

Coated wafer mapping using an Agilent Cary 7000 Universal Measurement Spectrophotometer (UMS) with Solids Autosampler

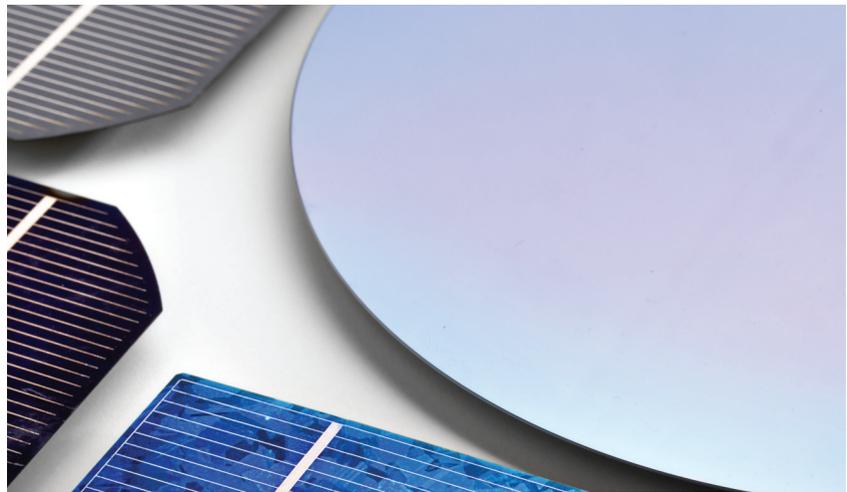
Application note

Materials testing and research

Authors

Travis Burt, Farinaz Haq

Agilent Technologies, Australia



Introduction

Spectral reflection (R) and transmission (T) are the fundamental measurements for characterizing the optical properties of materials and optical coatings. Mutliangle Photometric Spectroscopy (MPS) measures the reflectance and/or transmittance of a sample across a range of angles (θ_i) from near normal to oblique angles of incidence (AOI). A recent development in the field of MPS by Agilent Technologies, the Cary 7000 Universal Measurement Spectrophotometer (UMS), combines both reflection and transmission measurements from the same point on a sample's surface, without moving the sample between measurements. Alleviating the need for multiple accessories and accessory changeover or reconfiguration ensures unprecedented data quality and prevents sample non-uniformity effects or spectral inconsistencies that occur when multiple analysis techniques are used to perform a single measurement.



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In this paper we describe a new autosampler capability for the Agilent Cary 7000 UMS with rotational (Φ) and radial (z) sample positioning control. The Agilent Solids Autosampler allows for the automated and unattended mapping of single large diameter samples (up to 8 in diameter). Examples are provided which demonstrate spatial spectroscopic information obtained on a Zinc Tin Oxide (ZTO) layer deposited onto a 4 in diameter sapphire substrate to a resolution of 2 mm x 2 mm square. The method allows the band gap energy to be mapped across the diameter of the substrate.

Experimental

Instrumentation

- Agilent Cary 7000 Universal Measurement Spectrophotometer
- Agilent Solids Autosampler

The Cary 7000 UMS is a versatile new system designed for MPS applications over the UV-Vis-NIR 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 Cary 7000 UMS combines

both reflection and transmission measurements from the same patch of a sample's surface in a single automated platform for angles of incidence in the range $5^\circ \leq |\theta_i| \leq 85^\circ$. The Cary 7000 UMS also provides 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.

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 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_0). A variety of sample holders allow mounting of multiple individual samples (up to 32 x 1 in diameter) or single large diameter samples (8 in diameter). Figure 1b shows the 8 in diameter



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



Figure 1b. Image of the 8 in diameter sample holder

sample holder. The autosampler was operated in spatial mapping mode for the large sample characterization study of ZTO. In the mapping mode spectra can be collected at user defined points within the sample holder.

Mapping analysis

The optical or electronic band gap properties of semiconductors are core to their effectiveness in end-use devices such as sensors (e.g. detectors) or emitters (e.g., LED's). The band gap energy of a semiconductor can be determined from diffuse reflection spectra when the material is in a powdered form [2] or from transmission spectra where an epitaxial semiconductor layer has been deposited on a transparent substrate.

In this experiment, the band gap energy was mapped across the diameter of a wafer coated with a ternary metal oxide by acquiring transmission spectra using a Cary 7000 UMS with Solids Autosampler. Transmission spectra were collected from 700 nm (1.7 eV) to 200 nm (7.8 eV) with a 4 nm spectral band width and 0.1 s signal averaging time. UV-Vis spectral data was acquired at 5 mm intervals from the bottom of the wafer (-40 mm) to the top (45 mm) as indicated in Figure 2b.



Figure 2a. 100 mm (4 in) diameter sapphire substrate coated with Zn/Sn

Samples

The sample was comprised of a graded Zinc Tin Oxide (ZTO) layer deposited onto a 100 mm (4 in) diameter sapphire substrate thickness = 600 μm , Figure 2a. Zinc and tin metal targets were sputtered in an oxygen ambient atmosphere from opposite ends of the wafer simultaneously by high power impulse magnetron sputtering (HiPIMS) and direct current magnetron sputtering (DCMS) respectively producing a coating of approx 14 nm (140 \AA). The epitaxial layer formed a graded ZTO layer stretching from almost pure tin at the bottom of the wafer to almost pure zinc the top as indicated in Figure 2b.

