Optica Applicata, Vol. XL, No. 4, 2010

Studies of polymer surface topography by means of optical profilometry

Janusz JAGLARZ[*], Jerzy SANETRA, Jan CISOWSKI

Institute of Physics, Cracow University of Technology,
ul. Podchorążych 1, 30-084 Kraków, Poland

[*]Corresponding author: pujaglar@cyfronet.pl

The optical reflection measurements of polyvinylocarbazole (PVK) and polyazomethine (PPI) thin films have been done by means of optical profilometry (OP) exhibiting many advantages in surface and subsurface investigations. The obtained OP images clearly demonstrate that the thickness of the polymer films under investigation is not uniform over their lateral dimensions. For the PVK thin film, the fast Fourier transform (FFT) in the inverse space of the OP image is also presented along with distribution of the surface highs.

Keywords: optical profilometry, thin polymeric films.

1. Introduction

Thin transparent polymer films are currently intensively investigated because of their promising optical and electrical properties for applications in electronics [1]. Among others, the reflection study is an old classical method to achieve reliable results of opto-electronical parameters and much interesting data may be obtained, for example, from the non-specular scattered radiation measurements. This type of light scattering, known as diffusive scattering, originates from the roughness of the film surface and from the volume scattering caused by particles concentrated in the medium layer.

According to fundamental optics, the light reflected from any real surface involves two components. The first is simply the specular beam whose intensity depends on physical structure of reflecting surface (specular reflectance R_{spec}). The second, including scattered beams from the sample (diffuse reflectance R_{diff}), depends on surface irregularities [2] and it often complicates an analysis of the optical spectra of thin polymer films.

An expression for the relation between specular and diffuse reflectances as well as root-mean square (rms) roughness σ may be obtained from the statistical treatment of the reflection of electromagnetic radiation from a rough surface. In the first approximation it is given by [3]:

$$R_{\mathrm{diff}} = R_{\mathrm{spec}}\left[-\frac{(4\pi\sigma)^2}{\lambda^2}\right] \tag{1}$$

where λ is the wavelength of illuminating light. Formula (1) should be used when $\sigma \ll \lambda$. If $\sigma < 10$ nm, a measurement of diffuse reflectance with λ of the order of a few hundred nanometers yields a reliable roughness, comparable with values obtained from other studies. In the case of polymer films, the surface scattering may be connected with irregularities appearing on the film–substrate and polymer–air interfaces.

For many polymer films obtained by chemical vapor deposition (CVD), the scattering centers may appear, such as spherulites, crystallizing during the deposition process and mainly responsible for the light scattering. The type of scattering can be concluded from the wavelength dependence of the scattered light intensity. In agreement with the Rayleigh classical theory, the volume scattering coefficient s_v, for an average size of spherulites less than 0.1λ, is equal to [3]

$$s_v = \frac{32\pi^3}{3N\lambda^4}\left(n^2 - 1\right)^2 \tag{2}$$

where N is a concentration of scattering centers and n is the refractive index. The volume scattering is a part of elastic scattering caused by the medium. It is assumed that there is no energy loss accompanying this type of scattering and that the scattering medium is spherically symmetric [4].

The scattered radiation measured by optical profilometry (OP) is a function of highs of irregularities and slopes of microfacets, but sensitivity of this method follows mainly from detection of the slope change. The presence of long lateral irregularities is often caused by manual or mechanical treatments and may have a periodical nature. The short spatial waves result rather from random process of the surface formation and their contribution to the total profile is easy to determine from atomic force microscopy (AFM) and scanning electron microscopy (SEM) techniques.

2. Experiment

The scheme of optical profilometer (OP), shown in Fig. 1, is a multifunctional experimental setup for surface topography investigations. It works in two modes. The first – the specular mode, employs the He-Ne laser as a light source with the collimating system allowing one to achieve a 5 μm diameter light beam. It allows one to obtain the optical map of surface with a 5 μm lateral resolution. OP measurements can be normalized with the calibration sphere method [5].

The information related to the average film thickness d and roughness σ can be obtained mainly from the spectral reflectance measurements $R(\lambda)$. For non-diffusive films, the intensity of light is proportional to changes of thickness, provided $d > 100$ nm and assuming that the refraction coefficient has a value typical of transparent polymers, *i.e.*, from 1.5 to 2.

