

Full Length Research Paper

Effect of oxygen plasma treatments on the structural and optical properties of polyimide films

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A detailed study of some physical properties of Polyimide films after exposing them to oxygen plasma is presented. The structure of the sample has been analyzed by X-ray diffraction technique (XRD) and is found to be semi crystalline. Thermogravimetric Analysis (TGA) studies revealed that the thermal stability of polyimide films have improved after being exposed to oxygen plasma, at different time intervals, ranging from 1 to 5 min. Optical constants such as refractive index (n), extinction coefficient (K) and complex dielectric constant have been determined. A great dependence of the fundamental optical constants on the exposure time was noticed. Upon exposure to oxygen plasma, decrease in the refractive index was obtained. On the other hand the driving absorption coefficient (α) and consequently the optical band gap have been estimated. The real (ϵ') and imaginary parts (ϵ'') of the dielectric constant have been determined. The dispersion parameters such as E_0 (single-oscillator energy), E_d (dispersive energy) and M_{-1} , M_{-3} (moments) were calculated and discussed.

Key words: Plasma deposition, X-ray diffraction (XRD), crystal structure, optical properties, thermogravimetric analysis (TGA).

INTRODUCTION

Polymer-based optical materials are particularly attractive in integrated optical waveguide devices because they can offer rapid processibility, cost-effectiveness, high yields, and high performance, such as smaller birefringence compared to silica. Furthermore, optical polymers provide an ideal platform for incorporation of more complex material functionalities through different doping or reaction agents, thereby enabling amplification and electro-optic effects to be achieved, once the passive optical polymer technology is established (Ma et al., 2002). Conventional optical polymers, such as polymethylmethacrylate (Kagami et al., 1995),

polystyrene (Gokan et al., 1982) and polycarbonate (Singh et al., 1996) possess different structures and demonstrate many attractive properties, but not all of them possess the needed thermal stability for direct on-chip interconnect applications.

Polyimide has found widespread applications in industry for insulation of electrical machines (Verdianz et al., 2006) as optical materials, for flexible print circuits (Choi et al., 2009). Polyimide based materials have also been applied as electrodes in biomedical micro-devices (Mercanzini et al., 2009).

In this regard, polyamides are a proven class

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of polymers in the microelectronics industry due to their outstanding properties, especially for their high thermal stability. Polyimides (PIs) are a class of representative high performance polymers possessing the cyclic imide and aromatic groups in the main chains. At present, Polyimides (PIs) are extensively used in the microelectronics, photonics, optics, and aerospace industries not only for their very excellent thermal stability but also for their good mechanical properties, low dielectric constant, low coefficient of thermal expansion, and high radiation resistance. Significant effort has been expended to improve their properties further by modification of the chain backbones and higher-order structure control (Atta, 2013; Hasegawa and Horie, 2001). Due to its easy synthesis and attractive optical properties, polyimide is expected to play an important role in the field of organic optical waveguides.

Plasmas generated are widely used to activate polyimide properties. The properties are characterized by the modification of chemical and physical properties which is based on chain scission, the formation of functional groups, and cross-linking (Atta et al., 2014). The energetic electrons, ions, excited atoms, molecules, and UV photons present in the gas phase are responsible for the material modification, but their role in the interaction with the polymer is yet not fully understood due to the complexity of the gas phase and the polymer structure (Hasegawa and Horie, 2001). The polymer films are bombarded by the plasma active species mentioned above just described that leads to chain scissions and polymer degradation. If a plasma reactive gas is used, the appearance of new functional groups on the film will be expected. The most important feature of the plasma technique is the ability to control the modification of the polymer properties. Reactions between plasma species and polymer film make it possible to form polar groups on the film for instance C-O, C=O, O-C=O are chemically reactive and cause an increase of the surface energy and wetting qualities (Atta et al., 2013). Here, the intention of this study is to investigate the effect of oxygen exposure time on the polyimide films has been carried out using X-ray Diffraction (XRD), Thermogravimetric Analysis (TGA) and optical properties.

