TRANSPARENT INHOMOGENEOUS THIN FILM CHARACTERISATION USING INTERFEROMETRIC TECHNIQUE

Mona MIHAILESCU[1], Raluca GABOR[2], Valentin RADITOIU[3], Cristi-Andi NICOLAE[4], Niculae N. PUSCAS[5]

In this paper, we present our results obtained using an interferometric technique to investigate a constant thickness sample, which presents inside regions with different values for the refractive index. The sample is prepared on a polycarbonate (PC) substrate. On its surface, some microstructures are imprinted using controlled values for temperature and forces, after we determined experimentally the value of the temperature for glass transition. These microstructures were then filled with an epoxy resin. Using the images acquired experimentally, we computed the phase shift map introduced by the sample in the optical path. It is proportional with the geometrical height of the microstructures and with the difference between the refractive indices of the polycarbonate and the epoxy resin. In this way, we find the phase shift profile inside of a transparent thin film of a sample with plane parallel surfaces.

Keywords: inhomogeneous transparent thin film, digital holographic microscopy, polycarbonate, epoxi resin.

1. Introduction

Inhomogeneous thin films are important in many fields like: medicine for implants, memories, integrated optics, plasmonics [1-5]. To obtain transparent inhomogeneous thin films, different techniques are used - controllable changes (1) of the refractive index by direct laser writing, infusion, thermal modifications, other techniques or (2) of the microrelief which is then filled with another material with an appropriate value for refractive index [6-8]. They are made in different polymer or glass types (polymethyl metachrylate, polycarbonate, chalcogenide glass for example). In their morphological characterization is

[1] Lecturer, Dept.of Physics, University POLITEHNICA of Bucharest, Romania, e-mail: mona@physics.pub.ro
[2] Eng., National Institute for Optoeloctronics, Romania
[3] Researcher, Dept.of Polymer Science, University POLITEHNICA of Bucharest, Romania
[4] Eng., National Institute for Optoeloctronics, Romania
[5] Prof., Dept.of Physics, University POLITEHNICA of Bucharest, Romania, e-mail: pnt@physics.pub.ro

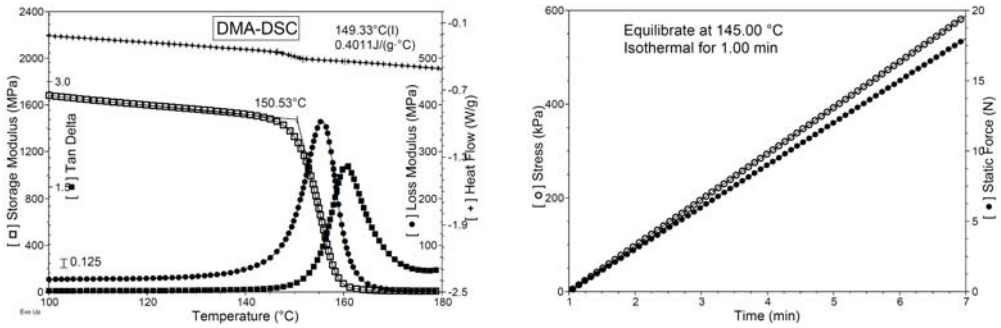
important to have information about the refractive index variations. In this paper we describe our investigations to establish the phase shift profile, which is proportional with the difference between the refractive index of the substrate and the inhomogeneity. The interferometric technique employed is a non-contact, non-invasive, non-destructive technique, which offers quantitative information about the phase shift introduced by the sample in the optical path. We use an experimental setup based on the modified Mach-Zehnder interferometer, off-axis configuration. The object beam carries information about the sample and interferes with the reference beam on a CCD camera sensor. From these images, intensity images, we reconstruct digitally the phase map of the sample which is proportional with the microstructure height and with the difference between the refractive indices of the substrate (polycarbonate) and the epoxy resin which fills the microstructure.

2. Sample preparation

In recent years, polymeric integrated optics is of interest due to low cost of materials. Polycarbonate is a promising material for substrates in which the refractive index can be easily changed locally in a controllable way. Our investigations are focused to characterize a sample with a desired microstructure imprinted at controlled values for temperature and forces, after we determined the glass transition temperature. These microstructures are then filled with a different material characterized by appropriate values of refractive indices. In our case, the substrate is from polycarbonate and the filled material is an epoxy resin.

Typical *Dynamic mechanical analysis* (DMA) and *Differential scanning calorimetry* (DSC) investigations were done using TA Instruments on a polycarbonate (PC), rectangular sample (60:12.7:5.02 from LEXAN-as received). The investigated interval for temperature was 100-180^{0}C and the aim was to establish the glass transition temperature for this sample [9-13].