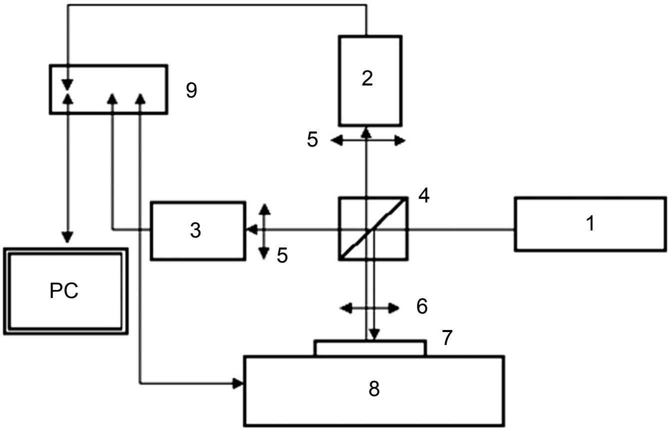

Fig. 1. Scheme of optical profilometer: 1 – laser diode ($\lambda = 635$ nm), 2, 3 – detectors, 4 – beam splitter, 5 – collecting lens, 6 – objective, 7– sample, 8 – *XY* stage, 9 – controlling/collecting unit.

The intensity of light, scattered by diffuse films into the solid angle 2π is proportional to the film thickness (the thicker the film, the larger the number of scattering centers and the greater the intensity of backscattered light).

3. Results and discussion

OP measurements have been used to obtain surface images of PVK films formed by spin coating and PPI films prepared by CVD.

Figure 2 shows the OP image obtained for a PVK film deposited on the BK7 glass substrate.

Colors in Fig. 2 represent a relative thickness change over the lateral dimensions of the PVK film, *i.e.*, red represents thicker regions, while blue – thinner ones.

From the OP profile, the power spectra, shown in Fig. 3, have been calculated by means of the sensor probe imaging processing (SPIP) program [6] using the fast Fourier transform (FFT).

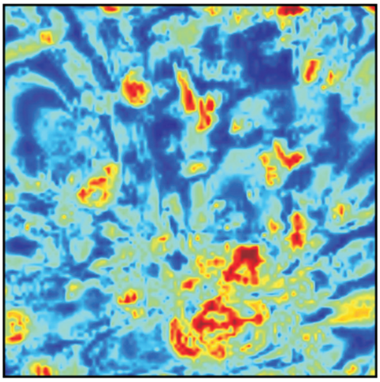

Fig. 2. OP image of a PVK film on the glass substrate.

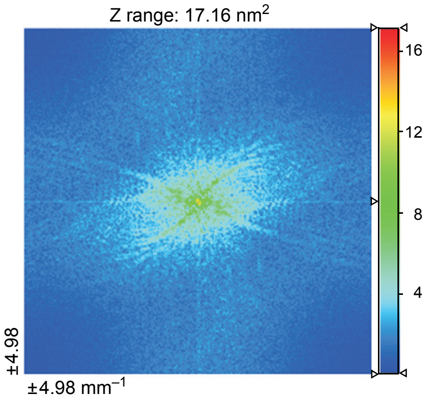

Fig. 3. Fast Fourier transform (FFT) for the PVK film from Fig. 1.

Fourier transform analysis of optical profiles, shown in spatial frequencies, allows one to distinguish periodic features and to specify interference of spatial waves, and, especially, to evaluate the surface anisotropy. Since the PVK film was formed by the centrifugal force during the spin-coating process, random irregularities observed along *X* and *Y* axes are similar and axially symmetrical, as seen in Fig. 3.

The high distribution of surface features has also been calculated by means of SPIP program and Fig. 4 shows a relevant histogram of surface highs.

As can be seen in Fig. 4, the distribution of highs on the PVK film surface can be described using the Gaussian function centered at about 480 nm.

The OP image of a diffusive PPI film is shown in Fig. 5.

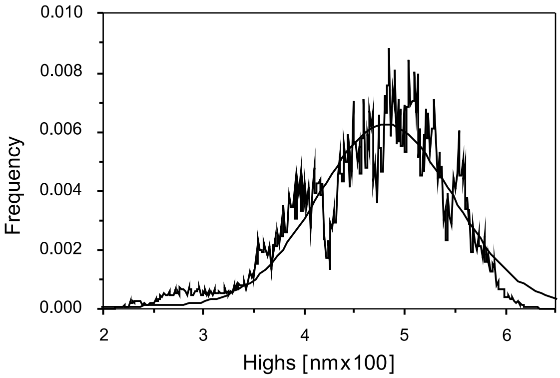

Fig. 4. Distribution of highs for a PVK film.

