this approach is experimentally simple, it can lead to unreliable results due to the limited information available in near normal T and R data sets and the influence of measurement errors within those data sets [1]. In particular, reflection data from broadband reflectors or transmission data from to broadband antireflection (AR) coatings can be considered as examples of such low-information data sets. Historically simple normal incidence T measurements have been available using a wide variety of spectrophotometers and near normal incidence R measurements similarly so by fitting an appropriate reflectance accessory.

This application note demonstrates a new form of multi-angle photometric spectroscopy using a unique automated double beam UV-VIS-NIR multi-angle spectrophotometer, the Cary 7000 Universal Measurement Spectrophotometer (UMS). Example measurements of multilayer coatings used to create a spectral beam splitter and two 43 layer quarter-wave stack mirrors on differing substrates are presented alongside the reverse engineering analysis enabled by the obtained multi-angle spectral photometric data set.

Experimental

Samples

Measurements of three different coatings are summarized from the work of Amotchkina et al. [2]. The first coating, BS-AR-Suprasil, is a specialized beam splitter designed for an oblique AOI of 45°. The 52 layer reflector is deposited on a 1 mm thick Suprasil substrate. The front surface coating specification calls for a spectral transmission profile of greater than 98% T between 935 nm and 945 nm and greater than 98% R between 967nm and 971 nm. Additionally a 10-layer broadband AR coating is deposited on the rear surface. Optical coatings typically consist of alternating layers of varying thickness of high and low refractive index materials. For this first sample the high index material used was Niobium Pentoxide (Nb_2O_5) and the low index material was Silica (SiO_2) and the coating was deposited using a Leybold Optics GmbH Helios magnetron-sputtering system.

Both the second and third samples are high reflectors constructed of 43 layer quarter-wave stacks with the design wavelength for the reflector at 800 nm. The coatings were deposited on two differing substrate types, 6.35 mm thick fused silica and 1.0 mm thick B260 glass. The sample names were HR800-FusedSilica and HR800-Glass. The high index material used in these coatings was Hafnia (HfO_2) and the low index material used was Silica (SiO_2). The coatings were deposited using e-beam evaporation in a Leybold Optics GmbH SYRUSPro 710 coating machine.

Instrumentation

The reflectance and transmittance of the completed coatings were obtained using the Cary 7000 UMS which is a highly automated variable-angle absolute specular reflectance and transmittance UV-Vis-NIR spectrophotometer. The Cary 7000 UMS provides users with automated independent motorized control over both the sample AOI and the angular positioning of the detector, see Figure 1. This independent control of both sample AOI and detector position allow for rapid, accurate and unattended measurements of optical multilayer coatings.

Reflectance and transmittance measurements have traditionally been performed using different spectrophotometer accessories. This leads to different areas of the sample being measured for reflectance and transmittance. Deposition processes, though well controlled, are not perfect and films are deposited of non-uniform thicknesses. As a result reflectance and transmittance measurements can vary across the surface as the coating thickness changes. With the development of the Cary 7000 UMS, it is now possible to measure R and T from the same point on the sample surface without moving the sample when changing from R to T measurement modes. Additionally the sample can be rotated 180° to permit static transmittance measurements to be performed in the forward or reverse direction. In a similar way the AOI in a reflectance measurement can be varied to either side of the sample normal and the detector can be moved to make R measurements at ±AOI. In either case both R and T can be measured from the same point without removing and replacing the sample in the spectrophotometer or changing to another accessory.