EXPERIMENTAL

Polyimide (Kapton) films of thickness 160 μm (Du Pont, USA) were used for oxygen plasma exposure process. We use the reactive ion etching (RIE) technique system with the following Oxygen plasma parameters, the working pressure is 7.3×10^{-3} mBar; Oxygen gas flow rate is 25 s/cm; and the incident power is kept constant at 100 W, with a corresponding DC self-bias voltage – 275 V and the exposure time varied from 0 to 5 min. The exposure process was performed using a Radio-Frequency (RF) of 13.56 MHz at National Laboratory of Advanced Technology and Nano-Science (INFMTASC), Trieste, Italy. Before performing the plasma treatment, the polyimide polymeric samples were first cleaned by acetone using ultrasonic technique in order to remove contamination on the polymeric surface. XRD scanning was carried out using fully

computerized X-ray diffractometer (Shimadzu type XD-DI). Filtered copper radiation, $\lambda=1.542\text{\AA}$ was used in this investigation. The X-ray tube was operated at 40 kV and 30 mA anode current throughout the measurements. The pattern was recorded at a scanning rate of $4^\circ/\text{min}$. The above operation conditions were maintained during all the relevant measurements. Hence, it is a plot of the intensity against the angle 2θ , where θ is the reflection angle of X-ray given in Bragg's equation $2d \sin \theta = n\lambda$. The thermal stability of the polyimide films was investigated by Thermo Gravimetric Analysis (TGA) (Shimadzu, Japan). The TGA measurements were carried out in N_2 atmosphere from room temperature to 500°C using a heating rate of $10^\circ\text{C}/\text{min}$.

Optical density of absorbance for the deposited and exposure films was measured over range of 200 to 2500 nm using Kontron – UV/VIS spectrophotometer (made in Switzerland). Different parts along the entire film were measured until good reproducibility of the respective absorption and transmission spectra was achieved. The obtained absorption data against incident light wavelength were used to calculate the electronic absorption coefficient ($\alpha \text{ cm}^{-1}$) and hence the optical energy gap as well as the volume and surface energy loss functions.

RESULTS AND DISCUSSION

Oxygen plasma exposure effects on X-ray diffraction studies

The X-ray diffraction scans of Polyimide films as a function of Bragg angle (2θ) are displayed in Figure 1 for virgin and all oxygen plasma exposure time. The diffraction pattern of virgin polymer clearly indicates that this polymer is semi-crystalline in nature. The X-ray diffraction data of the virgin sample (Table 1) shows three peaks at $2\theta= 14.88^\circ$, 21.78° and 26.255° with lattice spacing $d = 5.94882\text{\AA}$, 4.0773\AA and 3.39162\AA , and the corresponding full widths at half maxima (FWHM) are 2.84° , 3.08° and 2.23° , respectively. The diffraction pattern of virgin sample showed partial crystallinity and the most intense peak appeared at $2\theta= 26.255^\circ$. However, in case of increasing the plasma exposure, a significant change in the diffraction pattern has been observed (Table 1). The diffraction patterns of the virgin sample and exposed samples at different time are quite different and the three original peaks are found at slightly higher angular positions respectively. It is evident that the diffraction pattern of the exposure Polyimide is broader than the virgin one, which indicates a decrease in crystallinity. Similar finding was represented in (Qi et al. 2006 and Shalaby et al. 2013).

As shown in Table 2 by increasing the time of exposure to the oxygen plasma, there is always a possibility of development of strain (stress), which affects the mechanical properties of the samples such as the stability of microstructure, the strain (stress) in a sample can be intrinsic, caused by the conditions prevailing during deposition. On other hand, strain (stress) can be extrinsic to the sample but intrinsic to the composite – substrate system, caused by the difference in thermal expansion coefficient (Bin Ahmed et al., 2012). The

