Gaining deeper insights into thin film response — overcoming spectral oscillations using the Cary Universal Measurement Accessory

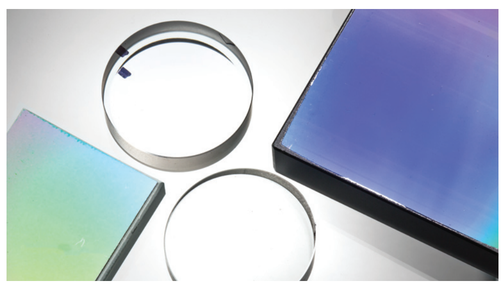
Application note

Materials

Authors

Robert Francis and Travis Burt

Agilent Technologies
Mulgrave, Victoria, Australia



Introduction

A more detailed account of this work was first published in Optics Express 16129, 2 July 2012, Vol. 20, No. 14 [1].

Designers and manufacturers of high quality multilayer optical coatings require reliable methods to measure optical constants of thin film materials with a high degree of accuracy. This is normally achieved using UV-Visible-IR spectrophotometry to acquire normal and quasi-normal transmittance (T) and reflectance (R) spectra of a sample. Understanding the accuracy of the data produced and the source of any errors (random or systematic) will lead to more reliable sample characterization [2, 3].

Random errors (random noise) vary from one point to another in the measurement data set, and it has been shown in [2] to have minimal impact on the characterization results. However, systematic errors which result in an offset of spectral characteristics as a whole, or cause large-scale wavelength variations of T and R curves, are especially critical for the accurate determination of thin film parameters [2].

Valuable information relating to data accuracy is provided by calculating the total losses (TL) of the thin film sample [4, 5] using $TL(\lambda) = 100\% - R(\lambda) - T(\lambda)$. Typically, in the spectral range where the substrate and the thin film are non-absorbing and non-scattering, zero total losses would be expected, whereas with absorbing films, $TL(\lambda)$ decreases with increasing λ.

When analyzing spectra for TL, researchers often observe oscillations, which may cause doubt about the quality of the data. Sources of such oscillations include:

- The difference in angles of incidence (AOI) at which T and R are measured
- Absorption in a thin film acting in combination with interference effects
- A slight thickness non-uniformity of the film

A full report on the origins of oscillations in TL is discussed in [1]. In this paper we demonstrate how an Agilent Cary 5000 UV-Vis-NIR spectrophotometer equipped with a new Universal Measurement Accessory (UMA) is able to provide previously unreported insights into thin film characterization due to its ability to measure T and R without moving the sample.

Experimental

Samples

A Ta_2O_5 film of 292 nm thickness was deposited onto a Suprasil substrate of 25 mm diameter and 6.35 mm thickness using a magnetron-sputtering Leybold Optics HELIOS plant [1]. A second, pre-prepared Ta_2O_5 sample with a slightly different film thickness was also used. Transmittance data for s-polarized light was measured at 7° and 10°, and s-polarized reflectance data was measured at 10°.

Instrumentation

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

The UMA is a highly automated variable angle absolute specular reflectance and transmittance system. The linearly polarized beam that illuminates the sample can be measured in transmission, and by rotating the detector assembly about an axis through the sample and perpendicular to the plane of incidence, in reflection, as indicated in Figure 1. Thus the same spot on the sample is used for both T and R measurements. This multiple measurement mode capability of the UMA results in more accurate, rapid and complete optical characterization of thin films. This data could have also been collected on a Cary 7000 Universal Measurement Spectrophotometer (UMS).

Results and discussion

Traditionally, reflectance and transmittance measurements have been performed using spectrophotometers fitted with different accessory attachments. In practice, different areas of the sample surface may be tested. If the deposition process produces a film with a non-uniform thickness, it is reasonable to expect that reflectance and transmittance measurements are affected.

With the development of the UMA, it is now possible to measure T and R at the same sample point, overcoming one source of oscillations on the results. In this study, an Agilent Cary 5000 double beam UV-Vis-NIR spectrophotometer equipped with a UMA was used to acquire transmittance data for s-polarized light at 7° and 10°, and s-polarized reflectance data at 10°. In order to verify the capabilities of the new UMA, the same sample was reanalyzed a few months later using a second UMA unit and different sample mounts.

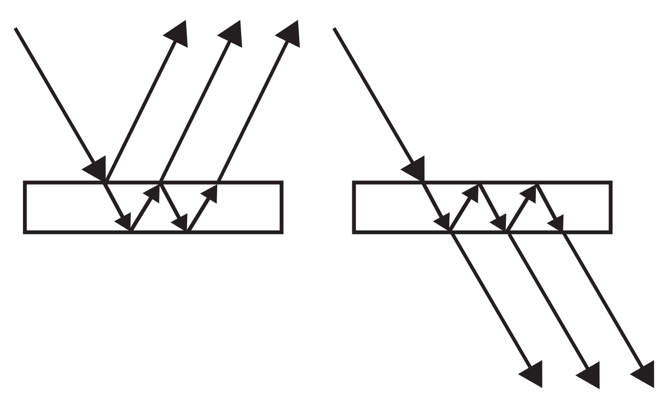


Figure 1. Schematic of the Agilent UMA, an absolute variable angle reflectance and transmission accessory capable of front and back surface reflection (left) and direct and inter-reflected transmission (right) measurement.

