Interferometric Method Application for Sub-micrometers Thickness Measurements of Spin-coated PEPC and PETPC Polymer Films

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Abstract **- This paper deals with the interferometric thickness measurements of spin-coated thin polymer films. Spin coating is currently the predominant technique employed to produce uniform thin films of polymers in sub-micrometer range. But the thickness measurement of such thin films requires the application of high precision methods. In the paper we design and develop the system based on the common interferometer MII-4 and digital camera for measurement of the thin PEPC and PETPC polymer films. Different concentration of polymer solution and spin speed were used in order to obtain thin films with variable thickness (from 100 nm to 1000 nm) by spin coating technique.**

Index Terms – thin polymer layers, spin-coating, interferometric thickness measurements.

I. INTRODUCTION

Nowadays polymers play a critical role in the advancement of the microelectronics and optoelectronic industry. They serve as photoresists in microlithography and as insulating dielectric materials in chips, displays, interconnects, and photonic devices [1, 2]. A large number of different deposition techniques are used for the production of thin films for optical applications. The most important categories are thermal vaporization, sputtering, and chemical deposition. It is obvious, that suitable coating materials are required for each deposition technique. Properties of thin films including optical, mechanical, electrical and thermal properties are influenced by deposition parameters. Polymeric films can be fabricated by use of various techniques [3]. The self-assembly [4], the co-extrusion [5] and the spin coating [6, 7] have ever been used for fabrication of the layer structures based on polymers. However, obtaining of thin polymer films with required thickness and accurate control of the layer thickness remains one of the important problem in thin film researches. Spin coating is one of the technological and accessible method of obtaining polymeric thin films. M. Kimura et al. [8] first fabricated multi-layered structures using polystyrene and polyvinylalcohol. Recently, A.L. Alvarez et al. [9] also demonstrated polymeric multi-layered structures obtained by spin coating of polyvinylcarbazole and polyvinylalcohol. In both cases, accurate control of the optical thickness up to a quarter wavelength was not achieved. So, the measurement of the thickness of transparent films become one of the important problem in optics research and in industry. The most common thickness measurement types available commercially are ellipsometry [10], the spectral reflectance/transmittance method [11] and interferometry [12]. Ellipsometry measures reflectance from a thin film at two different polarizations. Its precision is very high in the sub-micrometer range, but its measurement range is limited to several micrometers. The operations and calculations of ellipsometry are very complex. For the spectral reflectance/transmittance method, the incident light should cover a range of wavelengths and be adjusted normal to the sample surface. The spectral reflectance/transmittance method is simpler and less expensive than ellipsometry, but it can be used for films with thickness comparable with used study wavelength. Determination of film thickness by optical interferometry technique is widely used. Measurements are nondestructive and relatively inexpensive. Interferometry relies on the interference of two or more beams of light. The optical path difference of these beams is related to film thickness.

In this paper, we report the results of the fabrication of polymeric films based on polyepoxypropylcarbazole and polyepitiopropylcarbazole and application of interferometric PC based measurement system for high precision analysis of film thickness.

II. EXPERIMENTAL

Synthesis of polymer. Polyepoxypropylcarbazole (PEPC) and polyepitiopropylcarbazole (PETPC) were selected since they are known to have excellent film forming properties and photoinduced properties. A set of PEPC and PETPC used in this investigation were synthesized by polymerization of epoxypropylcarbazole and epithiopropylcarbazole at the presence of 1-3% potassium methylate on the anionic mechanism at temperature 80-120˚C within 2-6 hours. For the full drying they were stored in a vacuum drying chamber at 50˚C up to constant mass. From the characterization by polymer viscosity using calibrated standards results a molecular weight Mw 2000-3000. In Fig. 1 the chemical structure of polymers PEPC and

PETPC are presented.

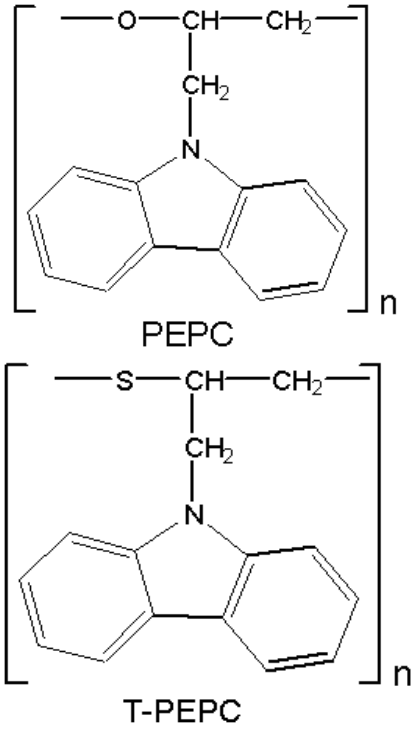
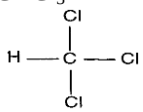


Fig. 1. Chemical structure of polymers PEPC and PETPC.