ZnO has a large direct band gap of ~ 3.4 eV at room temperature where tin has an optical band gap of 3.6 eV as SnO_2 [3]. The optical band gap of the amorphous phase of ZTO has been reported as low as ~ 2.8 eV [4], which is of interest as it may be a suitable replacement for the widely used but relatively expensive Indium Tin Oxide (ITO) in a range of applications including organic photovoltaics and flexible displays.

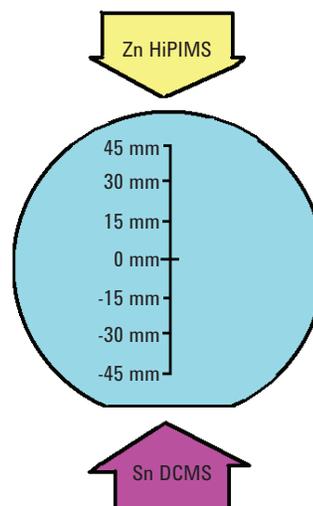


Figure 2b. Schematic of deposition direction and coordinate system with respect to wafer orientation. Tin (Sn) was deposited by DCMS and Zinc (Zn) by HiPIMS. Spectral measurements were made at 5 mm intervals from -40 mm to 45 mm.

Results and Discussion

Transmission spectra

Transmission spectra were collected at 18 evenly spaced (~5 mm) intervals through the diameter of the wafer from bottom to top position. The absorption edge of the transmission spectra (Figure 3a) can be seen to shift to lower frequencies at the top of the wafer where the Zn concentration is highest. Band gap was determined by extrapolating a linear fit through the absorption edge to zero absorption in a plot of $(\text{Absorption})^2$ vs eV. The intercept in eV at zero Abs was taken as the band gap energy. Figure 3b shows band gap energies determined through the diameter of the wafer. This profile can help to extract smaller samples from the wafer with very specific, targeted, band gap energies without the need to have precise control of the coating conditions.

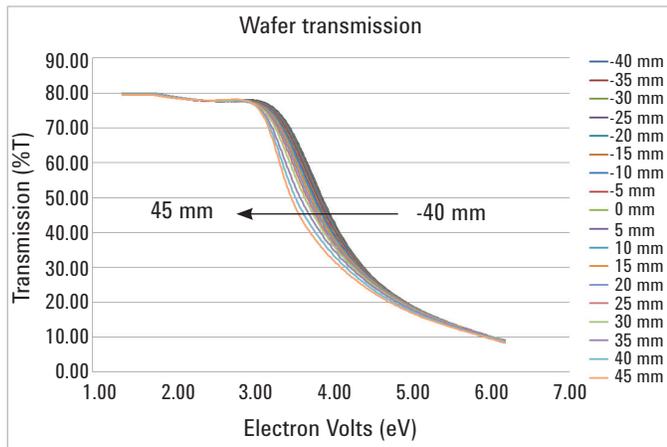


Figure 3a. Transmission spectra through wafer at 11 positions through its diameter as indicated in Figure 4b

Conclusion

The Agilent Cary 7000 UMS with Solids Autosampler has been used successfully for the large sample characterization of thin-film substrates. The autosampler was operated in spatial mapping mode for study of ZTO. The band gap energy of the ZTO substrate was mapped across the diameter of the wafer through the acquisition of transmission spectra. The data showed some variation; for example lower frequencies were observed at the top of the wafer where, as a result of the deposition process, the Zn concentration was highest.

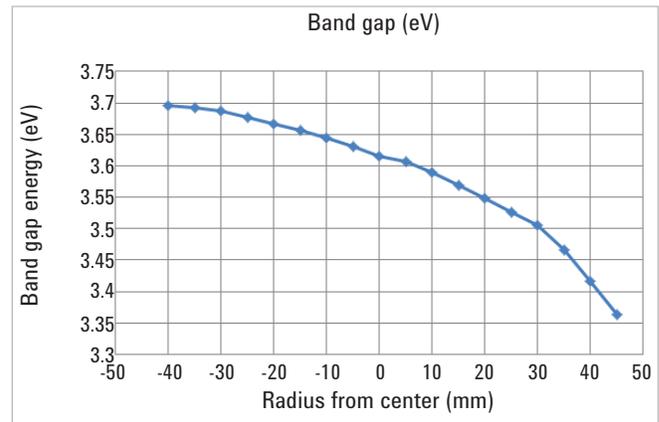


Figure 3b. Band gap was determined from the intercept of the extrapolation of absorption edge, $(\text{Absorption})^2$ vs Energy (eV), to zero absorption

As suitable replacements for relatively expensive substrates such as Indium Tin Oxide (ITO) are being sought, materials with similar optical band gap energies can be characterized using this method.

It is expected that the Cary 7000 UMS and Solids Autosampler will prove to be a valuable tool for the characterization of optical materials, coatings and components in a wide range of industrial and laboratory applications.

Acknowledgements

The authors gratefully acknowledge RMIT University for providing samples and assisting with data interpretation.

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Published February 28, 2014

Publication number: 5991-4072EN



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