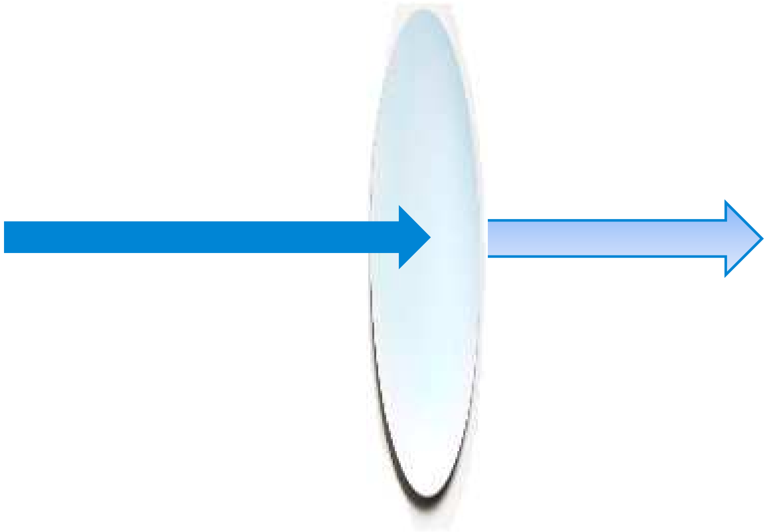
Agilent Cary 7000 Universal Measurement Spectrophotometer

Thin Film Coatings

Rob Wills – Agilent UK

Agilent Technologies

UV-Vis-NIR Spectrophotometry



%T and %R
Angular Control
Flexibility

Cost per analysis
Automation
Productivity

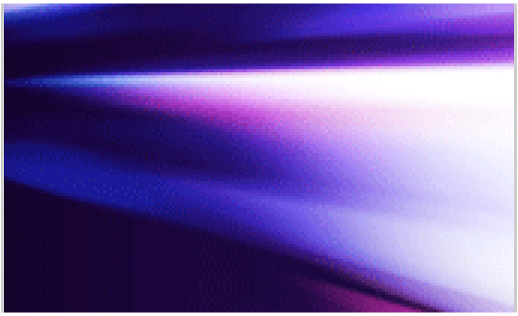
Consistency
(%T and %R)
Accuracy
Performance

Easy of Use
Accessible
Reliability

Research

QA/QC Testing

Trouble Shooting



Cary 7000 UMS Weblinks

Website – free downloads (brochure, app notes, technical notes)

http://www.chem.agilent.com/en-US/products-services/Instruments-Systems/Molecular-Spectroscopy/Cary-7000-Universal-Measurement-Spectrophotometer-(UMS)/Pages/default.aspx

Video

http://www.chem.agilent.com/en-US/products-services/Instruments-Systems/Molecular-Spectroscopy/Pages/cary_ums_w2_video.aspx

Interactive Brochure

http://www.chem.agilent.com/en-US/products-services/Instruments-Systems/Interactive-Brochures/Pages/5991-2392EN.aspx