Formation of polymer films from solution.

The thin polymer films were prepared from homogeneous polymer solution by spin coating procedure using programmable spin-coater "SGS Spincoat G3P-8". The thickness of the polymer film was varied by changing the concentration of polymer solution and the rotation speed of spin coating. In sample series A, PEPC concentration of the solution was kept fixed (10 wt% solutions of the PEPC polymer in chloroform) and spin speed was varied which results in different film thicknesses. In sample series B, the PEPC concentration was varied (from 2.5 to 12.5 wt% solutions in chloroform $CHCl_3$) at fixed spin speed. The physical and chemical properties of used solvent chloroform is shown in Table 1.

TABLE 1. PHYSICAL AND CHEMICAL PROPERTIES OF CHLOROFORM.

Chemical name	Trichlormethane
Chemical formula	$CHCl_3$
Chemical structure	
Molecular weight	119.38
Color	Colorless
Melting point	-63.5 ˚C
Boiling point	62 ˚C
Density, at 20˚C	1.483 g/cm^3
Refractive index, n_D^{20}	1.4459
Organic solvents	Miscible with principal organic solvents. Miscible with alcohol, benzene, ether, petroleum ether, carbon tetrachloride,carbon disulfide, oils.

Operation conditions for polymer solution deposited on 5 cm diameter optical glass substrate (BK7) was as follows: 2 cm^2 of liquid dispensed on the disk at rest, subsequently accelerated in about 10 s to 3000 rpm and spun for 20 s. The broad range of thicknesses can be covered by using polymer solution with increasing solids content or for a given solution by changing the final spin speed.

The used coating cycle is presented in Fig. 2.

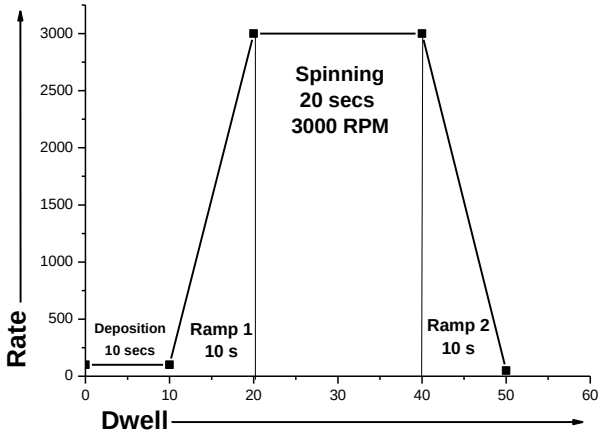


Fig. 2. Rate/time schedule of spin-coating for polymer films.

Determination of film thickness.

For the determination of film thickness in this work the modified interferometric PC based measurement based on MII-4 interference microscope was applied.

A thickness characterization of the samples was achieved by a MII-4 interference microscope (Fig.3). with CCD-camera recorded the micrographs. The interference pattern of light reflected from a flat reference surface and the investigated sample was recorded in PC. A magnification of 490 times was used. The area from which a data analysis is performed was 0.3 mm diameter circle. This enables a height resolution better than 100 nm [12].

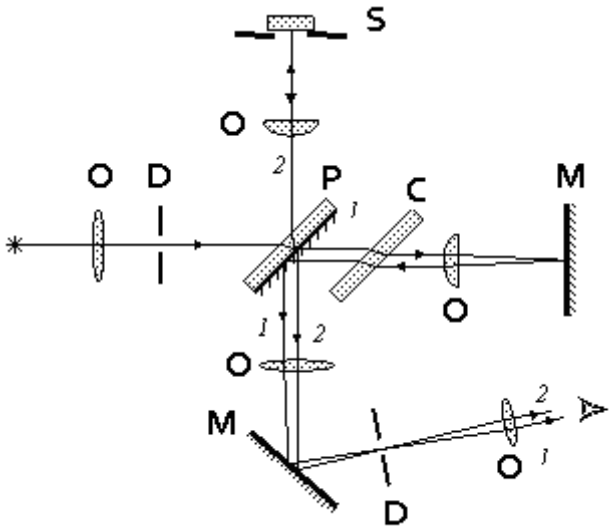


Fig.3. Optical scheme of MII-4 interference microscope. 1 – reference beam, 2 – object beam, O – objectives, D – diaphragms, M – mirrors, P - beam-splitting plate, C – compensating plate, S – sample.

The interference fringe shift in interferogram introduced by different height of layer is shown in Fig. 4.

