High volume optical component testing using an Agilent Cary 7000 Universal Measurement Spectrophotometer (UMS) with Solids Autosampler

Application note

Materials testing and research

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Introduction

Manufacturers of high quality multilayer optical coatings require reliable methods to accurately measure optical performance of thin film materials. Traditionally this is accomplished using two separate measurements: normal incidence transmission (T), typically conducted within the sample chamber of a spectrophotometer, and near normal reflectance (R) measurements, which require the use of a separate reflectance accessory. Ensuring that both measurements are made from exactly the same patch on the sample is difficult using this approach due to sample repositioning during changes in instrument configuration between R and T measurements. However, a recent development by Agilent Technologies, the Cary 7000 UMS overcomes this limitation by measuring multi-angle transmission and absolute reflection measurements from exactly the same point of a sample's surface, without sample repositioning. This method eliminates systematic errors often introduced due to small variations in angle of incidence (AOI) when a variety of %R and %T measurement techniques are employed.

QA/QC of optical thin film coatings

Effective quality assurance and quality control (QA/QC) of optical thin film coatings has relied on accurate spectroscopic measurements taken during and at completion of a coating procedure. Current QA/QC testing is typically limited to representative witness pieces introduced into the coating process for testing purposes. Witness piece testing is preferred to comprehensive testing of large numbers of finished-goods because of the prohibitively high cost-per-analysis in high volume multiple sample testing.

In this study, we demonstrate increased productivity and reduced cost-per-analysis with automated, unattended multi-angle R/T analysis for multiple samples of uncoated fused silica using an Agilent Cary 7000 UMS fitted with an Agilent Solids Autosampler.

Experimental

Instrumentation

- Agilent Cary 7000 Universal Measurement Spectrophotometer
- Agilent Solids Autosampler

The Cary 7000 UMS is the latest generation of high performance UV-Vis-NIR spectrophotometers designed for Multi-Angle Photometric Spectroscopy (MPS) applications over the wavelength range 250–2500 nm. MPS measures the absolute reflectance and/or transmittance of a sample across a range of angles from near normal to oblique incidence [1]. The UMS performs variable angle transmission and absolute reflectance measurements from the same patch of a sample's surface. The linearly polarized beam that is incident on the sample can be used to measure transmission, and by rotating the detector assembly about an axis through the sample and perpendicular to the plane of incidence, in reflection. The UMS also functions as a goniospectrophotometer providing further capability for diffuse reflectance measurements of non-specular surfaces and diffuse transmittance measurements of translucent materials. The addition of an automated polarizer further enables accurate measurement at S, P or user specified polarization angles.

Figure 1a. Plan view of the Cary 7000 UMS measurement chamber with the Solids Autosampler installed

Figure 1b. A multi-sample holder with capacity to mount up to 32 x 1 inch diameter samples

The accessory component of the Cary 7000 UMS, the Cary UMA (Universal Measurement Accessory), is available as an upgrade option for existing Cary 4000/5000/6000i UV-Vis-NIR spectrophotometer users.

The Solids Autosampler is an independently controlled sample holder designed specifically for the Cary 7000 UMS and UMA. It can be mounted inside the Cary 7000 UMS measurement chamber as shown in Figure 1a. In addition to the angle of incidence (AOI) control (θ_i) provided by the UMS, the Solids Autosampler provides two additional degrees of freedom; radial (z) and rotational direction (Φ) about the incident beams axis (I_o). A variety of sample holders allow mounting of multiple individual samples (up to 32 x 1 inch diameter), Figure 1b, or single large diameter samples (8 inch diameter).

Recent studies have shown that the inclusion of MPS data at angles beyond near normal incidence provides better reverse engineering of complex thin films [2]. Additionally multi-angle photometric spectroscopic data has also provided insight into oscillations in the total losses in thin dielectric films [3]. Recently measurements made using MPS measurements performed on a Cary 7000 UMS have been used to validate and optimize reverse engineering strategies applied during coating production runs [4].