In the first experiment, we consider TL calculated on the basis of T and R measurements taken at different AOI using the first UMA unit according to: $TL^{(s)}(\lambda) = 100\% - T^{(s)}(7°, \lambda) - R^{(s)}(10°, \lambda)$. The experimental total losses are plotted in Figure 2(a) (red line). Oscillations of approximately 0.4% magnitude are clearly observed. The solid curve (black line) in Figure 2(a) presents a theoretical approximation of total losses as expected when T and R are measured at different angles of 7 and 10 degrees respectively. There is good agreement between the experimental and theoretical results shown in Figure 2(a). This agreement confirms that the oscillations are due to only the difference in AOI and that the effect of thickness non-uniformity does not contribute to total losses spectral behavior.

Secondly, in Figure 2(b), we show experimental total losses, calculated from T and R measurements taken at equal AOI: $TL^{(s)}(\lambda) = 100\% - T^{(s)}(7°, \lambda) - R^{(s)}(7°, \lambda)$. The absence of any oscillations is clearly observed. This is further confirmation that thickness non-uniformity does not affect total losses when T and R measurements are acquired at the same point of the sample.

In the second experiment, we acquired T and R data from the original sample on a new sample mount using a different UMA unit coupled to the Cary 5000 spectrophotometer. The solid black curve in Figure 3(a) represents the data acquired using a UMA unit. The same sample was re-analyzed using a different UMA unit four months later and the results are represented by the red line. Visually, both data sets are indistinguishable, demonstrating the excellent reproducibility between multiple UMA units. This gives confidence in results obtained from different UMA/UMS systems, for example, within a manufacturing environment where more than one instrument can be used.

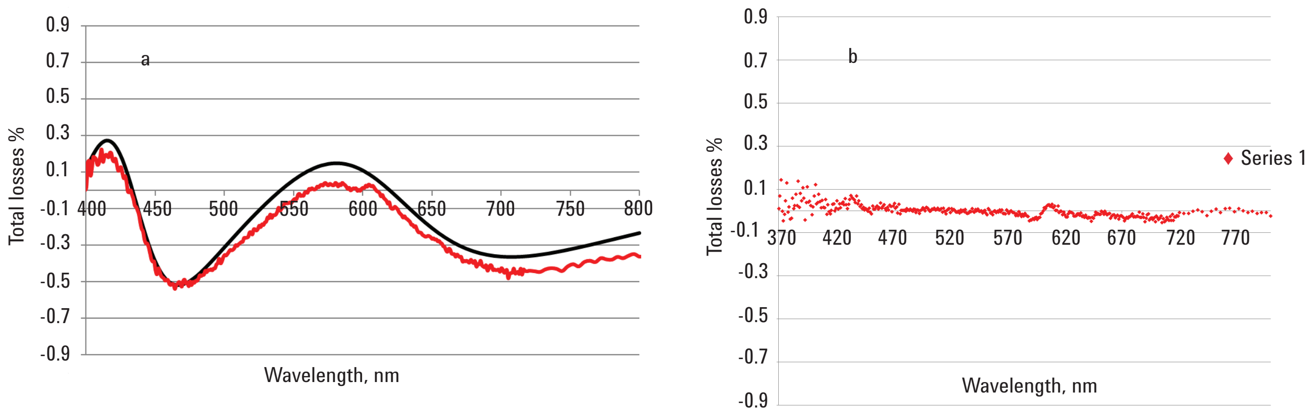


Figure 2. (a) Comparison of $TL^{(s)}(\lambda) = 100\% - T^{(s)}(7°, \lambda) - R^{(s)}(10°, \lambda)$ calculated from experimental data and $TL^{(s)}AOI(\lambda)$ calculated by theoretical equation [1]. (b) Total losses $TL^{(s)}(\lambda) = 100\% - T^{(s)}(7°, \lambda) - R^{(s)}(7°, \lambda)$ calculated from experimental data.

