# Optical characterization of thin films using a new Universal Measurement Accessory for the Agilent Cary UV-Vis-NIR spectrophotometers

## Application note

Materials

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## Introduction

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The accurate determination of the optical parameters of thin films and multilayer coatings (using reverse engineering of optical coatings) is paramount to the manufacturing of high quality products. The data provides a feedback to the design/production chain. Reverse-engineering results — where each layer is assessed in turn — can be used to adjust deposition parameters, recalibrate monitoring systems, and improve control of the thicknesses of individual layers.

Typically, optical characterization is based on the analysis of normal- or near-normal-incidence transmittance (T) and/or reflectance (R) data of a thin film sample on a transparent substrate using UV-visible-near-IR (UV-Vis-NIR) or Fourier transform IR (FTIR) spectrophotometry. However, optical characterization based on normal incidence T and R measurements and reliable reverse engineering based on normal-incidence or near normal-incidence T and R measurement data is challenging.

In this application, we demonstrate the applicability of multi-angle spectral photometric data to the optical characterization of single thin films and the reverse engineering of multilayer optical coatings using a Cary 5000 UV-Vis-NIR spectrophotometer equipped with a new Universal Measurement Accessory (UMA). We consider dense thin films and a multilayer produced by magnetron sputtering, as well as electron-beam (e-beam) evaporated thin films, which are typically more difficult to characterize. This data could also be collected on the Agilent Cary 7000 Universal Measurement Spectrophotometer (UMS).

## Experimental

### Samples

For our study we measured two sets of experimental samples using two different deposition techniques: magnetron sputtering and e-beam evaporation. Details can be found in reference [1].

### Instrumentation

- Agilent Cary 5000 UV-Vis-NIR spectrophotometer
- Agilent Universal Measurement Accessory

The UMA is a highly automated variable angle specular reflectance and transmittance system with full software control of the sample, detector and polarizer positions. It provides accurate, rapid and complete optical characterization of samples via transmission (%T) and absolute reflection (%R) measurements at various controllable angles of incident light (0–85 deg %T, 5–85 deg %R). The linearly polarized beam that illuminates the sample can be measured in transmission. It can be measured in absolute reflectance by moving the detector assembly in a plane at a constant radius from the sample. This multiple measurement mode capability of the UMA results in improved productivity and more precise characterization of samples. A schematic of the UMA is presented in Figure 1.

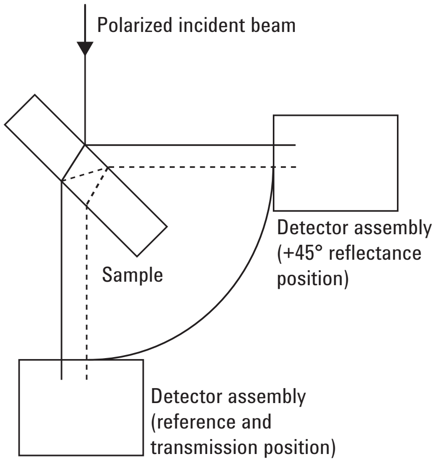

**Figure 1.** Schematic of the Agilent UMA, an absolute variable angle reflectance and transmission accessory

## Results and discussion

Multi-angle spectral photometric measurements were performed for all samples in the spectral range from 300–2500 nm at incidence angles of 7°, 10°, 20°, 30°, and 40° for s- and p-polarized light. In all optical characterization and reverse-engineering procedures throughout this study, we used measurement data taken in the spectral range from 330–1100 nm only. Substrate internal absorption is significant above 1100 nm wavelength, making estimation of accuracy uncertain.

### Dense dielectric thin films

The UMA was used to acquire multi-angle spectral photometric data for the optical characterization of $Ta_2O_5$ and $SiO_2$ thin films produced by magnetron sputtering. In Table 1, we present numerical results of optical characterization: measured film thicknesses and refractive index values at $\lambda = 600$ nm. There is excellent consistency of the results obtained using T and R data measured at different incidence angles and with different polarization states. For both materials, deviations of thickness and refractive index values (n) from mean values in all columns of Table 1 are lower than 0.1%.

**Table 1.** Parameters of $Ta_2O_5$ and $SiO_2$ films found by using oblique-incidence T and R data acquired using the Agilent UMA

| | $Ta_2O_5$ | | $SiO_2$ | |
|---|---|---|---|---|
| **Polarization state/angle of incidence** | **Physical thickness (nm)** | **n at 600 nm** | **Physical thickness (nm)** | **n at 600 nm** |
| s, 7° | 292.3 | 2.162 | 401.4 | 1.486 |
| s, 10° | 292.5 | 2.160 | 401.7 | 1.485 |
| s, 20° | 292.4 | 2.161 | 401.5 | 1.484 |
| s, 30° | 292.4 | 2.161 | 401.9 | 1.484 |
| s, 40° | 292.4 | 2.161 | 401.6 | 1.483 |
| p, 7° | 292.7 | 2.159 | 401.9 | 1.484 |
| p, 10° | 292.5 | 2.160 | 401.4 | 1.485 |
| p, 20° | 292.5 | 2.160 | 401.5 | 1.484 |
| p, 30° | 292.5 | 2.160 | 401.9 | 1.486 |
| p, 40° | 292.4 | 2.161 | 401.7 | 1.483 |