Results and Discussion

Single-sample analysis

The Cary UMS was used to provide sequential multi-angle measurements for both absolute specular reflectance and direct transmittance on the same patch of sample of 1 mm thick fused silica without repositioning of the sample (Figure 2). This simple measurement of the transmission and reflection from a 1 mm thick plate of fused silica was conducted at angles of incidence ranging from 0 to 82° in transmission and 6 to 82° in reflection in both S and P polarization. The physical size of the silica sample limited the measurable range of angle of incidence to <82° without the incident beam falling off the

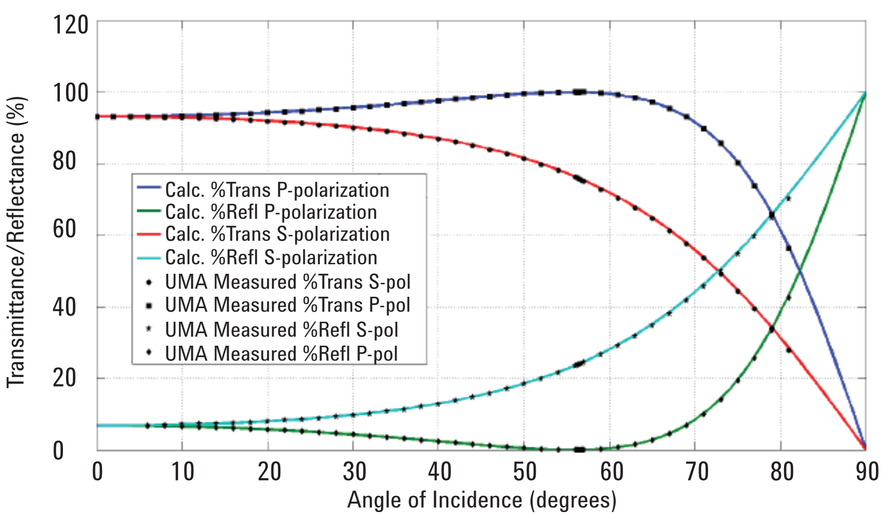


Figure 2. Absolute reflectance and transmittance of a 1mm thick silica sample plate as a function of angle of incidence. The solid lines are calculated from the Fresnel equations and the symbols are values measured using the Cary 7000 UMS. Measurement wavelength: 500 nm; physical size of the fused silica sample limited AOI range to 0–82°.

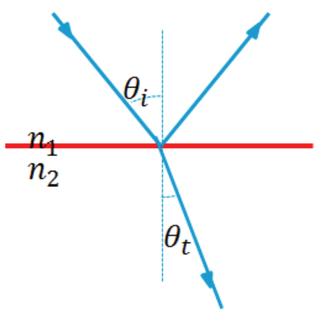
sample surface. The measurements shown include contributions from both the front and internal rear surface reflections and transmissions. The individual points represent the measured values and the underlying solid lines indicate the total reflectance and total transmittance predicted by the Fresnel equations:

Reflection (R) and Transmission (T) Coefficients for s and p polarized light

$$R_s = \left|\frac{n_1\cos\theta_i - n_2\cos\theta_t}{n_1\cos\theta_i + n_2\cos\theta_t}\right|^2 \qquad R_p = \left|\frac{n_1\cos\theta_t - n_2\cos\theta_i}{n_1\cos\theta_t + n_2\cos\theta_i}\right|^2$$

$$T_s = 1 - R_s \qquad T_p = 1 - R_p$$

n_1 = refractive index of incident medium
n_2 = refractive index of sample
θ_i = angle of incidence
θ_t = angle of transmission



Where n_1 was taken to be 1.00 (air) and n_2 the refractive index of fused silica as determined by the Sellmeier Equation:

$$n_2(\lambda) = 1 + \sum_i \frac{B_i\lambda^2}{\lambda^2 - C_i}$$

λ = wavelength
B_i and C_i = Sellmeier coefficients

Sellmeier coefficients are typically supplied with the optical data sheets of transparent materials.