Cary 7000 UMS Summary

1 *Absolute specular reflection*

2 *Diffuse scattering*

3 *Glossy scattering*

4 *Direct transmission*

5 *Scattered transmission*

6 *Partial scattered transmsision*

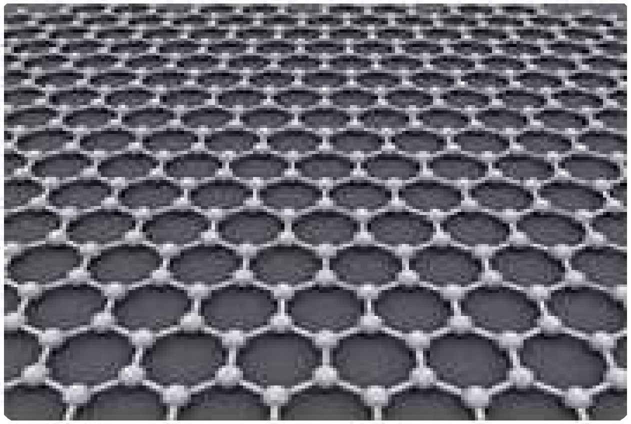
6
Modes

1
System

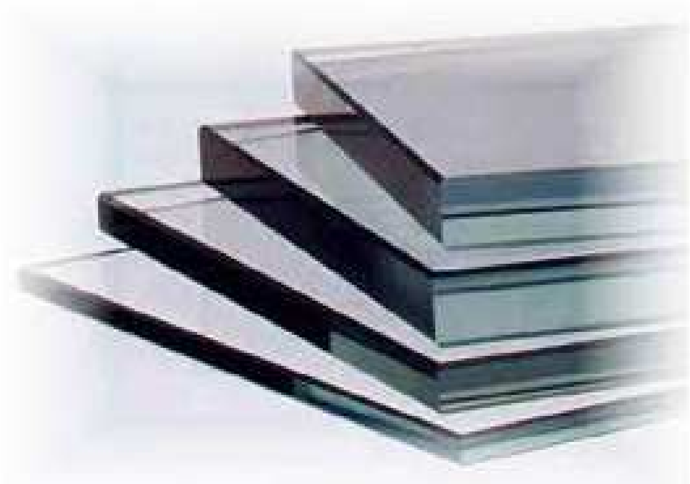
Perform all these measurements on the Cary 7000 UMS

Key Markets

Optics/Coatings/Thin Films



Academic/Industrial Research



Glass

Solar

Measurement Challenge – Thin Film Analysis

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.

Thin film coatings are designed based on interference effects, and information relating to data accuracy is provided by calculating the Total Losses (TL) of a film sample, so if you want to keep these Total Losses to a minimum it's important to understand what's causing them.

The following slides show a very brief summary of work that was done on the UMS during the development cycle of the product by the group of **Alexander V. Tikhonravov** and **Michael K. Trubetskov** at **Moscow State University** (www.optilayer.com)

Total Losses (TL)

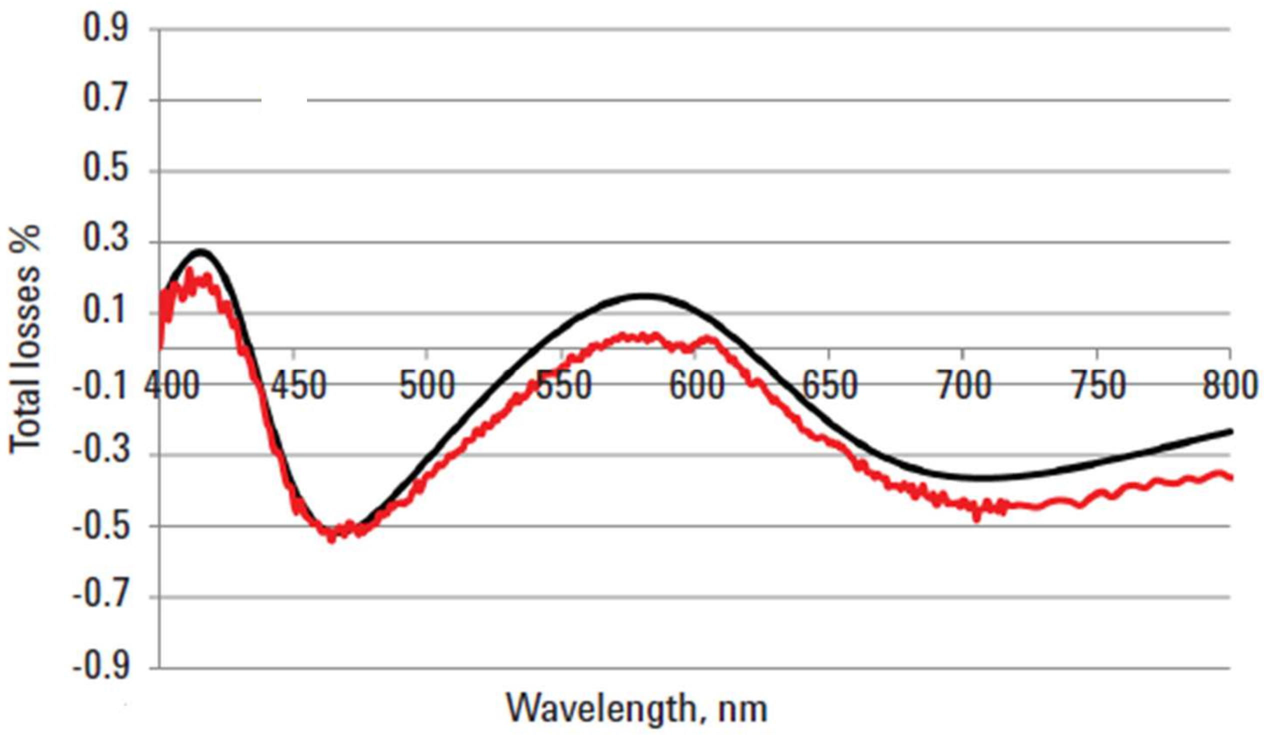
Valuable information relating to data accuracy is provided by calculating the total losses (TL) of the thin film sample using $TL(\lambda) = 100\% - R(\lambda) - T(\lambda)$.

