Application Note

Glass, ceramics, optics

Optical Characteristics and Thickness of 2-layered Structures

Refractive index and film thickness measured using a Cary 5000 with UMA accessory

Authors

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Introduction

Multilayer optical coatings are widely used in technologies that exploit the properties of light from the ultra-violet through to the infrared (*1*). Successful optical coating design and manufacturing demands high quality information about the refractive index, absorption coefficients and thickness of layered thin-film structures.

For the successful study of the properties of a thin-film structure use of non-destructive methods are preferred. The fundamental approaches to the characterization of layered thin-film structures differ fundamentally from optical characterization techniques for bulk materials. The optical properties of the films are typically characterized using ellipsometry—an optical technique based on the analysis of the polarization state of light reflected from the specimen. Now there is an alternative multipurpose nondestructive optical technique—multiangle spectrophotometry (*2, 3*).

Spectral and angular functions of reflectance and transmittance for incident polarized light is carried out using a spectrophotometer fitted with an accessory.

Although, optical parameters are very much dependent on

- substrate and film growth conditions
- film homogeneity
- substrate homogeneity and
- optical properties, *(4, 5)*

we expect here to maintain film homogeneity during the structures lifetime.

The aim of this work is to determine the parameters; thickness (d), refractive index (n) and extinction coefficient (k) of the films using the Agilent Cary 5000 spectrophotometer with a unique, and automated, Universal Measurement Accessory (UMA).

Experimental

The Cary 5000 spectrophotometer with UMA allows automated and unattended measurement of:

- absolute reflectance R (at 5–85° incidence) and transmittance T (at 0–85° deg incidence light) with a minimum step interval of 0.02° ;
- T and R measurements at different angles and polarization within one working sequence;
- a 190–2800 nm working wavelength range for unpolarized light;
- a 250–2500 nm wavelength range for *s*- and *p*-polarized light.

Thus, it is possible to obtain a full description of the sample, without moving it. The Cary 5000 with UMA is a universal measurement system that eliminates the need to use multiple consoles, replacement and/or reconfiguration. It provides high quality data, measuring all the characteristics from one area of the sample. A huge advantage of this accessory is the ability to measure the optical characteristics by varying the polarization of the incident light, at different angles of incident light, on the same region of the sample. The UMA scheme consists of a fixed light source, 360° rotatable sample holder and independent detector. The detector can move around the sample in a horizontal plane.

The refractive index and thickness of the films were characterized using double angle light incidence *(6)*. This method is only suitable for the spectral range in which the film is transparent or exhibits negligibly low absorption.

Results and Discussion

Two samples, each with a different substrate type (transparent or opaque in the visible wave range), were characterized :nanocomposite coatings Zr-Si-B-(N) films on quartz substrates (transparent in the visible range) *(7)* and layered lithium niobate $LiNbO_3$ structure specimens on (001) single crystal silicon substrates (opaque in the visible range) *(8, 9)*.

Both structures were grown using high-frequency magnetron sputtering. The ability of the substrate to transmit light affects the choice of the measured parameter in this method: for transparent substrates we can use transmittance, for opaque substrates we can use reflectance.

To determine the thickness (d) for the first sample transmission spectra were obtained at two arbitrary different unpolarized light incidence angles, in this case, at normal incidence φ1 = 0° and φ2 = 20° (Fig. 1).

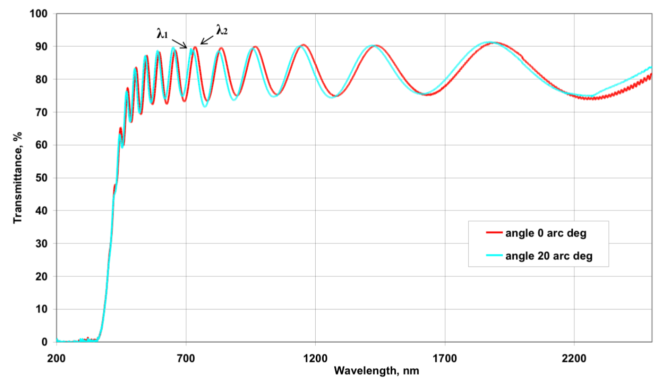

Figure 1. Transmission spectra of nanocomposite coatings Zr-Si-B-(N) films on quartz substrates at normal incidence ($λ_2$) and 20° ($λ_1$).

To evaluate the refractive index of the sputtered layer we used the spectral reflection dependences recorded at two different unpolarized light incidence angles, i.e. φ1 = 6° and φ2 = 20° (Fig. 2).

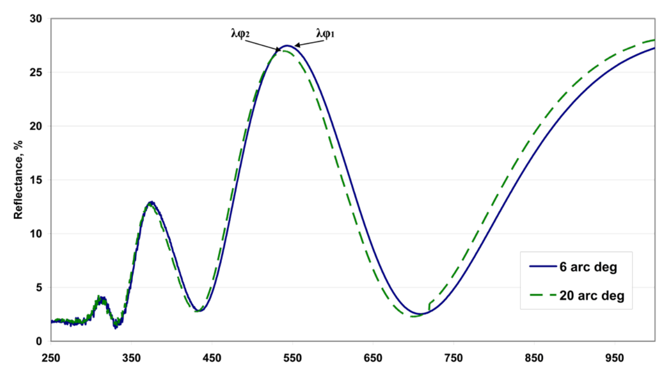

Figure 2. Reflection spectra of $LiNbO_3$ structure specimens on silicon substrates at two incidence angles 6° and 20°.