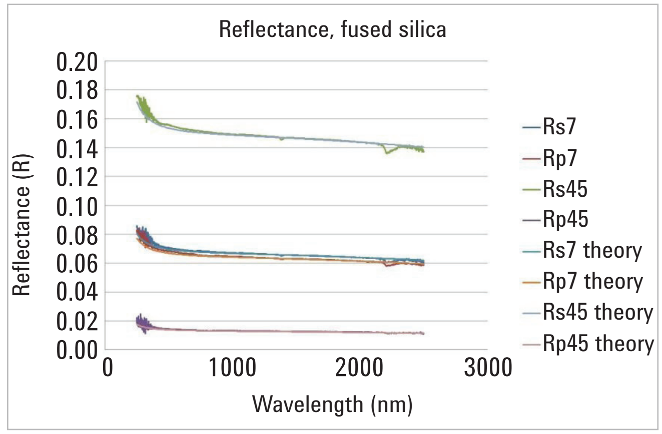


Figure 3a. Reflectance of fused silica at sample position #1 in the multi-sample holder. Theoretical lines are calculated from the Fresnel equations. Measured results were made using the Cary 7000 UMS with Solids Autosampler

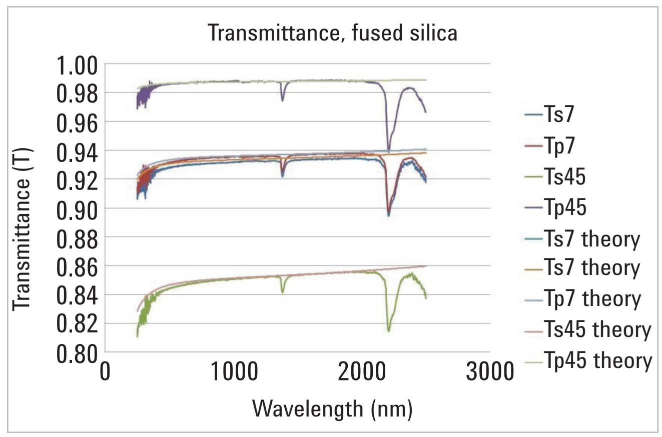


Figure 3b. Transmittance of fused silica at sample position #1 in the multi-sample holder. Theoretical lines are calculated from the Fresnel equations. Measured results were made using the Cary 7000 UMS with Solids Autosampler

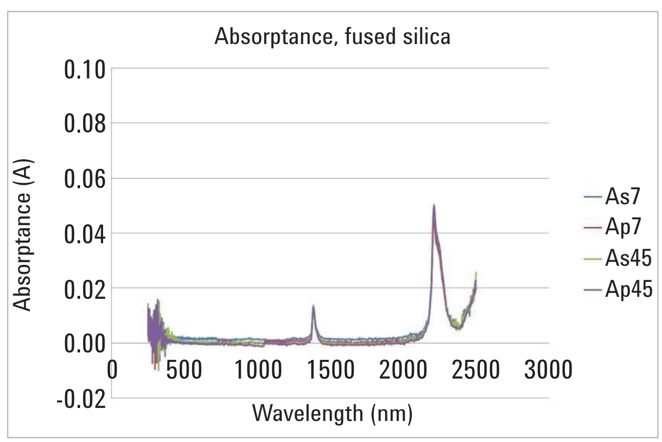


Figure 3c. Absorptance (A) of fused silica at sample position #1 in the multi-sample holder. Absorptance data is calculated from T, and R data using A = 1 – R – T. Aside from the water absorption band at ~1400 nm, absorptance data 500–2000 nm is expected to be approx 0.00

Multi-sample analysis

The Solids Autosampler was used to extend MPS measurements from a single piece of fused silica to 11 individual pieces of uncoated fused silica (38 mm x 42.5 mm x 1 mm), over the wavelength range of 250–2500 nm. Each sample was measured in R and T, at AOI +/-7° and AOI +/-45°, under S and P polarization. The positive(+) and negative(-) collection angles were automatically averaged post data collect and the final spectra denoted as; Ts7, Rs7, Tp7, Rp7, Ts45, Rs45, Tp45, Rp45. The time taken to collect the 16 spectra per sample was approx 40 minutes and the total collect time for the 11 samples was less than 8 hours. Data was collected in a single unattended overnight run without further user intervention. Absorptance, defined as A=1-R-T, was calculated for each angle and polarization.

