The determination of thin film thickness using reflectance spectroscopy

Application Note

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Abstract

The reflectance spectrum of a coated polycarbonate sample was used to determine the film thickness of a polymeric coating. Absolute reflectance spectra were acquired over the range 400–800 nm using a Cary 5000 UV-Vis-NIR spectrophotometer and ‘VW’ absolute specular reflectance accessory. Based on the interference fringes observed, the thickness of the film was calculated to be 4.95 µm.

Introduction

Thin films, layers of one material deposited on another material, are significant in many high-technology industries. Thin films are used in a wide variety of applications including antireflection coatings, beam splitters, color filters, narrow bandpass filters, semi-transparent mirrors, heat control filters, high reflectivity mirrors, polarizers and reflection filters. The characterization of thin films is thus extremely important in many optics/photonics applications (semiconductor, micro-machining, defence, architectural glass and flat panel displays to name but a few), with parameters of interest including film thickness, refractive index, coating homogeneity and reflectivity.

The measurement of film thickness using reflected light is a well-established technique[1]. Such optical techniques for the determination of thin film characteristics rely upon the interaction of the film with light, and can be used to determine not only thickness, but also roughness and optical constants. They are dependent upon the interference pattern (or fringes) resulting from partial reflection/transmission through two partially reflecting surfaces. This phenomenon was first observed over a century ago by Fabry and Perot[2] and, importantly, provides an investigative tool that is accurate, nondestructive, and requires little in the way of sample preparation.

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In this instance, an absolute specular reflectance measurement was used to determine the thickness of the coating on a polycarbonate substrate. The exact composition of the coating cannot be revealed, however it can be generally described as being polymeric in nature and having a refractive index of 1.51. This particular substrate/coating combination has application in the glass and automotive industries.

Theory

In the case of a thin film on the surface of another material, both the top and bottom surfaces of the film reflect light, with the total amount reflected being dependent upon the sum of these two reflections. Furthermore, these two reflections may add together constructively or destructively depending upon their phase relationship. This phenomenon is due to the wavelike nature of light, with the phase relationship determined by the difference in optical path lengths of the two reflections.

The resulting interference pattern (interference fringes) can be used to determine the thickness of the film in question, assuming that refractive index and angle of incidence are both known. Conversely, refractive index can be determined if film thickness is known. Film thickness can thus be calculated using the following expression:

$$d = \frac{m}{2D_n\sqrt{(n^2 - \sin^2\theta)}}$$

Where:

d = film thickness

m = number of fringes in wavenumber region used

n = refractive index

θ = angle of incidence

D_n = wavenumber region used (v_1 - v_2; cm^{-1})

Specular reflectance measurements were made using a 'VW' absolute specular reflectance accessory (SRA; see Figure 1). The 'VW' SRA is designed to measure 'mirror-like' reflectance from a sample surface, and has been described elsewhere[3]. The accessory uses a modification of the 'VW' configuration first described by Strong,[4] which calculates absolute specular reflectance using a pair of matched mirrors to perform the calibration and measure the sample reflectance. The Cary 'VW' absolute SRA eliminates the need for expensive, perfectly matched reference mirrors by using one movable mirror for both the calibration and sample reflectance measurements.

