Emerging applications of Spectroscopic Ellipsometry

Spectroscopic Ellipsometry (SE) is routinely used to measure optical coatings. Improvements in accuracy, speed, and spectral coverage have increased its utility. Emerging areas utilize the versatility and functionality of SE for single- and multi-layer stacks.

Spectroscopic Ellipsometry (*SE*) is a non-destructive technique routinely used to measure refractive index and film thickness for optical coatings. It is similar to spectrophotometric (*R*/*T*) measurements in that both measure the properties of light upon reflection or transmission from a coating or stack. Whereas *R*/*T* measurements collect the light *intensity*, ellipsometry measures the *polarization* change. While less intuitive than intensity, the change in polarization measured by ellipsometry has a few distinct advantages:

- polarization contains more information than intensity – both amplitude and phase;
- ellipsometry is more sensitive to material details such as index gradients, surface and interfacial regions;
- phase provides enhanced sensitivity to ultra-thin layers less than 10nm thick; and
- polarization is easier to measure with accuracy and precision.

REFRACTIVE INDEX AND COATING THICKNESS

The most common SE application is the accurate measurement of thin films to determine thickness and refractive index. The measurement will collect reflections from the coating *surface* along with light that has travelled through the film and reflects from the coating-substrate *interface*.

The two light beams recombine to produce a change in total polarization. Film thickness and refractive index can be determined from this change. In Spectroscopic Ellipsometry the polarization change is measured at many wavelengths, providing a “spectrum” of data. As the wavelength varies, so does the interaction between surface and interface light beams, causing periodic “interference” oscillations in the ellipsometric (polarization) spectrum. The shape and frequency of the interference pattern contains information about refractive index, film thickness and additional details of the film microstructure. Additional information can be gathered by varying the angle of incidence, since this changes the path length of the light travelling through the various films. *SE* is commonly used to measure a wide variety of optical coatings; including fluorides, oxides, nitrides, carbides, and more.

A common example using SE is for the accurate measurement of Indium Tin Oxide (ITO) on glass. Figure 1 shows a data spectrum collected with an SE system from the ultraviolet (UV) to the near infrared (NIR). ITO coatings are interesting over this range, as they change from dielectric-behaviour to metallic-behaviour. In the visible, the ITO film is transparent and the data spectrum is dominated by interference oscillations. However, these oscillations stop at longer NIR wavelengths when the ITO coating becomes absorbing. In this region, the light is absorbed by free-carriers. The free-carrier absorption in NIR region can be used to optically measure the ITO resistivity. Another interesting feature of ITO films is that due to processing, it is rare for the free-carrier absorption to be constant through the film. Thus, to properly describe the shape of the data spectrum requires a resistivity gradient. Figure 1 also shows the measured optical constants for the ITO layer at top and bottom of the film, showing the large variation in NIR absorption, which also couples into the index shape.

EMERGING AREA

Combined measurements

Although *SE* and *R/T* measurements contain similar information, they can be complementary. The combination of *SE* and Transmission Intensity (*%T*) has been used with semi-absorbing coatings. The *SE* measurement provides sensitivity to thickness and index, while benefiting from the sensitivity of *%T* to low amounts of absorption. Recently, variable angle *SE* measurements have been complemented by variable angle *%T* to measure anisotropic colour filter layers. More advanced data combinations involve *SE* measurements taken in reflection from front and back surfaces along with *SE* and *%T* transmitted through the sample. This wealth of information is necessary to characterize complex layers, such as phase-shifting photomasks – where the coating is both absorbing and intentionally graded to provide the optimum performance. Due to the similarities between *SE* and *R/T* measurements, they can often be measured with the same instrument. Figure 2 shows a Variable Angle Spectroscopic Ellipsometer configured for both reflected and transmitted measurements over a wide range of wavelengths and angles.