In DMA we choose the "multifrequencies-strain" mode (MS-mode). Using dual cantilever fixture suitable for this kind of sample, we applied an oscillatory force and maintained constant amplitude of 20 μm and a single frequency of 1 Hz in a temperature range RT-180°C with a heating rate 3°C/min, in air. Automatically, the variations of the parameters are measured experimentally inside the chamber while the mechanical properties: loss modulus, storage modulus and their ratio are computed using the dedicated Universal Analysis 2000 software form TA Instruments (see Fig. 1a).



a) b)

Fig. 1 a) Diagrams obtained in DMA and DSC modes for typical parameters b) The force with values which increase in time and the sample response in stress values

The sample was investigated in helium as purge gas (50ml/min) from RT to 180°C with a ramp of 10°C/min in an aluminium pan, using a DSC-Q2000. Heat flow variation with temperature was computed based on experimental data. We can see in Fig. 1a that around 149^0C this curve has a step, which corresponds with the glass transition temperature. An appropriate value is obtained also from DMA analysis (150^0C). From this reason we choose a value of 145^0C to imprint the microstructures.

The investigated sample was cut in pieces of 5/5 mm from a sheet of 5 mm thickness (LEXAN-as received).We imprint the microstructures in DMA-Controlled force mode using the penetration clamp in isothermal condition at 145°C with an increased force (ramp force 3N/min from 0.0001N to 18 N) using the same device DMA Q800. In Fig. 1b is represented this force, which increases in time and also the PC response in values of stress. In this kind, the microstructures from the penetration clamp in the form of concentric circles were imprinted in PC sample at controlled values for force and temperature.

The epoxy resin (Diglycidyl ether of Bisphenol A) was mixed with a stoichiometric amount of triethylentetramine under continuous mechanical stirring and then sonicated for 15min until a homogeneous mixture was obtained. The blend was transferred to PC-sample to fill the microstructures, degassed in vacuum and cured as follows: 2h at 40°C followed by 2h at 60°C and 2h at 80°C.

3. Experimental investigations

In classical microscopy, for transparent samples, differential interference contrast (DIC) is a technique which allows 3D visualization, but without quantitative information along the propagation axis. In Fig. 2 are two regions from the same sample visualized in DIC examination mode, when it was filled with

water and when it was filled with epoxy resin. For these investigations we used the NIKON Ti-U inverted microscope (20x objective with DIC slider). The sample was situated between polarizer and analyzer. The images were acquired using DS Fi-1 CCD camera and were processed using NIS Elements software.

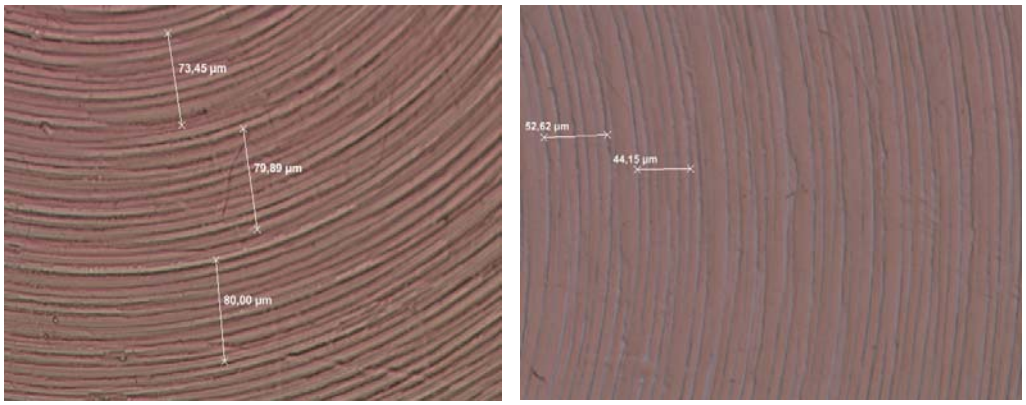


Fig. 2 The same sample visualized in DIC examination mode
a) filled with water b) filled with epoxy resin (different resolution)

To obtain quantitative information along the propagation axis about the phase shift introduced by the sample in the optical path, and consequently about the microrelief height, an interferometric technique was employed. The values for the phase difference introduced by the sample in the optical path resulted after the reconstruction step, are proportional with the microstructure geometrical height, h_{ms} and with the difference between the refractive indices of the PC, n_{PC}, and epoxy resin, n_{er}:

$$\Delta\Phi = \frac{2\pi}{\lambda} h_{ms} \left(n_{PC} - n_{er} \right) \tag{1}$$

where λ is the wavelength of the used laser (632,8nm from a HeNe laser double stabilized Spectra Physics).

