Application Note

Materials testing and research

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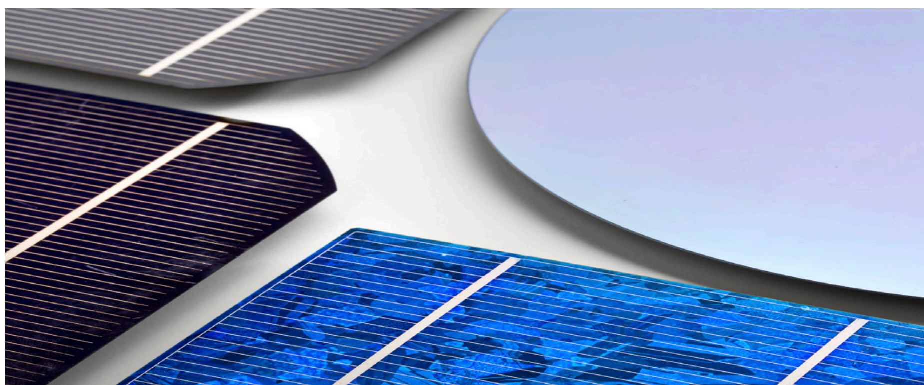
Spectrophotometric Spatial Profiling of Coated Optical Wafers

Efficient handling of large samples and multiple UV-Vis-NIR reflectance measurements using fully automated sample handling



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Introduction

Frequent and cost effective spectroscopic characterization is important in the development of competitive optical thin film coatings. Fully automated and unattended spectroscopic measurements can help reduce cost per-analysis, increase productivity, and help expand quality assurance programs. In the manufacturing process, large, usually circular substrate wafers are coated in deposition chambers that usually operate at full capacity. An efficient and productive optical characterization tool needs to deliver accurate and meaningful information at user-defined points across the surface of the wafer before it is cut. The Agilent Solids Autosampler, designed for the Cary 7000 Universal Measurement Spectrophotometer (UMS) and Universal Measurement Accessory (UMA), can mount samples up to 200 mm (8") in diameter and provide angular absolute reflection and transmission data in the UV-Vis and NIR spectral range.

The capabilities of the Cary 7000 UMS, together with the autosampler, have been previously demonstrated for the automated and unattended analysis of multiple samples mounted in a 32x sample holder (*1*) and spatial mapping of a linear energy band gap gradient of a zinc tin oxide (ZTO) layer (*2*). This study examines the use of the Cary 7000 UMS with autosampler for automated, angular resolved mapping, of the coating uniformity across a 200 mm diameter wafer.

Experimental

Instrumentation and sample

Cary 7000 UMS UV-Vis-NIR spectrophotometers (Figure 1) are designed to perform multi-angle photometric spectroscopy (MPS) measurements over a wavelength range of 250 nm to 2500 nm. In MPS applications, the absolute reflectance and/or transmittance of a sample is measured over a wide range of angles of incidence from near normal to oblique (*3*). MPS data has proved helpful for reverse engineering complex thin films (*4*), gaining deeper insights into oscillations in the total losses in thin dielectric films (*5*), and improving reverse engineering strategies applied during coating production steps (*6*).

The simple but versatile design of the UMA allows it to position the sample and the detector at any angle, independently of each other, without operator intervention. In a single sequence, the UMA acquires both transmission and reflection data from exactly the same patch of a sample's surface at variable angles of incidence (θ_i) in the range $5° \leq |\theta_i| \leq 85°$ (that is, angles on either side of beam normal noted as +/-). Adding an automated polarizer based on nano-wire grid technology makes it possible to obtain accurate measurements at S, P, or any user-specified polarization angle.

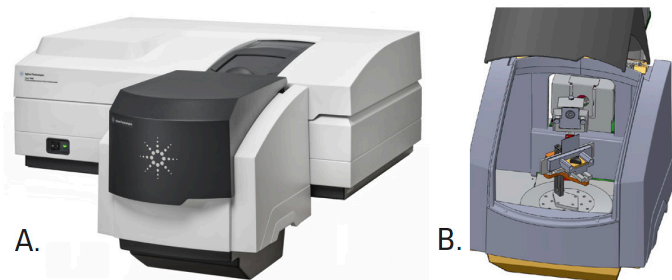


Figure 1. A. The Agilent Cary 7000 UMS. B. The Universal Measurement Accessory, included with the Cary 7000 UMS. The UMA is a true multi-modal measurement system capable of absolute reflectance, transmission and scattering analysis.