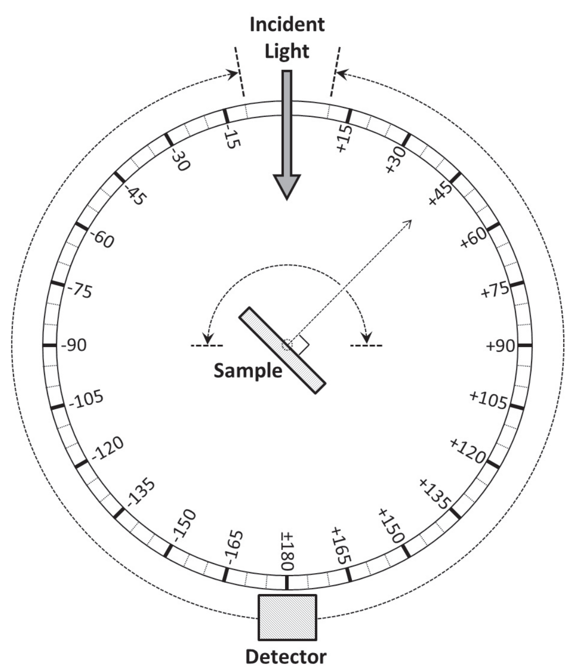


Figure 1. The AOI on the sample and the detector position can be set independently with 0.02° resolution. The AOI on the sample ranges from -85°≤AOI≤85°. The detector can be placed at angles ranging from -10° to 10°. As a result the Cary 7000 is able to determine R over the range 5°≤|AOI|≤85° and T over the range 0°≤|AOI|≤85°.

Results and Discussion

Small variations in the layer thickness as each is deposited and variations in the optical characteristics of the materials used under differing coating conditions result in the overall performance of an optical coating not meeting the original design intent. The design and analysis of optical coatings are accomplished using sophisticated computer software packages which rely on the accurate knowledge of both the physical thickness of each layer and the optical constants of the materials used to build the coating. The three coatings described were designed using OptiLayer, a suite of software consisting of modules for the design of multilayer coatings, prediction of performance, characterization of optical materials and reverse engineering of coatings from measured transmittance and reflectance data.

Some modern coating machines have the facility to monitor the normal incidence transmission of the coating as it is deposited [3-4]. These *in-situ* measurements made at normal incidence are used as a basis for predicting the final design oblique incidence performance of the coating laid down. Detailed analysis of this *in-situ* data using OptiLayer typically shows rough agreement between positioning of the reflectance and transmittance bands with the initial design. However *in-situ* normal incidence measurements are no real substitute for actual oblique angle R and T measurements of the completed coating. In their article Amotchina et al. describe how measurements made using the Cary 7000 UMS allowed them to reverse engineer the deposited coating and hence fine tune the coating process using *in-situ* measurements to more closely match the original design intent of the coating.

In the example of the BSAR-Suprasil beam-splitter the primary uncertainties lie in the thicknesses of the individual layers as the optical properties of the Nb_2O_5 and SiO_2 are quite well understood. In this study, the Cary 7000 UMS was used to measure the sample after coating. The coating specification calls for the beam-splitter to be used at a 45° AOI. Using the Cary 7000 UMS it was possible to measure both R and T at a number of different AOI from the same patch on the surface of the sample. Increasing the number of measurements in the data set (more AOI) served to reduce the level of uncertainty in the results from reverse engineering the coating. Using this data and analysis it was then possible to correlate with *in-situ* measurements and construct an optimization strategy for the deposition. Finally measurements using the Cary 7000 UMS were used to validate the optimization.

Figure 2 compares the predicted and measured spectral transmittance of the optimized BSAR-Suprasil beam-splitter at an AOI of 45° Figure 2(a). The unpolarized transmittance measurements were made using the Cary 7000 UMS. The spectral agreement between the theoretical curve (Optilayer) and the measured data points is exceptionally good. Differences in peak heights are primarily due to the spectral bandwidth used to collect the measured data. Further measurements of the BSAR-Suprasil sample were also made at an AOI of 30° for both S and P incident polarization, this data is shown in Figure 2(b) along with the predicted spectral transmittance. Once again the agreement between measurement and theory is excellent. The close agreement between these measurements made at oblique AOI of 45° and 30° and the model predictions serves to validate the reverse engineering and model refinement used to optimize the coating deposition based on the *in-situ* normal incidence transmittance measurements.

The second and third samples measured were examples of multi-layer quarter wave stack mirrors designed to be oblique incidence high reflectors. Each mirror consisted of 43 quarter wave alternating layers of Hafnia (HfO_2 — high index material) and Silica (SiO_2 — low index material). The difference between the samples being the type and thickness of the substrate used: HR800-FusedSilica — 6.35 mm thick fused silica; HR800-Glass — 1 mm thick B260 glass. In these coatings uncertainty in both the optical properties of the Hafnia and individual layer thickness need to be considered. Once again the mirror is designed for an oblique AOI of 45° with un-polarized incident light.