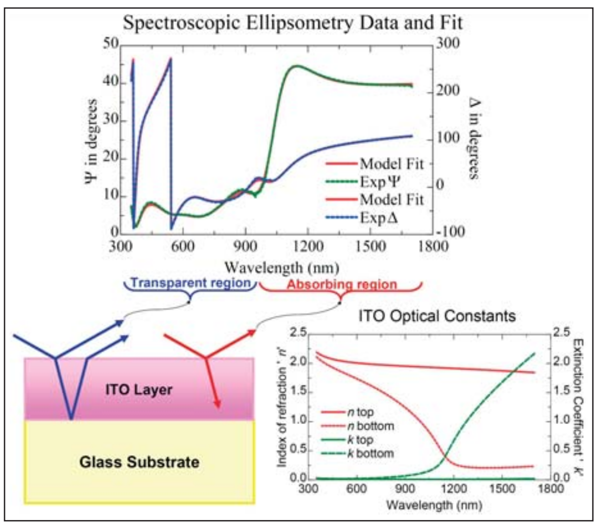


Fig. 1 - *SE* Measurement of ITO coating on glass provides thickness information from the transparent region and resistivity information from the absorbing region. The resistivity of the ITO layer was also found to vary through the film depth as shown by the optical constants at surface (top) compared to substrate interface (bottom)

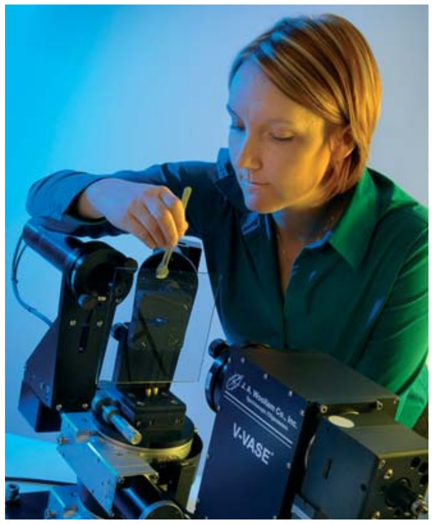


Fig. 2 - Research *SE* system with variable angle allows measurements of reflected or transmitted beam

Anisotropic materials

Plastic substrates, such as PET and PEN are becoming increasingly popular. Optical characterization of these materials is complicated by the substrate anisotropy (nx ≠ ny ≠ nz). The direction-dependent optical properties can introduce data oscillations from interference between the ordinary and extraordinary light rays that travel different paths through the substrate. Unfortunately, these oscillations may hide the optical coating information. The easy solution is to eliminate the substrate anisotropy interference by collecting only the light reflecting from the coated surface. This can be accomplished by: 1) spatially separating the front and backside beams, which would require a very small spot given the typical plastic substrate thickness, or 2) suppressing the back-

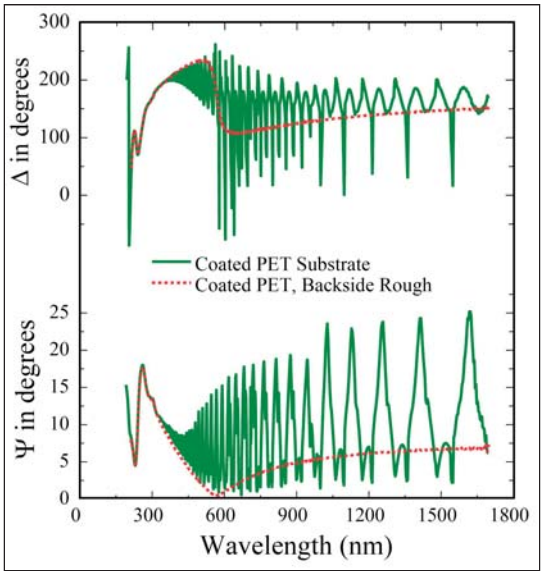


Fig. 3 - Comparison of two ellipsometry measurements of coated PET substrate. When the backside of PET substrate is smooth, the data are dominated by high-frequency oscillations from the substrate anisotropy. When backside is rough, these high-frequency oscillations are eliminated to leave only the data features pertaining to the surface coating

side reflection. Two techniques are common for suppressing backside reflections – the use of index matching materials to minimize this reflection, and roughening the backsurface to scatter the backside reflection. Figure 3 shows data collected from a coated PET substrate with and without backside reflections. Because of the "anisotropy" effects in the substrate of the data set with backside smooth (green curves), it is difficult to recognize the coating oscillation. With the backside rough (red curves), the high frequency oscillation is eliminated, allowing easy and accurate identification of the coating properties.

In a research lab, it may be acceptable to roughen the backside of anisotropic substrates. However, it is impractical to avoid collection of the backside reflections in many industrial environments. Thus, substrate anisotropy becomes part of the experiment. New *SE* measurement technologies have been developed to aid the measurement of anisotropic substrates. Generalized Spectroscopic Ellipsometry (*g-SE*) provides six measured values compared to the two terms of standard ellipsometry. The additional four values are related to cross-polarization that only occurs for anisotropic materials. The *g-SE* method has been used to analyze a wide variety of anisotropic materials, including bulk crystals and plastic substrates.