The experimental setup is based on the Mach-Zehnder interferometer. Two additional microscope objectives are added in both arms and the sample is placed in the focal plane of one of them. In the literature, this examination mode is named digital holographic microscopy technique (DHM) which is a non-contact, non-invasive, marker free method to analyze transparent samples and which offers quantitative information with nanometric accuracy in the axial direction. It is applied to investigate different sample types: microstructured material, fiber optic, all kind of cells [14-18]. The lateral resolution is around 1 µm restricted by the diffraction limit, numerical aperture and magnification of the objective,

dimensions of the recording sensor pixels, distances inside the experimental setup. We choose the off-axis geometry and the angle between the beams ensures a proper interfringe value which matches the microstructure transversal dimensions and the pitch of the CCD camera sensor (Pike F421 with Kodak sensor 2048x2048pixels, pixel pitch $\Delta x = 7.4\,\mu m$, acquisition rate of 16fps at full resolution). The used objectives have magnification of 40x which permits 0.9μm lateral resolution. A condition for a proper visualization of the inhomogeneity in the focal plane of the microscope objective is: the distance between the inhomogeneity and the sample surface must be smaller than the focal distance of the objective.

To visualize the inhomogeneity regions situated inside the sample, we recorded the holograms formed through superposition between the reference and object wave on the CCD camera sensor. The last carries the diffracted field from the object. In the holograms (see Fig. 3) we can see the specific maxima and minima of the diffraction pattern from sample edges and also the linear fringes from the interference pattern between object and the reference wave.

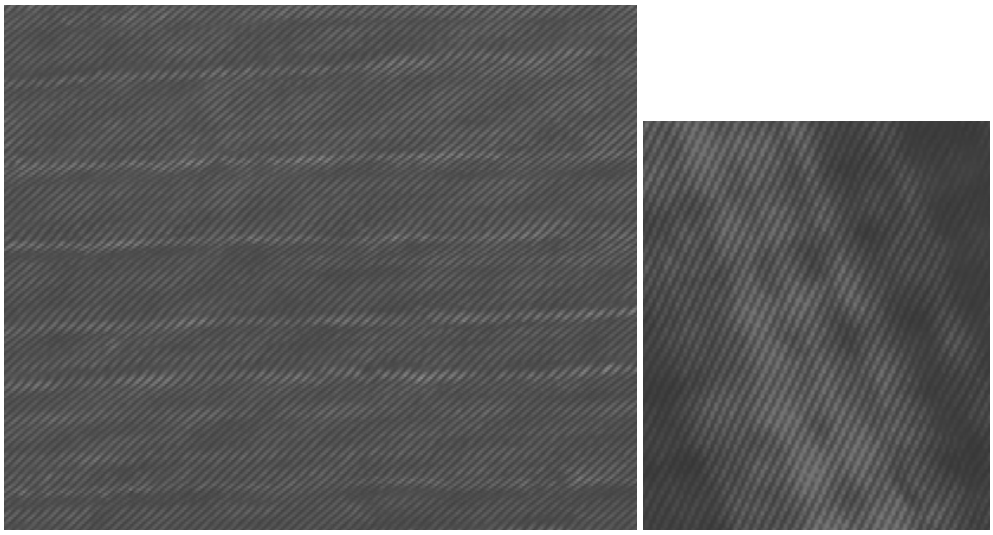


Fig. 3 Holograms recorded experimentally for the microstructures

Calibration of the dimensions in the transversal plane was done using an object made with electron beam lithography, with precise dimensions. In the Fig. 4 is the hologram for the calibration sample recorded experimentally in the same conditions.

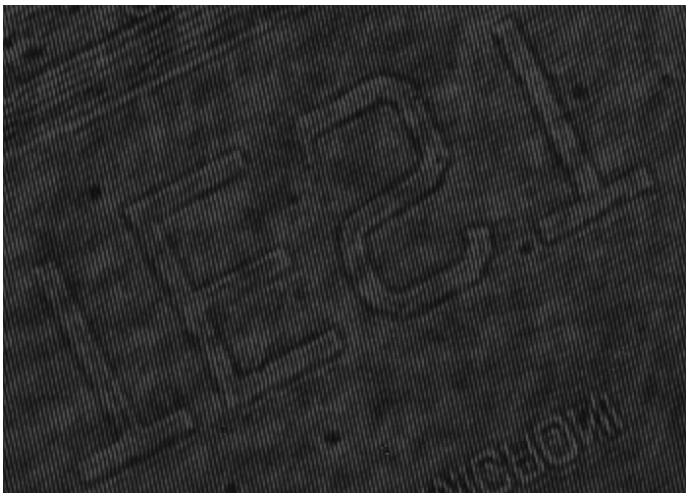


Fig. 4 Hologram for the calibration sample

4. Results

To reconstruct the object image, we use an algorithm based on the diffraction scalar theory, Fresnel approximation [19-22]. We follow the standard procedure [23] with steps which include: Fourier transform to separate the twin images, Fresnel transform to reconstruct the object image from the +1 order, correction for tilt and spherical aberrations, numerical focalization. The main advantage of digital holographic microscopy technique is the fact that we record one single hologram from which we reconstruct the whole object, without mechanical scanning along propagation axis or in transversal plane. In this way, depending of the used objective, we can visualize large portions from the sample in a short time.