The centerpiece of the Cary 7000 UMS, the Cary Universal Measurement Accessory (UMA), is available separately to upgrade existing UV-Vis-NIR spectrophotometers from the Cary family, specifically the 4000, 5000, and 6000i.

Autosampler

The Agilent Solids Autosampler is an independently controlled sample holder especially designed to work with the UMA. The autosampler mounts inside the large UMA sample chamber over the axis of rotation of the sample stage. In this way, the autosampler doesn't limit the basic functions of the UMA. In fact, it enhances the measurement capabilities by adding two degrees of freedom to the sample position. As seen in Figure 2 these additional degrees of freedom are in radial (z) and rotational direction (Φ) about the incident beams axis (I_o). Depending on the type of sample, a variety of sample holders allow mounting of multiple individual samples (up to 32 x 1 inch diameter), or single large diameter samples (up to 200 mm, 8 inch diameter). That makes the Agilent Solid Autosampler the ideal upgrade to the Cary 7000 UMS for the optical characterization of large sets (batches) of optical components or the spatial mapping of larger individual samples down to a practical resolution limit of 2 mm x 2 mm.

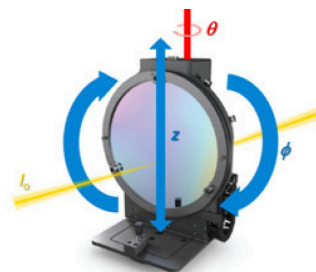


Figure 2. MPS(θI,Φ, z) co-ordinate system of the Agilent Solids Autosampler with respect to incident beam I_o.

Sample holder

The 8-inch round sample holder securely mounts the sample into the autosampler. The sample holder touches the sample only by a 3.0 mm ring around the edges and three (removable) spring-loaded clamps. These clamps minimize sample contact and protect the integrity of the sample. This is of great importance for sensitive end products and coated precursors.

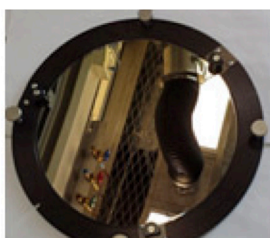


Figure 3. A 200 mm diameter precut wafer mounted in the 8" sample holder that attaches into the Solids Autosampler.

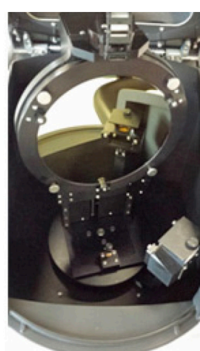


Figure 4. Front view of the Cary 7000 UMA sample compartment with the Agilent Solids Autosampler installed and the 200 mm diameter coated wafer mounted into the autosampler.

Determining the measurement limits

Prior to the mapping experiment, we determined the radial limit of measurement optically, recording transmission signals at distances from 90 to 95 mm from the center position at 1 mm increments (Figure 5). A drop in the transmission signal at 95 mm diameter indicates the limit of measurement where the sample holder inner ring starts to obstruct the measurement beam. The maximum measurement point was set to 94 mm radius for all profiles to avoid beam obstruction. The optical interference coating on the wafer doesn't cover the entire substrate but reaches to about 3 mm to the edge of the wafer (Figure 5).

That means we could confidently measure 94% of the total coated wafer surface using the Solids Autosampler.

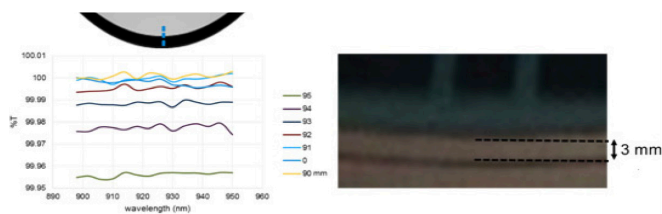


Figure 5. Left: The maximum measurable edge point was confirmed by measuring at 1 mm increments from 90 mm to 95 mm. A signal drop at 95 mm indicates the beam clipping the sample holder edge. Right: The coating reaches 3 mm from the edge of the wafer.

Reflection spectra

Although the Cary 7000 UMS can perform measurements at variable angles of incidence from 5° to 85°, we chose a 7° (near normal). Larger angles will spread the beam patch size on the sample surface, which leads to a reduced spatial resolution during mapping. We set the (adjustable) incident beam cone angles to 3° horizontal and 1° vertical (sample rotation takes place about the vertical axis) and used a 4 nm spectral bandwidth.