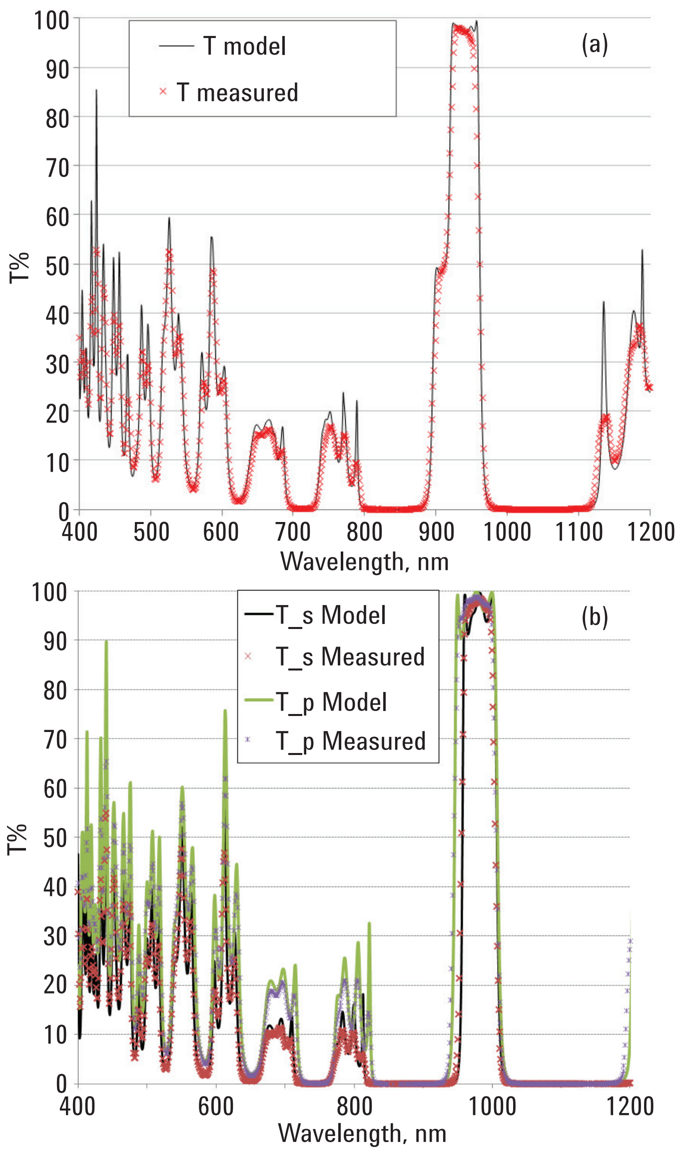


Figure 2. Comparison of oblique incidence experimental transmittance data for the sample BS-AR-Suprasil with model transmittance (a) non-polarized light at 45°, (b) s- and p-polarizations at 30°

As with the first sample, measurements of these samples using the Cary 7000 UMS were used to characterize the final coating performance, optimize the coating strategy and validate the outcomes of the reverse engineering analysis. The strategy developed involved reverse engineering *in-situ* normal incidence measurements during the coating procedure to make adjustments on the fly. As an example of the influence of material and layer thickness variations, Figure 3 compares the desired design specification with the *in-situ* and final transmittance measurement using the Cary 7000 UMS of the un-optimized coating. As can be seen there is significant deviation between the three data sets. The primary difference is observed as a shift of the reflectance band toward shorter wavelength and differences in the width of the reflectance band.

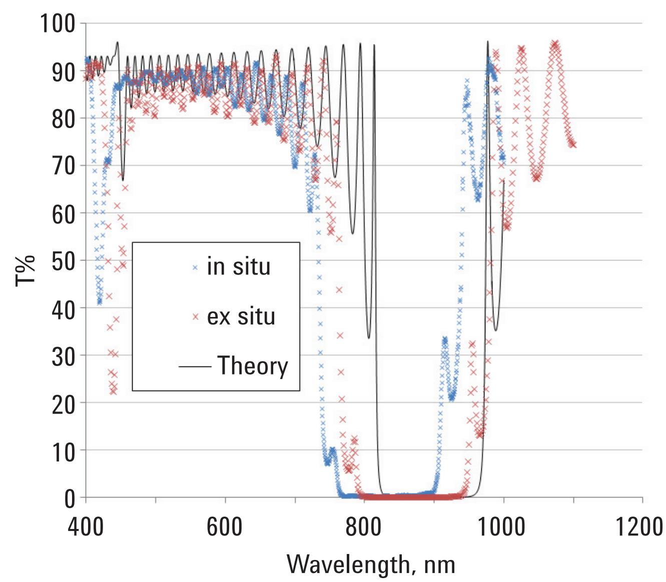


Figure 3. Comparison of *in-situ* normal incidence transmittance data, Cary 7000 UMS (ex-situ) measurements and theoretical transmittance for the HR800-Glass sample