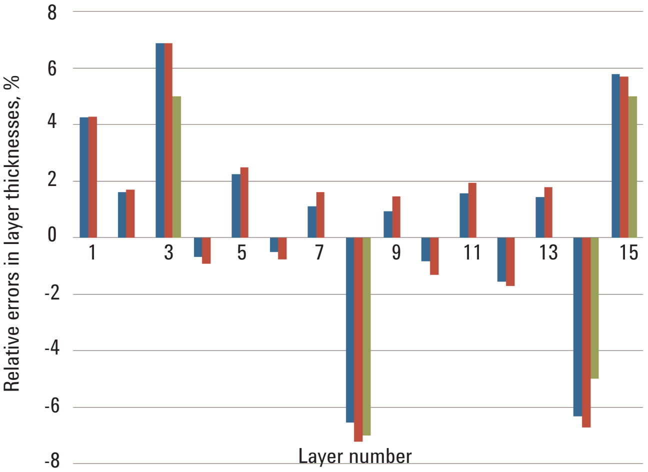

**Figure 2.** Comparison of errors in layer thicknesses of 15-layer quarter-wave mirror found on the basis of reflectance and transmittance data taken at 7°, 10°, 20°, 30°, and 40°, for the s-polarization case (blue bars) and the p-polarization case (red bars). Green bars show planned errors in the thicknesses of the third, eighth, 14th, and 15th layers.

## Reliability of reverse engineering based on multi-angle spectroscopy

To check the reliability of reverse engineering based on multi-angle optical photometric data, we analyzed a specially prepared 15-layer quarter-wave mirror with $Ta_2O_5$ and $SiO_2$ as high and low index materials. The mirror was produced by magnetron sputtering by using time monitoring of layer thicknesses. During the deposition of this mirror, intentional errors of +5%, –7%, –5%, and +5% were imposed on the third, eighth, 14th, and 15th mirror layers, respectively. Various combinations of input measurement data was acquired using the UMA and the intentional thickness errors were reliably detected in all cases [1]. A typical example of the consistency of obtained results is presented in Figure 2.

## Application of multi-angle spectroscopy to optical characterization of inhomogeneous e-beam evaporated thin films

We also applied multi-angle spectral photometric measurement to the determination of optical parameters of e-beam evaporated $HfO_2$ and $SiO_2$ films of various thicknesses. This was achieved by reverse engineering of a specially prepared multilayer mirror. It was found that the optical properties of the e-beam evaporated $HfO_2$ films are dependent on film thickness. Results of all reverse-engineering attempts were consistent. The offsets of $SiO_2$ refractive indices determined in the course of reverse engineering were in the range from 1.5% to 1.7% with respect to the nominal refractive index of $SiO_2$ found from characterization of the single $SiO_2$ layer. A good agreement in refractive indices of $HfO_2$ layers was also observed.

The variations of $HfO_2$ refractive index values, determined from separate oblique incidence T and R measurements, did not exceed 0.5%. It can be seen in Figure 3 that the measured refractive index wavelength dependence of $HfO_2$ film is in agreement with reference wavelength dependencies from a previous study [2]. This agreement confirms the previous conclusion that the crystalline state of $HfO_2$ depends on film thickness [3, 4]. As shown in these references, thin films are basically amorphous while thicker films are partially crystalline, and the larger the crystalline fraction, the thicker the film. This can explain the difference in refractive indices of our $HfO_2$ films that are 197 nm thick in the case of a single layer and approximately 50 nm thick in the case of a multilayer structure.

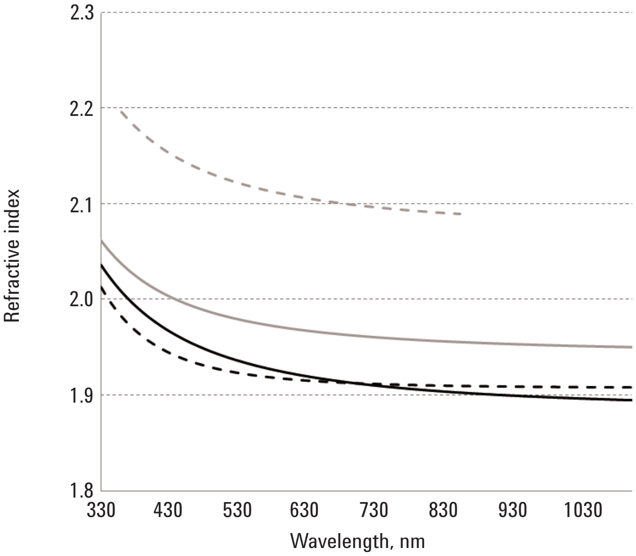

**Figure 3.** Nominal refractive index wavelength dependence of e-beam evaporated $HfO_2$ film (solid black curve), and reference refractive index wavelength dependencies of $HfO_2$ films produced by radio frequency sputtering (gray curve) and ion-beam sputtering (dashed gray curve). The dashed black curve shows the refractive index of thin $HfO_2$ film found from measurement data related to a 12-layer quarter-wave mirror.

## Conclusions

We studied the applicability of multi-angle spectroscopy to the optical characterization of thin films and reverse engineering of multilayer coatings. The UMA, a new advanced spectrophotometric accessory developed by Agilent (and fitted to an Agilent Cary 5000 UV-Vis-NIR spectrophotometer), supplied reflectance and transmittance data for multiple angle and s- and p-polarization states. The accuracy of measurement data was verified and it was confirmed that all measurement data was excellent over a wide spectral range from the UV to the NIR up to the incidence angles of 40°.

Multi-angle spectral photometry provides researchers with more experimental information than conventional spectroscopy. Our study demonstrates that the new UMA spectrophotometer accessory provides experimental information that permits the solving of various optical coating characterization and reverse-engineering problems.

Comparative analysis of various combinations of input multi-angle spectroscopic data provides self-verification of the results obtained. We believe that multi-angle spectral photometry is the perfect tool for the analysis of optical coatings under oblique light incidence or at diverged light illumination.

## References

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