These parameters lead to a beam patch of approximately 5.0 mm x 1.5 mm (height x width), which is slightly larger than the step size resolution of the autosampler (0.5° steps vertically and rotationally about the beam axis).

Table 1. Agilent Cary 7000 UMS measurement conditions used to acquire the reflection spectrum presented in Figure 4.

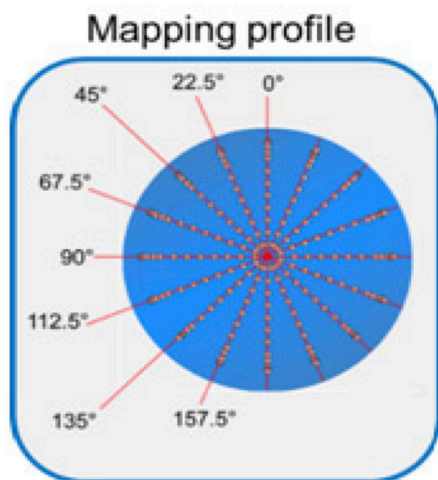
Parameter	Value
Angle of incidence	7°
Wavelength range	2500-250 nm
Data interval	UV-Vis 1.0 nm, NIR 4.0 nm
Spectral bandwidth	UV-Vis 4.0 nm, NIR 4.0 nm
Signal averaging time	0.5 sec
Polarization	s-polarization
Incident beam aperture	3° x 1° (vertical x horizontal)
Baseline correction	100% T

Wafer profiling

For the optical characterization of the coating surface, we configured the autosampler to drive the sample according to the mapping profile given in Table 2. The mapping profile consists of 8 chords through the wafer's diameter with an angular resolution of Φ= 22.5°. Each chord represents 27 spatial points spaced 5 mm apart, plus two smaller step intervals toward the edge at 92 mm and 93 mm. We set the Cary 7000 UMS to perform short wavelength scans over the analytic wavelength at every measurement point. Once prepared, the Cary 7000 UMS and autosampler worked together—fully automated—to collect the desired data set.

Table 2. Mapping profile and collection conditions applied on the wafer profiling experiment.

Tuning Parameter	Value
Angle of incidence	7°
Wavelength range	1065-1063 nm
Data interval	1.0 nm
Spectral bandwidth	4.0 nm
Signal averaging time	1 sec
Detector	silicon photodiode (manually set detector change)
Polarization	s-and p-polarization
Incident beam aperture	3°x1° (vertical x horizontal)
Baseline correction	100% T + 0% T



Results and discussion

Figure 6 shows the s-polarized reflectance spectrum from the UV (250 nm) to the NIR (2500 nm) of the coated wafer. We took the spectrum from a spot in the center of the wafer because that is where the quality of the coating can be expected to be best. The spectrum clearly shows the design intent of the optical interference coating with reflection around 1064 nm exceeding 99% over a narrow 950 nm to 1150 nm band pass.

In the mapping experiment, we used the Cary 7000 UMS and the autosampler to collect the reflection characteristics of the wafer at 1064 nm in s- and p-polarization following the mapping pattern given in Table 2. Figure 7 shows the mapping profiles we obtained with the reflection values at 1064 nm plotted against the distance of the measurement point from the center. These profiles reveal a decrease in reflection from the wafer center toward the edge in both s- and p-polarization. The high similarity and consistency between individual profiles indicate a centrosymmetric optical contour of the wafer. Outliers at 80 mm diameter on the 90° chord and at 85 mm on the 67.5° chord both in Rs and Rp could be directly attributed to contamination on the wafer surface by subsequent visual inspection.

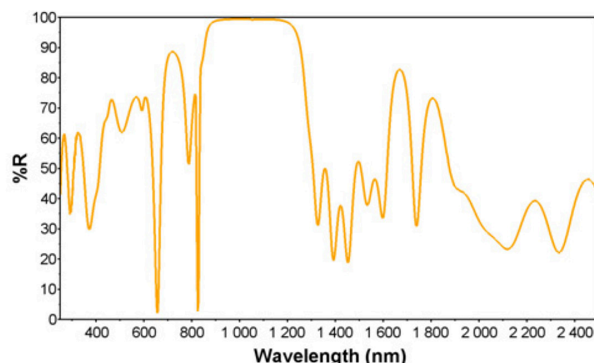


Figure 6. The Rs spectrum of wafer center at 7° angle of incidence.