The positioning of the reflectance band towards shorter wavelengths is understood in terms of an underestimation of the individual layer optical thicknesses as they are laid down. This may be associated with either an error in the physical thickness of the deposited layers and/or an uncertainty with respect to the optical properties of the layer materials (refractive index and absorption coefficient). Errors in the geometrical thickness of coating layers can arise from inaccurate calibration of the quartz crystal layer thickness monitors used to control the deposition. Errors in material properties on the other hand arise from incipient variation of the nominal refractive index with deposition temperature. HfO_2 exhibits such a variation of refractive index as a function of deposition temperature and at the relatively low deposition temperature used here (120 °C) there is a degree of uncertainty in its value. Further, the width of the reflection band of a quarter wave mirror stack depends on the ratio of the high and low refractive indices used [5]. On that basis the design specification should result in a width of 126 nm. The normal incidence *in-situ* measurements indicate a width of 133 nm and the Cary 7000 UMS measurements indicates a width approaching 143 nm. Considering that the uncertainties associated with the optical properties and porosity of the silica layers to be small it is apparent that the refractive index of the Hafnia layers is larger than the value assumed in the construction of the coating model. The apparently higher refractive index may be explained by the porous structure of the HfO_2 layers. Under vacuum the porous structure of the HfO_2 layers remains empty and the refractive index is correspondingly low. Exposing the coating to atmospheric air fills the pore structure with water vapor, thereby increasing the refractive index. This process is commonly known as a vacuum shift [6]. These effects can be accounted for reasonably accurately by allowing for random errors in the layer thicknesses and random offsets in the refractive index of the HfO_2 layers [2].

Figure 4 shows the measured transmittance curves for normal and oblique incidence compared with the predicted design curve for the 800 nm high reflectance coating on the 6.35 mm fused silica substrate (HR800-FusedSilica). Once again a wavelength shift is observed, but this time it is toward longer wavelengths. The widths of the high reflectance regions are again also

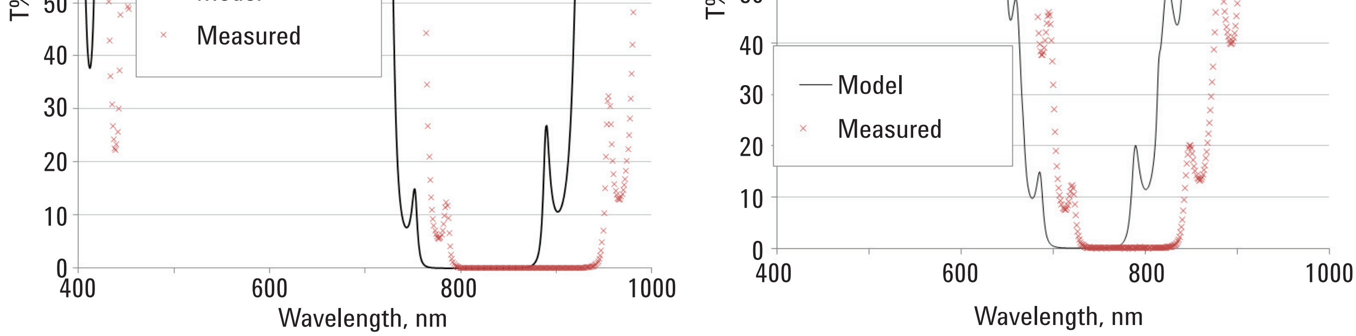
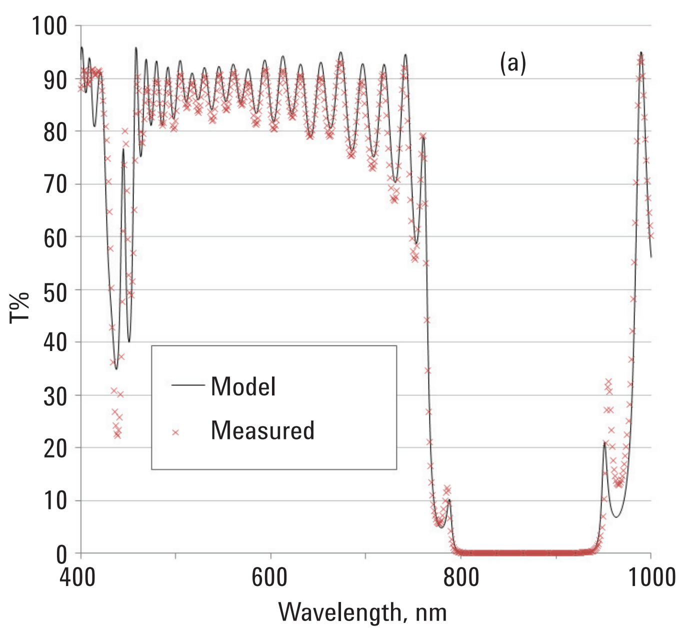


