

X-Ray Diffraction Pattern...

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X-Ray Diffraction Pattern and Optical Properties Of Disperse Red-1 Thin Films Deposited By Electric Field Assisted PVD Method

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Abstract. Disperse Red-1 (DR1) films have been prepared by Electric field-assisted Physical Vapor Deposition (E-PVD) method at various external electric field strengths on the ITO substrate. The resulted films were characterized by X-ray diffraction spectroscopy and the optical properties are investigated by Reflectometric and ATR measurements. The XRD data show growing diffraction peaks with increasing electric field corresponding to increasing number of molecules deposited with the head-to-tail stacking along the molecular chain. Further, the reflectometer data show a rising trend of the optical refractive index of the films produced with increasing electric field. This result is consistent with the frequency shift of SPR (Surface Plasmon Resonance) measured by ATR method.

Keywords: Disperse Red-1, E-PVD, electric poling field, SPR.

INTRODUCTION

Organic thin films of excellent optical properties have long been produced and studied for their potential application in integrated optics such as optical switching, optical data storage and information processing [1-5]. While good quality and strong polymer films are most commonly fabricated by spin coating process and to a lesser extent, by other solution based methods [6], the same methods are often not suitable for the smaller organic molecules and an alternative deposition method should be explored.

The Disperse Red 1 compound is among the widely studied azobenzene-based chromophores due to its rich functionalities associated with the conjugated chain structure of the azo molecule. The potential applications of this photoresponsive molecule, together with a whole class of azobenzene-based molecules in nonlinear optics have been proposed by a number of researches [7-10]. For the purpose of device applications, the materials must be prepared in the form of thin films.

In a previous research on the fabrication of small-molecules organic films, it was found that DR1 molecules deposited by means of PVD (Physical Vapor Deposition) method resulted in a free-standing thin film with the molecules oriented perpendicular to the substrate surfaces in an anti-parallel dipole arrangement. It was also found that

the molecules were deposited with regular head-to-tail stacking leading to periodic structure along the resulted molecular chain [11]. It is understood that due to the dipole interaction between the polar DR1 molecules, the deposited molecule may tend to aggregate as indicated in a previous study [12]. Since this effect is likely to affect the lateral structure and property of the deposited molecules, a modification or further improvement of the method is needed.

We present in this report the result of structural and optical study of DR1 thin films deposited on ITO substrate by PVD method under the influence external electric field. This electric field is expected to affect the polar arrangement of the deposited DR1 molecules in the film. Under the assumption of strong anchoring force provided by hydrogen bonding with the ITO substrate, the deposited films are expected to be quite stable and should exhibit a different XRD data for films deposited under different applied electric fields as confirmed in this study. It is further shown that the optical properties of the films also vary with the applied electric field as revealed by reflectometric and SPR measurements.

EXPERIMENTAL

The DR1 (4-[*N*-ethyl-*N*-(2-hydroxyethyl)] amino-4'-nitroazobenzene) powder used in this experiment was obtained commercially from Aldrich. The molecule has a formula weight of 314.34, a melting point of 153 °C with the onset temperature of thermal decomposition at 219 °C as determined by TGA measurement [11]. This molecule is known to have the Donor-Bridge-Acceptor polar structure.

Samples in the form of thin films were deposited on an ITO substrates using VPC-410 Vacuum Evaporator from Ulvac Sinku Kiko, which was operated at about $(2-4) \times 10^{-5}$ torr, with crucible temperature at 168 °C and substrate temperature at 26 °C. The ITO substrate also acts as a counter electrode for the generation of the electric poling field. It was placed at about 10 cm above the crucible with a stainless steel mesh electrode. The films were prepared with various external electric field strengths of 0 MV/m, 0.59 MV/m, 1.9 MV/m, 2.6 MV/m and 3.3 MV/m. The duration of the deposition process of all the thin films was about 1 hour. No further treatment was performed on the deposited films.

The crystallinity or crystal structure were examined on the basis of XRD patterns obtained with PANalytical Diffractometer which was operated with the CuK α ($\alpha=1.540598$ angstrom) X-ray source operated at 40 KV, and 30 mA. The measuring angle was scanned over the range from $2\theta = 3^\circ$ to 30° with a step size of 0.0167 degrees and a time step of 15.240 s.