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

TL Calculated at Different AOI

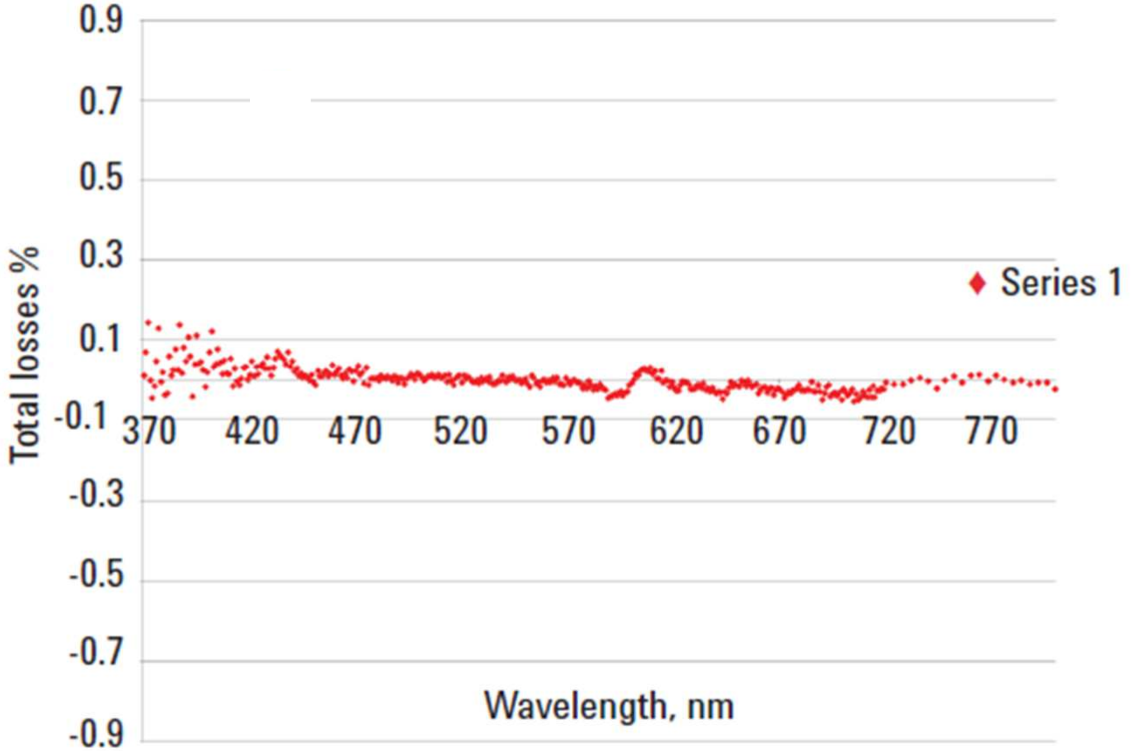


Comparison of TL$^{(s)}$(λ) = 100%−T$^{(s)}$(7°, λ)− R$^{(s)}$ (10°, λ) calculated from experimental data and TL$^{(s)}$AOI(λ) calculated by theoretical equation