Figure 4. Comparison of normal (a) and AOI = 45° (b) experimental transmittance data related to HR800-FusedSilica sample with model transmittances calculated for intermediate design

different. For this coating the physical layer thicknesses were found to be reasonably accurate. The shift in position of the reflectance band was in this case due to uncertainties in the refractive index values assumed for the HfO_2 layers. The HfO_2 layers are considered to be inhomogeneous due to inter-diffusion of the HfO_2 and SiO_2 coating materials [1]. Random variations in the refractive index of the various HfO_2 layers were thus included in the model and the predicted transmittance recalculated for normal and oblique (45°) incidence and compared with measurements mad on the Cary 7000 UMS.

The final fitting of the normal incidence measurements by model data is shown in Figure 5(a). The model fit to the measurement is reasonable indicating that there is still room to improve the model. The model takes into account all the main features of the deposited coating however it may be further improved by allowing for variations in the degree of material inhomogeneity on a layer by layer basis and the definition of interlayers caused by diffusion of HfO_2 and SiO_2 materials. This level of sophistication cannot be based on the results of one set of transmittance or reflectance measurements at one particular angle of incidence. The Cary 7000 UMS is uniquely placed to provide both R and T data from a given sample at the multiple angles required to provide confidence in modelling these effects.

Figure 5(b) shows the comparison between the coating model and the measured transmittance at 45°. The results demonstrate good agreement between Cary 7000 UMS measurement and the predicted curve. Further the close agreement between measurement and model is confirmed in measurements at all angles made using the Cary 7000 UMS from AOI of 0° to 45° at 5° intervals.



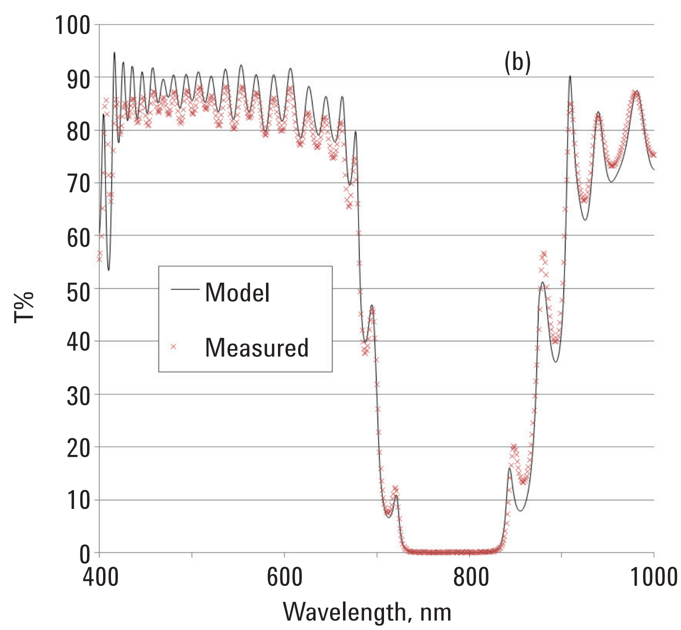


Figure 5. Final fitting of normal (a) and oblique incidence AOI = 45° (b) experimental Transmittance data related to HR800-FusedSilica sample by model transmittances

Figure 6 show the residual difference between the measured and calculated transmittance for both S and P polarization at 800 nm wavelength. As can be seen there is good agreement between the absolute transmittance measured by the Cary 7000 UMS and the value predicted by the model.