Full spectral range results of sample #1 are presented in Figure 3 showing close agreement with theory over a wide range of signal levels, angles, polarization states and wavelength. Residual errors, calculated as the difference from Fresnel theory for the central wavelength 1500 nm, are given in Table 1.

Factors that may affect the accuracy of these measurements include the residual uncertainty in the symmetry of the system, sample mounting and long term drift of the instrument.

- Spectral data from the positive(+) and negative(-) angles were averaged to help correct for any optical asymmetries in the measurement.
- Samples were located by the perimeter of their front face against a precision machined surface. They were then clamped between two plates to help ensure they were reproducibly located and perpendicular to the incoming beam I_o.
- The fully automated collection of the data was conducted unattended, without any need to open the measurement chamber. An initial baseline was collected prior to analyzing the 11 samples. Drift correction was not applied.

The quality of the data obtained, and the close agreement between the measured and theoretical results, indicate that the reproducibility and stability of the system is sufficient for fully automated and unattended data collection and that the symmetry of the Cary 7000 UMS was very close to optimal.

Table 1. Residual error calculated for the 8 collect conditions for each of the 11 samples in the multi-sample holder

Residuals	**Sample number**											**Average**	**StDev.**
	1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**		
Ts7	0.01	-0.01	-0.02	-0.01	-0.01	-0.01	-0.02	-0.03	-0.03	-0.05	-0.07	0.02	0.019
Rs7	-0.06	-0.05	-0.06	-0.06	-0.05	-0.06	-0.06	-0.05	-0.05	-0.07	-0.05	0.06	0.005
Tp7	0.00	-0.02	-0.02	-0.01	-0.02	-0.03	-0.04	-0.05	-0.06	-0.08	-0.10	0.04	0.030
Rp7	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.01	0.02	0.004
Ts45	-0.04	-0.06	-0.07	-0.07	-0.05	-0.07	-0.08	-0.08	-0.09	-0.10	-0.12	0.08	0.024
Rs45	-0.16	-0.16	-0.16	-0.16	-0.16	-0.16	-0.16	-0.16	-0.17	-0.17	-0.17	0.16	0.004
Tp45	-0.04	-0.05	-0.06	-0.05	-0.05	-0.06	-0.07	-0.08	-0.09	-0.11	-0.14	0.07	0.031
Rp45	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.02	0.03	0.002
Average	0.04	0.05	0.05	0.05	0.05	0.06	0.06	0.06	0.07	0.08	0.09		
StDev.	0.049	0.048	0.048	0.050	0.048	0.047	0.044	0.048	0.047	0.049	0.056		

Conclusion

The Agilent Cary 7000 UMS with Solids Autosampler has been shown to provide automated and unattended routine measurement of the optical properties of multi-samples of uncoated fused silica with good agreement between the measured and theoretical results. The total collect time for the 11 samples was less than 8 hours, acquired in a single overnight run, compared to days needed by non-MPS methodology. The increased productivity offered by the 7000 UMS would lead to a significant reduction in QA/QC cost-per-analysis of industrial optics.

Further, the UMS provides a wide range of functionality which enables routine MPS measurements of both the absolute reflectance and transmittance from the same place on the surface of a wide range of specular and/or diffuse samples. The measurement of spectral data across a wide range of AOI provides better characterization of the performance of materials and coatings employed for precision optics. This data can also assist in the validation of optical coating designs by reducing the uncertainties encountered in reverse engineering of coating parameters.

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