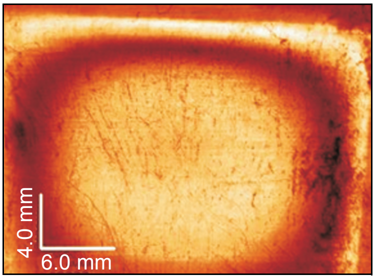

Fig. 5. OP surface profile of a PPI thin film.

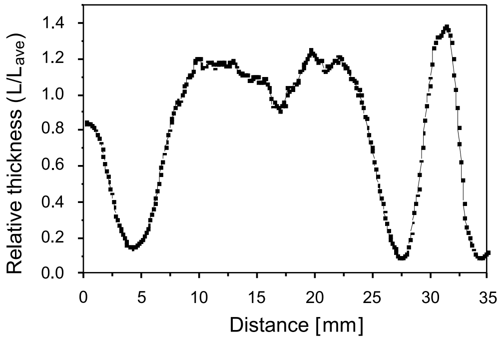

Fig. 6. Relative thickness of a PPI thin film as a function of distance along the *X*-axis of profilometer.

The intensity of the light reflected by diffuse films into the specular direction depends on the film thickness (the thicker the film, the larger the number of scattering centers and the greater the intensity of backscattered light).

From the OP profile of the PPI film from Fig. 5, its relative thickness along the *X*-axis of profilometer has been found, as shown in Fig. 6.

As can be seen from Fig. 6, the film thickness is not uniform along the lateral dimension of the PPI sample investigated.

4. Conclusions

OP investigation enables one to find many interesting features concerning surfaces over a much larger area than with AFM and SEM techniques. From the image analysis, it is possible to determine the most important statistic parameters of surfaces, such as roughness and high distribution function.

In general, the values of root-mean square (rms) roughness obtained from AFM are smaller than those measured by OP, being a result of different scanning areas ($100\ \mu\text{m} \times 100\ \mu\text{m}$ and $50\ \text{mm} \times 50\ \text{mm}$ for AFM and OP, respectively). OP also completes the surface description in longer space wavelengths [7]. The FFT of OP images allows one to find distribution of features in the inverse space, like their periodicity and anisotropy. The presence of long lateral irregularities of the periodical nature is often met in the polymer technology. On the other hand, the short spatial waves result rather from the random process of the surface formation and their contribution to the total profile is easier to determine by AFM and SEM techniques than by OP.

In summary, the results obtained for PVK and PPI thin films treated as examples, clearly demonstrate the usefulness of the optical profilometry for the surface topography studies of various materials.

References

[1] Łużny W., Stochmal-Pomarzańska E., Proń A., *Structural properties of selected poly(azomethines)*, Polymer **40**(23), 1999, pp. 6611–6614.

[2] JAGLARZ J., KASSIBA A., ARMATYS P., POKŁADKO M., GONDEK E., SANETRA J., *Polymeric photovoltaic devices*, Materials Science–Poland **22**(4), 2004, pp. 389–395.

[3] STOVER J.C., *Optical Scattering: Measurement and Analysis*, SPIE Press, Bellingham, 1995.

[4] JIAMIN ZHOU, JING SHENG, *Small angle light backscattering of polymer blends: 1. Multiple scattering*, Polymer **38**(15), 1997, pp. 3727–3731.

[5] ZIĘBA Ł., JAGLARZ J., DĄBROWSKI M., DURAJ R., CISOWSKI J., JURUSIK J., *Surface topography investigations of TiN layers on different substrates*, Reviews on Advanced Materials Science **15**(1), 2007, pp. 63–68.

[6] ZAHOUANI H., VARGIOLU R., KAPSA PH., LOUBET J.L., MATHIA T.G., *Effect of lateral resolution on topographical images and three-dimensional functional parameters*, Wear **219**(1), 1998, pp. 114–123.

[7] STOVER J.C., SERATI S.A., GILLESPIE C.H., *Calculation of surface statistics from light scatter*, Optical Engineering **23**, 1984, p. 4.

Received October 1, 2009
in revised form February 8, 2010