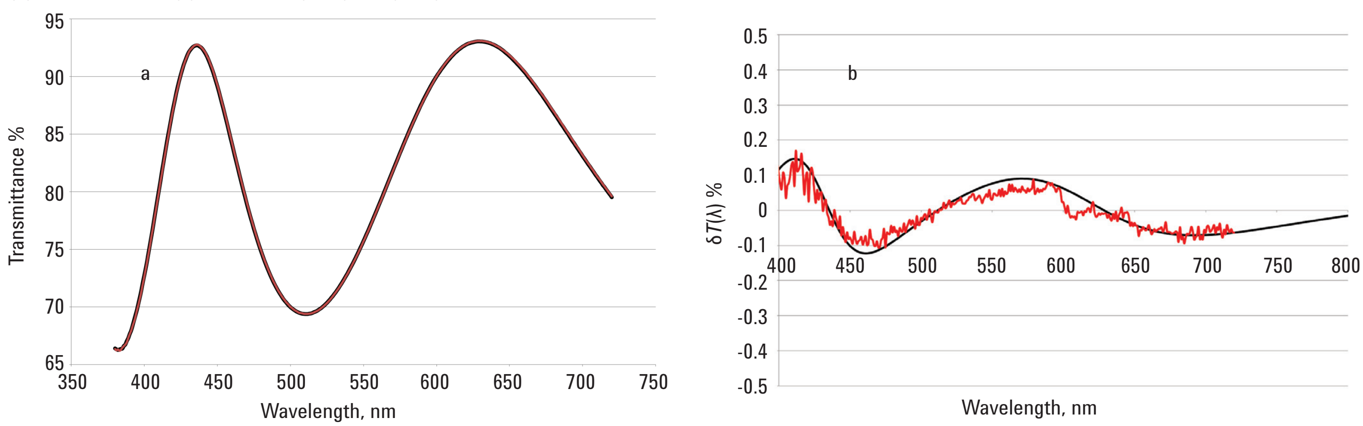


Figure 3. (a) Comparison of two transmittance spectra acquired using two different UMA accessories coupled to a Cary 5000 spectrophotometer. The data relates to the same Ta_2O_5 sample. (b) Comparison of $T^{(s)}(\lambda) = T^{(s,1)}(7°, \lambda) - T^{(s,2)}(7°, \lambda)$ calculated from experimental data (red line) and by theoretical equations (black line) [1].

The difference between the two transmittance spectra is indicated by the red line in Figure 3(b). Oscillations of about 0.15% magnitude can be clearly observed. The solid curve (black line) in Figure 3(b) indicates a theoretical approximation of total losses calculated with Δd = -0.3 nm. This thickness non-uniformity value corresponds to about 0.1% of the geometrical film thickness (d = 292 nm). This is in full agreement with the thickness non-uniformity level in the HELIOS deposition plant.

Conclusions

One cause of oscillations in total losses spectra arise from slight thickness non-uniformity of the thin film sample. However this source of error can be small when compared to variations in AOI between T and R measurements. AOI errors can be eliminated using an advanced spectrophotometric accessory (developed by Agilent Technologies) fitted to a Cary 5000 UV-Vis-NIR spectrophotometer. The UMA is a variable angle specular reflectance and transmittance system that acquires T and R data without moving the sample or the incident beam onto the sample.

The results presented in this report demonstrate that the residual oscillations observed after eliminating AOI variations are minimal and are in full agreement with the theoretical values. The findings confirm that the effect of thickness non-uniformity does not contribute to total losses spectral behavior when an instrument capable of T and R measurement at the same point of the investigated sample is used. The latest in multiangle spectral photometry provides researchers with more accurate spectroscopic information for the characterization of thin films than has been previously available.

References

1. Tatiana V. Amotchkina, Michael K. Trubetskov, Alexander V. Tikhonravov, Vesna Janicki, Jordi Sancho-Parramon, Olga Razskazovskaya, and Vladimir Pervak, “ Oscillations in spectral behavior of total losses (1−R−T) in thin dielectric films,” *Optics Express* 16129, 2 July 2012 / Vol. 20, No. 14

2. A. V. Tikhonravov, M. K. Trubetskov, M. A. Kokarev, T. V. Amotchkina, A. Duparre, E. Quesnel, D. Ristau, and S. Gunster, “Effect of systematic errors in spectral photometric data on the accuracy of determination of optical parameters of dielectric thin films,” *Appl. Opt.* 41, 2555–2560 (2002).

3. J. Woollam, “Ellipsometry, variable angle spectroscopic,” *Wiley Encyclopedia of Electrical and Electronics Engineering*, J. Webster, ed. (Wiley, New York, 2000). Supplement 1.

4. A. V. Tikhonravov, M. K. Trubetskov, T. V. Amotchkina, G. DeBell, V. Pervak, A. K. Sytchkova, M. L. Grilli, and D. Ristau, “Optical parameters of oxide films typically used in optical coating production,” *Appl. Opt.* 50, C75–C85 (2011).

5. A. Tikhonravov, M. Trubetskov, T. Amotchkina, A. Tikhonravov, D. Ristau, and S. Gunster, “Reliable determination of wavelength dependence of thin film refractive index,” *Proc. SPIE* 5188, 331–342 (2003).

www.agilent.com/chem

Published June 3, 2013
Publication number: 5991-2111EN

Agilent Technologies