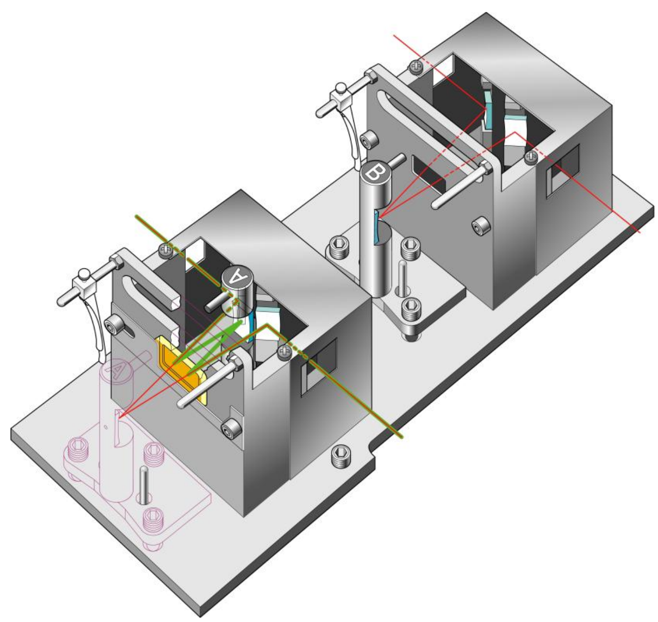

Figure 1. Optical diagram of the Cary VW absolute specular reflectance accessory

Materials and methods

For part numbers please see Reference 5.

Equipment

- Agilent Cary 5000 UV-Vis-NIR Spectrophotometer
- "VW" Absolute Specular Reflectance accessory

Protocol

The 'VW' SRA was installed into the spectrophotometer and aligned[6]. Reflectance spectra were collected between 400 nm and 800 nm using a spectral bandwidth of 2 nm and a scan rate of 600 nm/min (0.1 sec signal averaging time and 1 nm data interval). All measurements were made in double beam mode, using reduced slit height and zero/baseline correction.

In each case, the sample was positioned using the sample clip supplied with the 'VW' accessory. The 'Zero SRA' baseline correction was performed prior to the acquisition of sample spectra in order to set 0 and 100 %T values. This is particularly important when measuring samples with low reflectance (zero SRA baseline correction can be performed automatically by the Cary WinUV software).

Film thickness calculations were performed automatically using the Agilent 'Thin Film' ADL (Applications Development Language). The Applications Development Language is a spectroscopy programming language built into the Cary software. ADL uses simple programming terms to perform common spectroscopic functions and uses a commercial programming language, SAXBasic, to provide basic functionality with additional Cary-specific commands. ADL is a powerful spectroscopy tool that can perform everything from simple calculations on raw data to the production of a fully customized Cary interface for instrument setup, data collection, storage and retrieval of data, calculation of results, and report creation. The film thickness ADL and others are available for free on the Agilent web site.

Results and discussion

The absolute reflectance spectrum of the coated polycarbonate sample can be seen in Figure 2. The interference pattern due to the thin film is plainly evident over the entire wavelength range scanned, with the fringe spacing increasing with wavelength as expected.

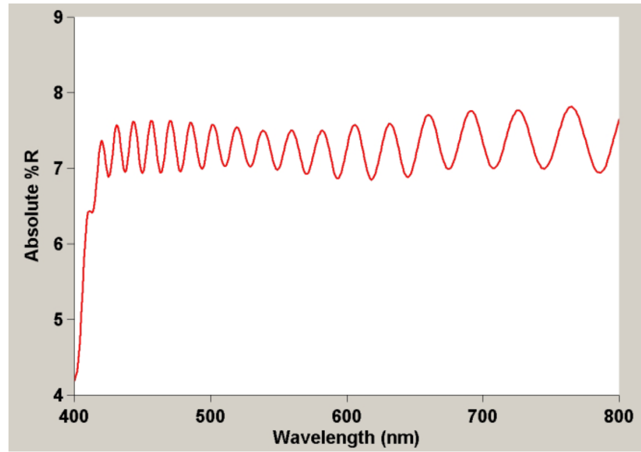

Figure 2. Absolute reflectance spectrum of coated polycarbonate sample showing interference pattern (or fringes) attributable to the coating (thin film) itself