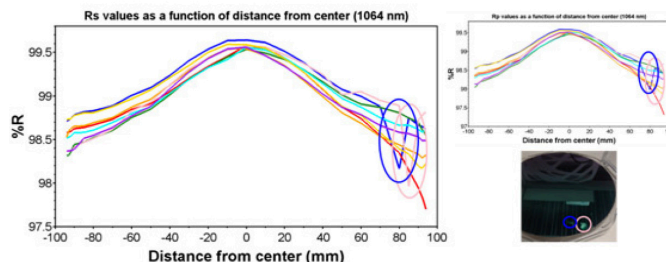


Figure 7. Mapping profiles (%Rs: left and %Rp: right, top) at 1064 nm.

The center position of the wafer was measured repeatedly as part of each chord, and this data was used to estimate reproducibility of the measurement (Figure 8). A reproducibility of <0.1% was achieved over the time course of the mapping experiment (~6.5 h), which is about 10 times better than the %R differences found between points at the center and at the edges (up to 1%). That not only demonstrates the long-time stability of the Cary 7000 UMS but also underlines the significance of the presented profiles.

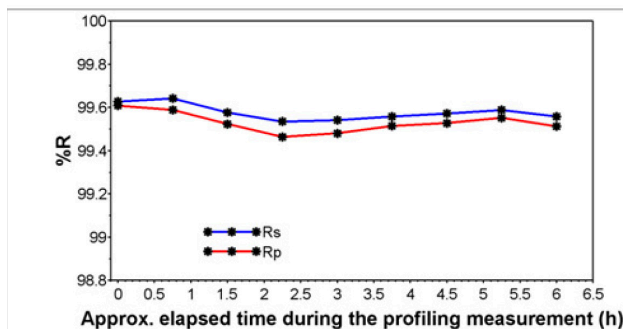


Figure 8. Variability of the system during the time course of the mapping experiment.

Conclusions

In this study, we used the Agilent Cary 7000 UMS with Solids Autosampler to successfully analyze the coating uniformity across a 200 mm diameter precut wafer.

The system was setup to execute %R measurements centered around 1064 nm in a user-defined pattern across the wafer's surface. The resulting profiles revealed a decrease in coating quality toward the wafer's edges. This knowledge could be used to find and overcome the underlying variability in the coating process.

This mapping analysis could be used for quality control or it could be implemented in development processes to maximize yields and minimize waste and investment.

The true-to-life example used for this application note serves to prove the extensive capabilities of the Cary 7000 and Solids Autosampler for automated routine multi-angle spectroscopic characterizations of optical materials, coatings, and components in a wide range of industrial and laboratory applications.

References

1. Burt, T.; Haq, F. High volume optical component testing using Agilent Cary 7000 Universal Measurement Spectrophotometer (UMS) with Solids Autosampler, Agilent Technologies, publication number 5991-4071EN, 2005.
2. Burt, T.; Haq, F. Coated wafer mapping using an Agilent Cary 7000 Universal Measurement Spectrophotometer (UMS) with Solids Autosampler. Agilent Technologies, publication number 5991-4072EN, 2014.
3. Death, D.L.; Francis, R.J.; Bricker, C.; Burt, T.; Colley, C. The UMA: A new tool for Multi-angle Photometric Spectroscopy. Optical Interference Coatings (OIC) OSA Topical Meeting, Canada, 2013.
4. Tikhonravov, A.V.; Amotchkina, T.V.; Trubetskov, M.K.; Francis, R.J.; Janicki, V.; Sancho-Parramon, J.; Zorc, H; Pervak, V. Optical characterization and reverse engineering based on multiangle spectroscopy. *Appl. Opt.* **2012**, 51, 245-254.
5. Amotchkina, T.V.; Trubetskov, M.K.; Tikhonravov, A.V.; Janicki, V. J.; Sancho-Parramon; Razskazovskaya, O.; Pervak, V. Oscillations in the spectral behavior of total losses (1 – T – R) in the dielectric films. *Opt. Exp.* **2012**, 20, 16129-16144.
6. Amotchkina, T.V.; Trubetskov, M.K.; Tikhonravov, A.V.; Schlichting, S.; Ehlers, H.; Ristau, D.; Death, D.; Francis, J.J.; Pervak, V. Quality control of oblique incidence optical coatings based on normal incidence measurement data. *Opt. Exp.* **2013**, 21, 21508-21522.

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