With given thickness of the ITO substrate (1 μ m), the thickness and refractive index of the films were determined by using reflectometer NanoCalc-2000 VIS which was operated with wavelength 400 nm – 850 nm, single channel 2048 pixel CCD spectrometer.

The frequency shift of SPR dip was measured by means of ATR (Attenuation Total Reflection), which was operated with He-Ne laser ($\lambda=632,8$ nm), with the angle of incidence scanned over the range from 30° to 90° .

RESULTS AND DISCUSSION

The XRD spectra of the thin films deposited on the ITO substrate at various external electric fields are presented in Figure 1, where the background peaks from the substrate appearing at $2\theta > 10^\circ$ were eliminated from the figure. It is observed that there are two visible sharp peaks located at $2\theta = 4.5014^\circ$ and 9.0064° , corresponding respectively to periodic vertical stacking of the DR1 molecules with periodicities of 19.62 \AA and its 2nd order peak. The former is associated with a fully stretched trans-configuration of the molecules in the perpendicular direction. The figure also shows visible increases in the diffraction peaks and their sharpness for larger poling field corresponding to increasing number of molecules deposited with the head-to-tail stacking along the molecular chain. This is consistent with the spectroscopic analysis described earlier [12].

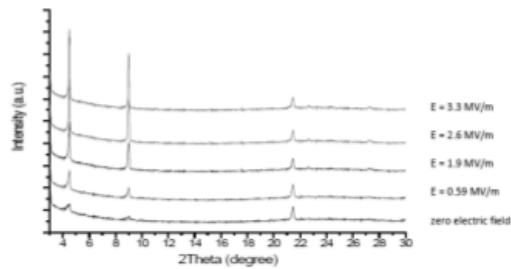


FIGURE 1. XRD pattern of the DR1 films as deposited on ITO substrate for various electric fields

The XRD spectrum of the thin films deposited on ITO substrate surface soon after the deposition along with that measured 3 months later are presented in Figure 2. This figure shows the structural stability of the film implying the strength of the hydrogen bonding.

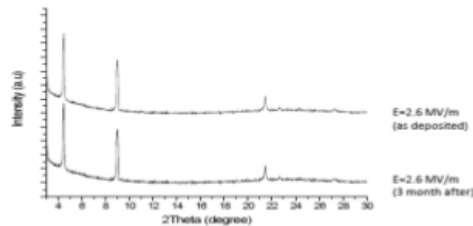


FIGURE 2. XRD patterns of the same films measured with 3 month interval

The result of reflectometry measurement on films deposited in various electric fields are shown in Table 1. This table shows perceptible increases in the refractive index corresponding to increased concentration of deposited DR1 molecules as a result of increased electric field.

TABLE 1. Result of reflectometry measurement on films deposited in various electric fields

Electric Fields E (MV/m)	Refractive Index (n)	Thickness of Films (nm)
0	1.512	126.2
0.59	1.526	126.4
1.9	1.568	126.3
2.6	1.582	126.4
3.3	1.598	126.4

The result of SPR is shown in Figure 3. This figure shows that the SPR dip occurred at increasing incident angle for films deposited in stronger electric field corresponding to higher refractive index of the film. This result is consistent with the theoretical prediction.

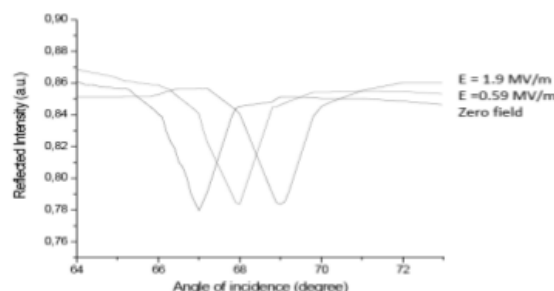


FIGURE 3. SPR result from films as deposited for various external electric fields

CONCLUSIONS

We have shown that the free standing DR1 thin films by E-PVD method exhibit regular vertical stacking of the deposited molecules with high stability, confirming the presence of strong anchoring force provided by hydrogen bonding of the molecule with the substrate. The results of optical measurements also show that increasing electric field leads to increasing refraction index corresponding to increased concentration of the vertically deposited molecules. This is further shown to be consistent with the angular shift of SPR dip measured by ATR.

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