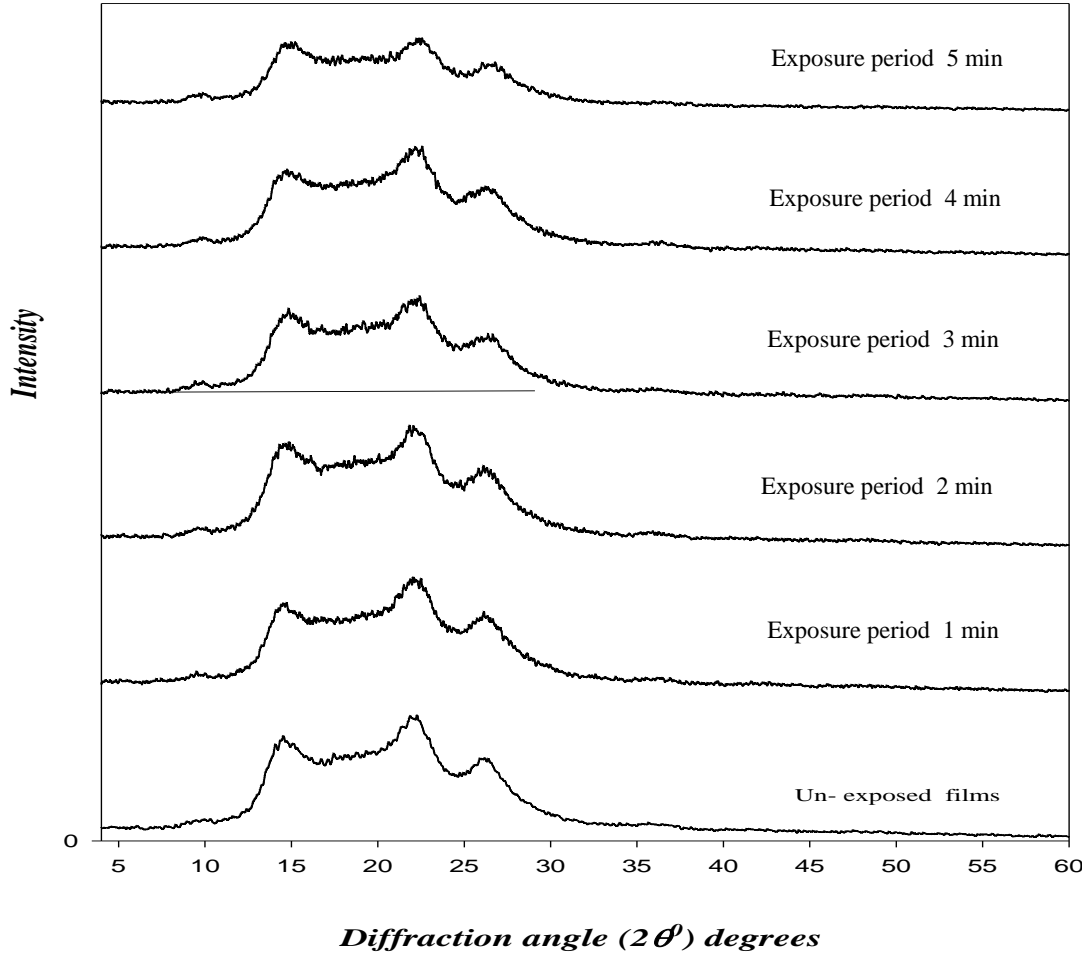


Figure 1. X- ray diffraction patterns spectra for polyimide film with oxygen plasma exposure time.

Table 1. The X-ray diffraction data for PI as a function of plasma exposure time.

Films	First Peak			Second Peak			Third Peak		
	d (Å)	2θ	FWHM	d (Å)	2θ	FWHM	d (Å)	2θ	FWHM
Virgin Film	4.077	21.780	3.08	5.949	14.88	2.84	3.395	26.290	2.88
Exposed 1 min	4.065	21.846	2.93	6.021	14.70	2.29	3.392	26.270	2.26
Exposed 2 min	4.056	21.543	2.92	6.062	14.60	2.14	3.390	26.255	2.50
Exposed 3 min	4.001	22.340	2.82	6.106	14.53	2.11	3.387	26.243	2.22
Exposed 4 min	3.813	22.200	2.32	6.155	14.38	2.04	3.351	26.230	2.23
Exposed 5min	3.781	22.109	2.28	6.197	14.26	1.56	3.317	26.060	1.96

crystallite size and the residual strain are calculated from full width at half maximum (FWHM) using the relation (1) for all phases

$$P = \frac{K\lambda}{\beta \cos \theta} \beta = \frac{\lambda}{D \cos \theta} - \varepsilon \tan \theta \quad \delta = \frac{1}{D^2} \quad (1)$$

where (β) is full width at half maxima (FWHM) in radians, the wavelength, λ of X-ray beam (1.5418 \AA), P the crystallite size in \AA and K is a constant for most cases it is close to 1. Assuming $K = 1$ in Equation (1), the crystallite sizes were calculated for strong peaks in all samples as given in Table 2. However, particularly in case of solid polymeric materials, the exact nature of the interrelation between spacing, crystallite size and the

Table 2. The crystallite size for PI as a function of plasma exposure time.