Graph shows plot of TL calculated from real measurements made at different AOI (red trace) overlaid against theoretical, calculated values (black trace).

Overall the measured and theoretical show good agreement, indicating that the source of TL stems from the difference in measurement AOI and that the effect of thickness non-uniformity does not contribute in this case.

TL Calculated at Same AOI



Total losses $TL^{(s)}(\lambda) = 100\% - T^{(s)}(7°, \lambda) - R^{(s)}(7°, \lambda)$ calculated from experimental data

When TL are calculated using the same AOI the result is a flat line with no oscillations, confirming that it’s the difference in measurement AOI that is the single biggest contributor to Total Losses.

This measurement is possible on the Cary UMS because the transmission and reflectance measurements are made at the exact same spot on the sample.

Measurement of 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.

The focus of the group at MSU is the production of attosecond light pulses. This requires "compression" of light using complex, multi-layer, high reflecting mirrors.
They have their own facility for producing these coated mirrors.

Oblique-Incidence T and R Data

Parameters of Ta_2O_5 and SiO_2 films found by using oblique-incidence T and R data acquired using the Cary UMS

	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	402.7	1.483

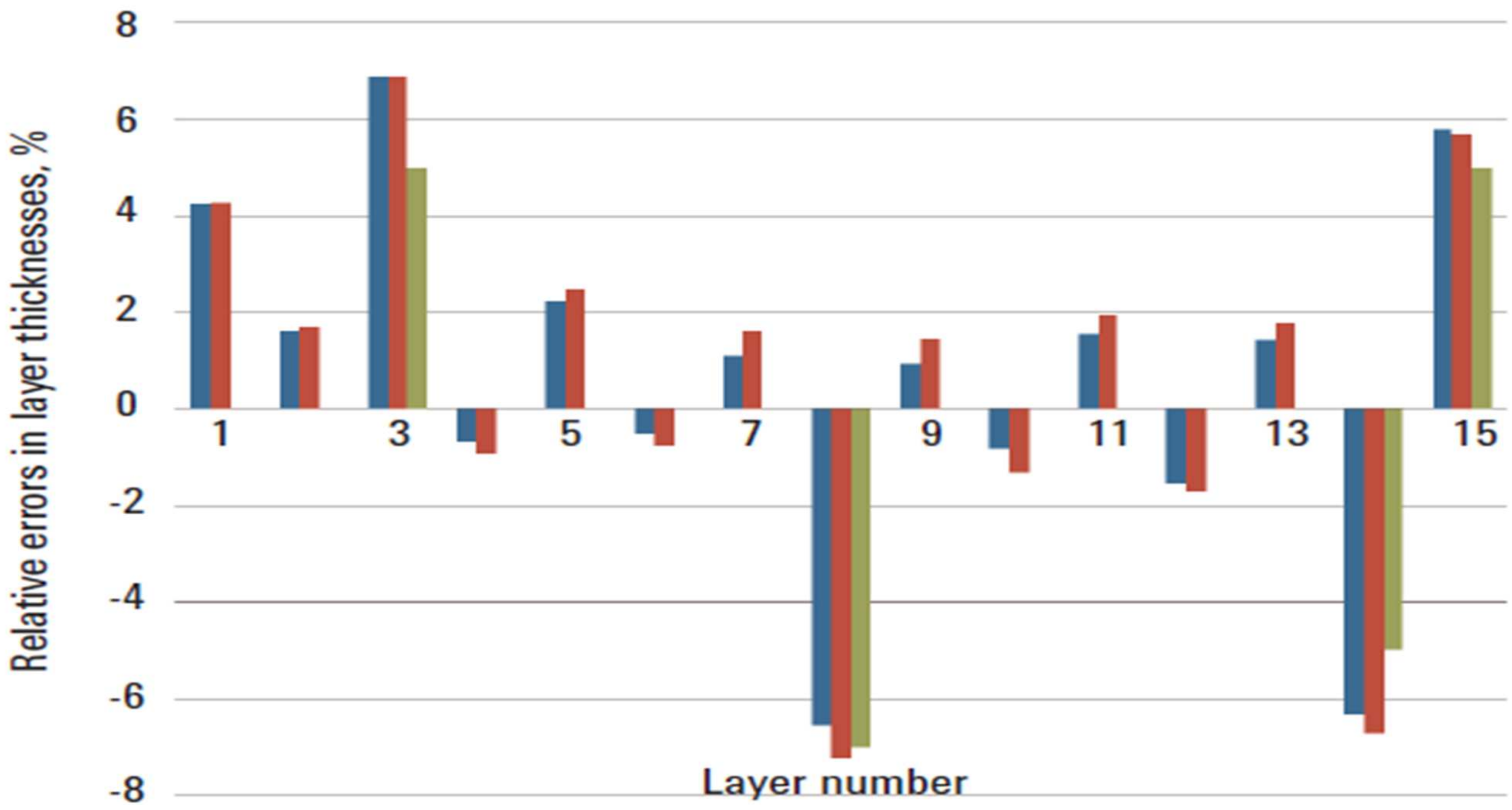
Reverse Engineering Based on Multi-Angle