III. RESULTS AND DISCUSSION

Good surface quality and uniformity of the films was confirmed by the smooth interference fringes in the

interferogram obtained by MII-4. Several samples were prepared out of the same solution. To check the reproducibility of observed structures, several samples were prepared and examined. In this experiment a very accurate surface cleaning was achieved and it was confirmed by many repetitions of the preparation that manifest the same morphology. Though each individual sample shows a different surface features the statistical features of the film morphology remain the same.

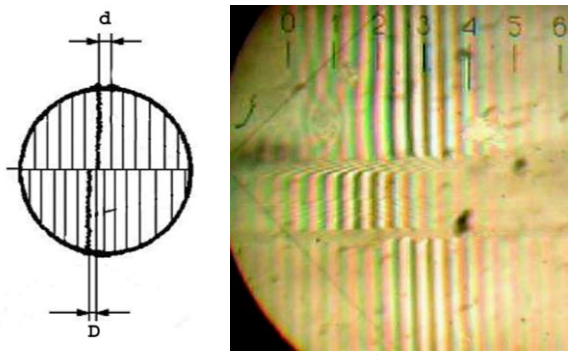


Fig.4. The interference fringe shift D in interferogram introduced by different height of layer. Photo of interferogram obtained by computerized MII-4.

The interferograms of the polymer films spin-coated from solutions with different polymer concentrations taken by a CCD camera are shown in Fig. 5.

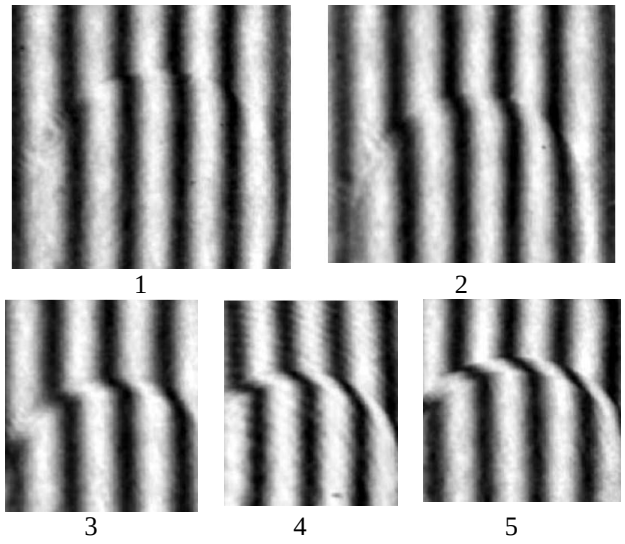


Fig. 5. Interference fringe shift in interferograms introduced by different thickness of layer obtained from following concentrations of PEPC solution: 1 – 2.5 wt%; 2 – 5.0wt%; 3 – 7.5 wt%; 4 – 10.0 wt%; 5 – 12.5 wt%.

The thickness of spin-coated films determined from the fringe pattern in the interferogram was found to be increased with the increase of polymer concentration in solution. It was shown that by raising the polymer concentration from 2.5 to 15.0 wt%, the final film thickness increase from 160 to 960 nm at a spin speed of 3000 rpm. Applied methods of thickness measurements have shown a quasi-linear thickness dependence on polymer concentration. (Fig.6.). Therefore, a smooth polymer films can be fabricated just by controlling the polymer concentration in solution. Applied method for determination of thickness of films in dependence on polymer concentration in solution can be successfully extended to other polymer films in order to obtain required thickness

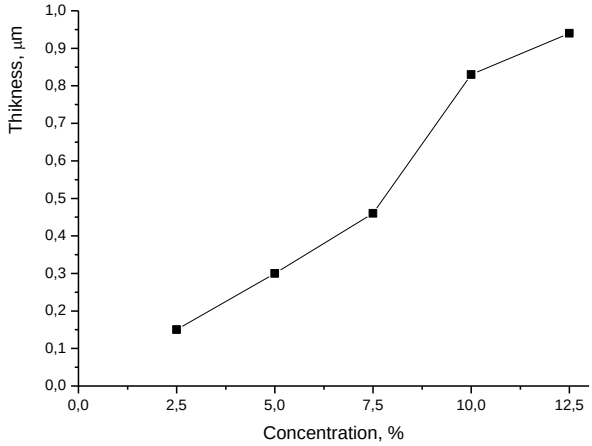


Fig. 6. Thickness as a function of PEPC solution. The thickness was analyzed from the shift of the fringes. One fringe shift corresponds to λ/2.

IV. CONCLUSION

The fabrication of a polymeric films based on PEPC and PETPC by spin-coating method was demonstrated. The thickness of layers was analyzed by interferometric measurements. It was shown, that the thickness of thin polymer films could be analyzed with high resolution by the proposed method. The described methods allow fabricating thin layers by controlling the concentration of polymer in solution and/or spin coating speed and provide accurate thickness measurement by interferometric method.

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