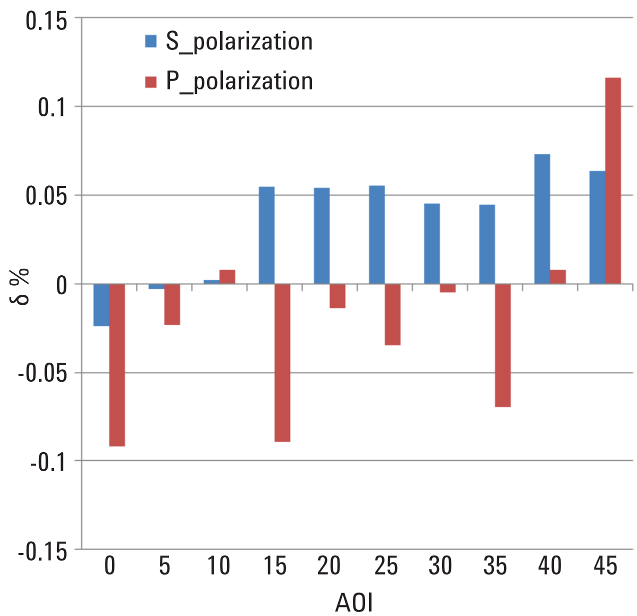


Figure 6. Residual differences between experimental and model transmission measurements data for the HR800-FusedSilica sample at the wavelength of 800 nm versus the AOI

Conclusion

The Cary 7000 UMS has been shown to be a valuable tool for the measurement and characterization of complex multilayer optical coatings. The Cary 7000 UMS provides independent and automated control of sample rotation and detector position giving it the unique ability to measure both reflectance and transmittance at different angles without having to move the sample. The facility of an automated polarizer gives the added benefit of being able to address both S and P polarization measurements, all done with the light incident on exactly the same place on the surface of the sample. The Cary 7000 UMS is an ideal candidate for QA/QC in optical coating environments because of its convenience, ease-of-use and ability to operate and produce accurate data completely unattended.

Amotchkina et al. demonstrate the value that accurate measurements of optical coatings made at a multiplicity of angles has for the accurate characterization, control and optimization of complex optical coatings. Such measurements enable and validate the adoption of intricate optimization strategies in coating deposition, particularly for coatings designed for application at oblique incidence. Such strategies often involve *in-situ* measurements made at normal incidence only followed by reverse engineering of the coating from a limited dataset. The Cary 7000 UMS enables optimization and validation through the provision of accurate measurement across a range of angles.

References

1. A.V. Tikhonravov, T.V. Amotchkina, M.K. Trubetskov, R.J. Francis, V. Janicki, J. Sancho-Parramon, H. Zorc and V. Pervak, "Optical characterisation and reverse engineering based on multiangle spectroscopy," Appl. Opt. 51(2), 245-254 (2012).

2. T.V. Amotchkina, M.K. Trubetskov, A.V. Tikhonravov, S. Schlichting, H. Ehlers, D. Ristau, D. Death, R.J. Francis, and V. Pervak, "Quality control of oblique incidence optical coatings based on normal incidence measurement data," Optics Express, 21,18, 21508–21522 (2013).

3. D. Ristau, H. Ehlers, S. Schlichting, and M. Lappschies, "State of the art in deterministic production of optical thin films," Proc. SPIE 7101, 71010C, 71010C-14 (2008).

4. H.E. Ehlers, S.S. Schlichting, C.S. Schmitz and D.R. Ristau, "Adaptive manufacturing of high-precision optics based on virtual deposition and hybrid process control techniques," Chin. Opt, Lett., 8, 62–66 (2010).

5. S.A. Furman and A.V. Tikhonravov, "Basics of Optics of Multilayer Systems, Editions Frontieres, (1992).

6. O. Stenzel, S. Wilbrandt, S. Yulin, N. Kaiser, M. Held, A. Tunnermann, J. Biskupek, and U. Kaiser, "Plasma ion assisted deposition of hafnium dioxide using argon and xenon as process gases," Opt. Mater. Express, 1(2), 278–292 (2011).

Published February 26, 2014
Publication number: 5991-4030EN