Mueller-Matrix Spectroscopic Ellipsometry (*MM-SE*) is an even more general measurement method that describes the reflection or transmission with up to 16 measured values. For most samples, there is significant redundancy in the Mueller-matrix. For highly complex samples which are both Anisotropic and Depolarizing, there are 7 distinct values. *MM-SE* has been applied to liquid crystal characterization to successfully measure the anisotropic optical properties and "twisting" behaviour of the LC layer.

Infrared spectroscopic ellipsometry

Historically, it has been difficult to accurately measure optical coatings in the infrared, which means that coating designs have relied on incomplete or inaccurate index and extinction coefficients found in the open literature. The open literature values are particularly questionable for thin films since the processing conditions can strongly affect optical properties. The optical components of an IR spectroscopic ellipsometer (*IR-SE*) – polarizers, retarders, light sources, detectors, etc. – are far from ideal. Innovative construction and calibration methods have allowed *IR-SE* to achieve the high accuracy required for successful thin film measurements.

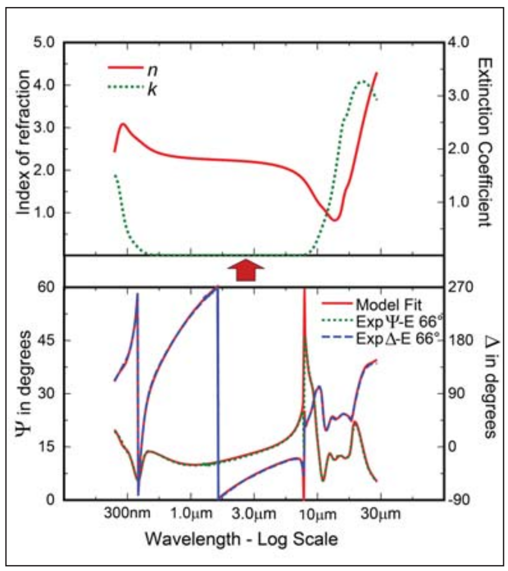
In the last decade, IR-SE has been used to measure thickness and the mid- and far-IR optical properties ($2 < \lambda < 33\mu m$) of a number of single and multi-layer films stacks. This has allowed better prediction and control of the mid-infrared Reflectance, Transmittance and Emissivity of film-substrate structures. *IR-SE* has been used to study both passive and active thermal control; for example, silver, tin oxides, electrochromic coatings, have all been measured. Figure 4 shows an IR-SE measurement of TiO_2 coating on float glass, along with the determined IR optical properties. TiO_2 has photocatalytic properties that are useful for self-cleaning windows. IR-SE can also be sensitive to water and other contaminants which may be incorporated in films or present on the film surface.

Large area uniformity

To produce Thin Film Transistor (TFT) screens, it is important to uniformly coat glass panels with the related thin films over large dimensions. To monitor the layer thickness uniformity on large glass panels, an ellipsometer mapping table was setup at Unaxis in Trübach under the direction of Dr. Sam Broderick. A compact SE moves over the glass panel as shown in Figure 5.

This development was made feasible by 3 features of modern elli-

Fig. 4 - Experimental SE data covering from the UV to IR for a TiO2 coated glass substrate. This data is used to accurately determine the TiO2 optical constants (n and k) – as shown



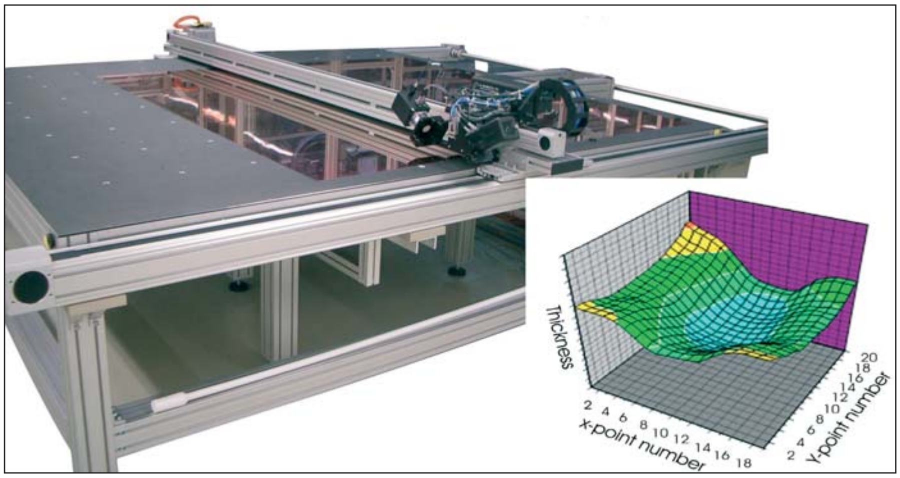


Fig. 5 - Large-area mapping SE used to test film uniformity on large glass panels