In Fig. 5 is the reconstructed image of the calibration sample.

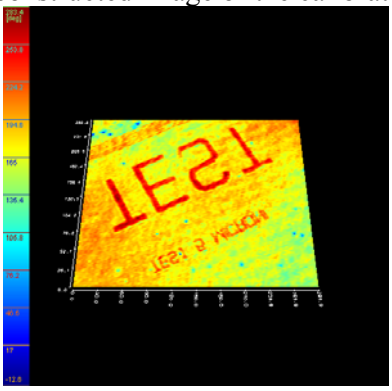


Fig. 5 The reconstructed image of the calibration sample

In Fig. 6 are some images of the microstructure reconstructed from holograms. The condition necessary to be fulfilled in this technique is that the phase difference between the beam which passes through one valley and mountain of the microstructure, to be smaller than 2π.

Fig. 6 Reconstructed images of the micostructures when they were filled with the epoxy resin

In Fig. 6 are 2D images in gray levels which are calibrated in values of the phase shift introduced by the sample in the optical path. In Fig.7 are 3D representations of the same microstructures and on the vertical axis are also values for the phase shift.

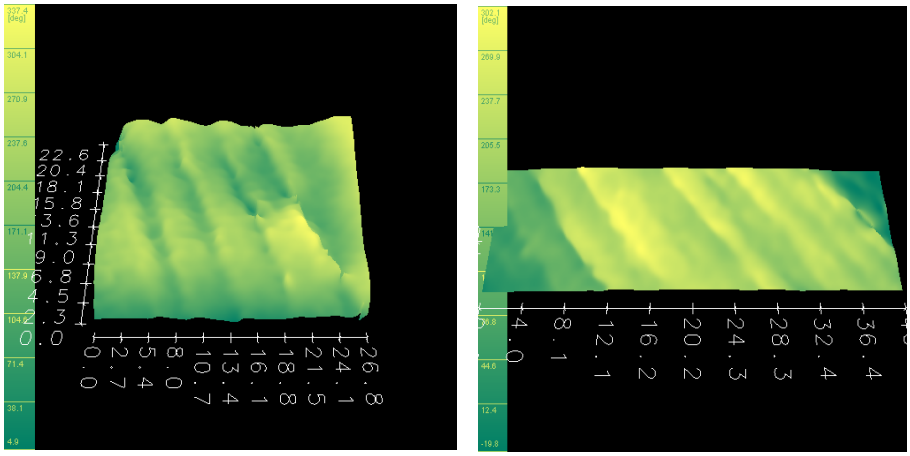


Fig. 7. 3D reconstructed images of different regions from the same sample.

One profile made using information from inside the sample is presented in Fig. 8. In these investigations using digital holographic microscopy technique, we consider that for polycarbonate the refractive index is 1,584 and for the epoxy resin it is 1,5719. In this case, the values for the microstructures height are tens of microns.

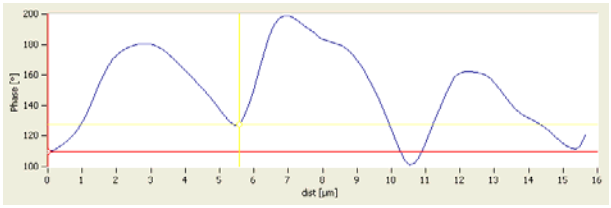


Fig. 8 One profile of the phase shift introduced by the microstructures

6. Conclusions

Inhomogeneous transparent thin films were prepared in PC by imprinting micronic structures using DMA technique, at controlled values for temperature and force on a polycarbonate substrate. The value for temperature was chosen under the glass transition temperature, which was determined experimentally for our sample using both DMA and DSC examination mode. The microstructures where then filled with epoxy resin which have an appropriate value for the refractive index.

Using an experiment based on an interferometric technique we can visualize the inhomogeneities situated few hundred of microns below surface. Using classical optical microscopy in DIC examination mode the sample inspection was done, but, in order to obtain quantitative information along propagation axis, we use digital holographic microscopy. From one single hologram, acquired in fractions of seconds, without mechanical scanning, we reconstruct the 3D image of the inhomogeneity situated few hundreds of microns below the plane surface. From the values of the phase shift between a valley and a mountain, knowing the refractive indices of the materials, we computed the microstructures height of tens of microns. This is a preliminary study and these procedures will be used to characterize the inhomogeneity induced in the chalcogenide glass by thermal effects.

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