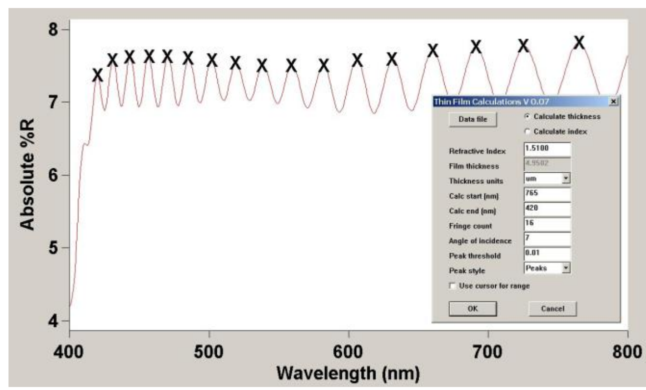

Figure 3. Interference fringes (16) identified between 420 and 756 nm using the Thin Film ADL

Using the aforementioned Thin Film ADL, 16 fringes were identified between 420 and 765 nm (Figure 3). Based upon an angle of incidence of 7 degrees and a thin film refractive index of 1.51, this fringe count resulted in a calculated film thickness of 4.95 µm (Figure 4)

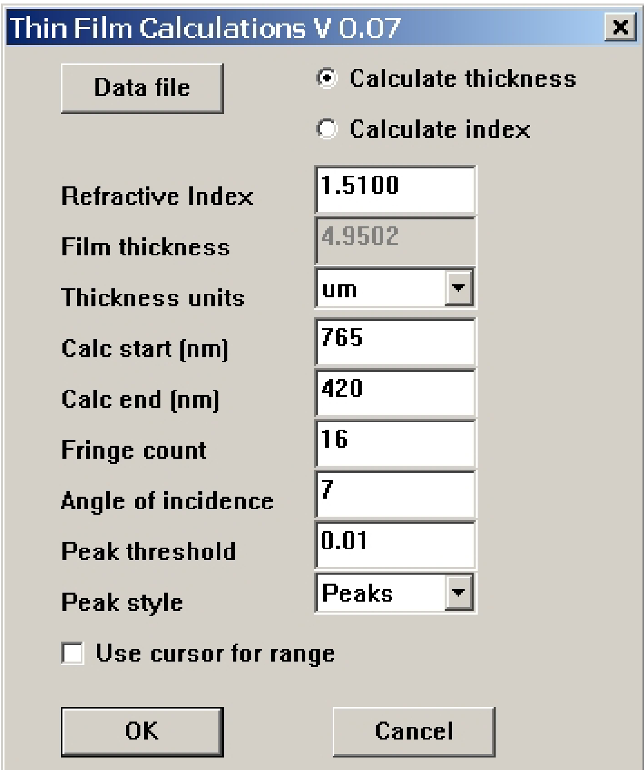

Figure 4. The Thin Film ADL dialog box showing parameters used for film thickness calculation. By selecting the 'Calculate index' option, refractive index may also be determined if film thickness is known

Conclusion

The thickness of a thin polymeric film deposited on polycarbonate has been calculated to be 4.95 µm. Measurement was based on the interference fringes observed in the reflectance spectrum of the coated polycarbonate sample. Absolute reflectance spectra were acquired over the range 400–800 nm using a Cary 5000 UV-Vis-NIR spectrophotometer and 'VW' absolute specular reflectance accessory.

References

1. Huibers, P. D. T. and Shah, D. O., Langmuir 13 1997 5995.
2. Fabry, C. and Perot, A., Ann. Chim. Phys. 16 1899 115.
3. Hind, A.R. and Soebekti R., 'The deep ultraviolet spectroscopic properties of a next generation photoresist', UV At Work 82, www.agilent.com.
4. Strong, J., 'Procedures in Experimental Physics', 1st Ed., Prentice-Hall, Inc., New York, 1938, 376.
5. Part numbers

Product	Part Number
Agilent Cary 5000 UV-Vis-NIR Spectrophotometer	0010079300
'VW' Specular Reflectance Accessory	0010043800
Cary WinUV Analysis Pack Software	8510195000

6. Cary WinUV Software, 'Cary Help' and videos, Version 3.0.

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