psometer technology: 1) compact optical design, 2) fast measurement speed, and 3) rotating compensator technology that allows accurate measurement with glass substrates. A panel can be scanned within minutes to provide the thickness profile. In addition, the ellipsometer can measure variation of the optical constants to provide layer 'quality' information to the process engineer.

Industrial integration

In situ *SE* measurements have been demonstrated with a large variety of processes, including Molecular Beam Epitaxy (MBE), Atomic Layer Deposition (ALD), Sputtering, Evaporation, Etching, and more.

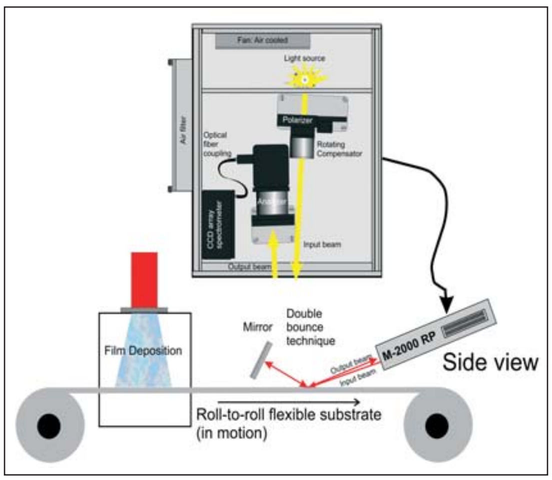


Fig. 6 - SE designed to work with roll-to-roll coatings by implementing a double-bounce method

However, the hardware integration is not always trivial. The substrates are often in constant motion, which requires rapid data acquisition and often a "triggering" method. It may also be difficult to approach the sample using a standard configuration with the source and detector at equal-yet-opposite angles. An innovative solution for web-coatings involves aligning the source and detector nearly parallel to each other in a single housing. The light beam exits the housing as shown in Figure 6 and reflects from the web. It is then incident on a mirror and returns to the same housing after bouncing a second time from the web. In this manner, coatings can be monitored on flexible roll-to-roll substrates.

For multi-layer applications, it can be beneficial to collect *SE* data after each new coating. This is possible with some planetary deposition chambers, where the sample can return to the measurement location. For other process designs, it may be helpful to use multiple *SE* systems. For example, Von Ardenne has incorporated a measurement system for in-line monitoring of multi-layer dielectrics on metal bands. Multiple *SE* systems are positioned after each layer deposition to provide accurate measurement of all layer thicknesses. Each *SE* communicates the current 'result' to the next ellipsometer in the process so it can analyze the new top layer with pre-determined results for the underlying structure. Figure 7 shows a typical Von Ardenne system with multiple ellipsometers mounted at key positions in the process.

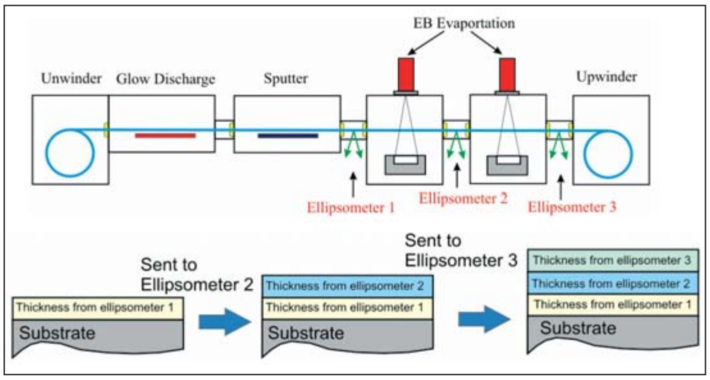


Fig. 7 - Multiple SE systems are established on a large processing chamber to study multilayer coatings at different steps during processing. Each SE system can feed the current information to the next SE system to be used as the basis for characterization when the sample reaches the next system

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