To check the reliability of reverse engineering based on multi-angle optical photometric data, MSU 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 3rd , 8th, 14th, and 15th mirror layers, respectively.

Various combinations of input measurement data was acquired using the UMS and the intentional thickness errors were reliably detected in all cases.

Results



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.

"I can recommend this product to all researchers…"

" I can recommend this product to all researchers and experimenters who are in a need of high precision and consistent multi-angle spectrophotometric data."

"This is an invaluable tool for all labs and companies producing multilayer optical coatings for different applications, especially high-demanding coating consisting of many dozens of layers."

"I was particularly impressed by the fact that even for large incident angles, multi-angle data is still consistent. Agilent was able to resolve very difficult problems of calibrations and collecting all light in the detector."

Dr.Michael K. Trubetskov

Leading Research Fellow for the Research Computing Center.

Moscow State University

Peer Reviewed Publications on UMS/UMA

- **Optical characterization and reverse engineering based on multiangle spectroscopy**
  Alexander V. Tikhonravov et al, 10 January 2012 / Vol. 51, No. 2 / APPLIED OPTICS 245
  http://www.opticsinfobase.org/ao/abstract.cfm?uri=ao-51-2-245
- **Oscillations in spectral behavior of total losses (1 − R − T) in thin dielectric films**
  Tatiana V. Amotchkina et al, Optics Express, Vol. 20, Issue 14, pp. 16129-16144 (2012)
  http://www.opticsinfobase.org/oe/fulltext.cfm?uri=oe-20-14-16129&id=239460
- **Quality control of oblique incidence optical coatings based on normal incidence measurement data**
  Tatiana V. Amotchkina, Michael K. Trubetskov, Alexander V. Tikhonravov, Sebastian Schlichting, Henrik Ehlers, Detlev Ristau, David Death, Robert J. Francis, and Vladimir Pervak, **Optics Express, Vol. 21, Issue 18, pp. 21508-21522 (2013),**
  http://dx.doi.org/10.1364/OE.21.021508

Questions…

If you have any questions, or requests, please feel free to contact me….

Rob Wills

Agilent Technologies UK Ltd

robert.wills@agilent.com

Tel : 07826 859 488