In the resultant spectra we select the wavelengths λφ1 and λφ2 corresponding to the same interference peak at the φ1 and φ2 incidence angles, respectively, and determine the refractive index of the film using the following formula *(6)*:

$$\left(\frac{n}{n_0}\right)^2 = \frac{\sin^2\varphi_1 - \beta\sin^2\varphi_2}{1-\beta},$$

where n_0 is the refractive index of the medium (here, n_0=1 for air) and β is the coefficient determined as follows:

$$\beta = \left(\frac{\lambda_{\varphi 1}}{\lambda_{\varphi 2}}\right)^2,$$

The film refractive indices n calculated using the first equation are summarized below for both samples in Tables 1 and 2, respectively.

The optical thickness of the film *dn* was calculated based on the position of the adjacent interference maxima λ1 and λ2 at the same light incidence angle (the lower one of the two) using the formula *(6)*:

$$d \cdot n = \frac{\lambda_1 \cdot \lambda_2}{4(\lambda_2 - \lambda_1)},$$

The film thickness as determined by dividing the optical thickness of the film *dn* by the refractive index *n* was 250 nm ± 30 nm. Unfortunately, the refractive indices determined using this technique are quite sensitive to the peak wavelength measurement accuracy. Thus, this method is only suitable for the preliminary estimation of refractive indices aimed at selecting the initial approximations.

These films have heterogeneous structure, which was detected by optical microscopy. This heterogeneity is apparent in the value ***n*** measurement accuracy. The accuracy of determining ***n*** for the films is not worse than 0,01.

Table 1. Obtained results for Zr-Si-B-(N) films on quartz substrates.

$\lambda_{\varphi 1}$ (20°), nm	$\lambda_{\varphi 2}$ (0°), nm	*n*	*d*, nm
470	474	2.64	1380±5%
502	507	2.44	
590	596	2.42	
650	657	2.35	
725	733	2.32	
824	834	2.22	
883	895	2.20	
955	966	2.17	
1037	1050	2.17	

Table 2. Obtained results for layered $LiNbO_3$ structure on single crystal silicon substrates.

$\lambda_{\varphi 1}$ (6°), nm	$\lambda_{\varphi 2}$ (20°), nm	*n*	*d*, nm
543.62	539.25	2.58	250±5%
708.83	700.12	2.09	

Based on the obtained results, we constructed dispersion curves of the refractive indices for nanocomposite coatings Zr-Si-B-(N) films on quartz substrates, and for $LiNbO_3$ structure specimens on silicon substrates, refer to figure 3 and 4, respectively.

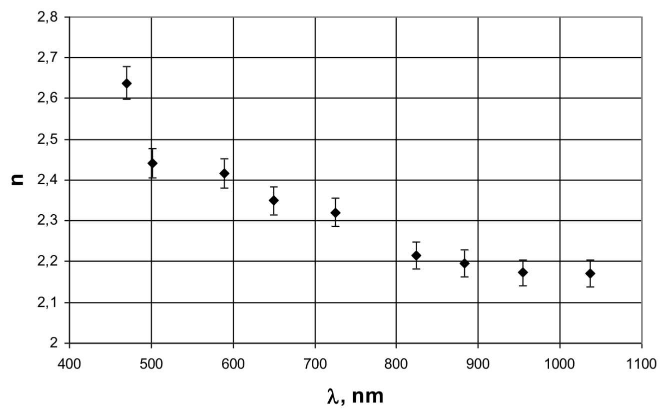

Figure 3. Dispersion curves of nanocomposite coatings Zr-Si-B-(N) films on quartz substrates.

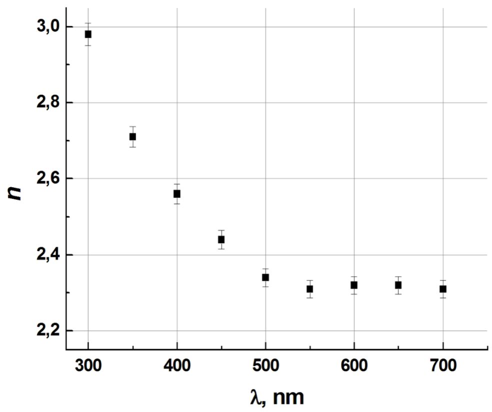

Figure 4. Dispersion curves of $LiNbO_3$ structure specimens on silicon substrates.

Conclusion

The refractive index of two samples was determined, using measurements performed on a Cary 5000 spectrophotometer, fitted with an Universal Measurement Accessory.

One sample consisted of a nanocomposite coating: Zr-Si-B-(N) films on a quartz substrate (transparent in the visible range). The second was a layered lithium niobate $LiNbO_3$ structure on a single crystal silicon substrate (opaque in the visible range).

The refractive index of both was calculated with an accuracy of ± 0.01. Film thickness was calculated from the determined reflective index for both samples.

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