Films	First Peak			Second Peak			Third Peak		
	P(m) E-09	δ E-16	Δ (ϵ) E-2	P(m) E-09	δ	Δ (ϵ) E-2	P(m) E-09	δ	Δ (ϵ) E-2
Virgin Film	2.746	13.267	1.783	2.949	11.501	2.423	2.961	11.407	1.374
Exposed 1 min	2.884	12.030	1.693	3.651	7.500	1.980	3.773	7.025	1.194
Exposed 2 min	2.883	12.021	1.717	3.912	6.534	1.861	3.412	8.597	1.079
Exposed 3 min	3.002	11.100	1.591	3.973	6.335	1.841	3.841	6.779	1.066
Exposed 4 min	3.647	7.517	1.318	4.103	5.941	1.802	3.823	6.841	1.061
Exposed 5min	3.711	7.262	1.300	5.365	3.457	1.389	4.349	5.288	0.944

Table 3. The interchain distance (r), interplaner distance (d) and the distortion parameter (g) for PI as a function of plasma exposure time.

Films	First Peak			Second Peak			Third Peak		
	r E-10	d E-10	g E-10	r E-10	d E-10	g E-10	r E-10	d E-10	g E-10
Virgin Film	5.101	4.081	0.279	7.442	5.954	0.38	4.237	3.390	0.215
Exposed 1 min	5.085	4.068	0.265	7.532	6.026	0.310	4.240	3.392	0.169
Exposed 2 min	5.156	4.124	0.269	7.584	6.067	0.292	4.243	3.394	0.187
Exposed 3 min	4.974	3.979	0.249	7.620	6.096	0.288	4.245	3.396	0.166
Exposed 4 min	5.005	4.004	0.206	7.699	6.159	0.282	4.247	3.397	0.167
Exposed 5 min	5.026	4.021	0.204	7.764	6.211	0.218	4.274	3.419	0.148

degree of disorder is yet to be understood in proper perspective. These bands agree with those elsewhere reported (Liu et al., 2002; Saha et al., 2009)

The strain (ϵ) was calculated from the slope of the ($\beta \cos \theta$) versus ($\sin \theta$) plot using the Equation (1). The dislocation density (δ), defined as the length of dislocation lines per unit volume of the crystal, was evaluated from the Equation 1. Table 3 showed that the decrease in X-ray intensity may be resulting from grain size. This appears as boarding beaks, on the other hand, decreasing in the interchain distance (r) and the interplaner distance in the peaks lead to decreasing in crystallinity.

$$r = \frac{5\lambda}{8\sin \theta} \quad d = \frac{\lambda}{2\sin \theta} \quad g = \frac{\beta}{\tan \theta} \quad (2)$$

Where interchain distance (r) and d is interplaner distance. Also, g is distortion parameter. From Table 2 it has been observed that the crystallite size decreases but strain and dislocation density in the samples increase with the increase in the time of exposure by oxygen. However, the decrease in intensity and the shift in angular position towards the higher angle can be explained by the decrease in lattice spacing (r and d). It also suggests a change in crystallite size of the samples exposed to oxygen plasma at different times. The crystallite sizes of the virgin and irradiated Polyimide-polymer have been calculated using Scherrer's equation

(Equation 1).

Thermogravimetric analysis (TGA)

Oxygen plasma exposure effects on the thermal stability of polymer were evaluated by TGA. TGA is a technique used to accurately track the *in situ* weight changes of a sample during the heating process, thereby providing information on thermal degradation. Figure 2a, b shows the TGA curves for deposited films and also exposed film at period 5 min. From Figure 2a, b, it is evident that the thermal oxidative degradation of the samples involved two different steps in the whole process. This is probably because the first step at 300°C (about 1.5% weight loss in TGA curve at this temperature) resulted in some decomposition and consequently hindered the crystallization of polyimide on the second step. It is clear that this polyimide exhibited excellent thermal stability, since no obvious weight loss was found in the differential thermogravimetric (TGA) curve below 500°C. This indicated that crystallization of the polyimide occurred rapidly during the thermal imidization of precursor, and semicrystalline polyimide can be readily obtained. The increase in the weight residues above 500°C illustrates successful incorporation of higher exposure period time (5 min of oxygen plasma) as shown in Figure 2b, and ultimately increases in thermal stability. The improvement of the thermal stability of the polyimide by increasing plasma exposure can be based on the fact that these

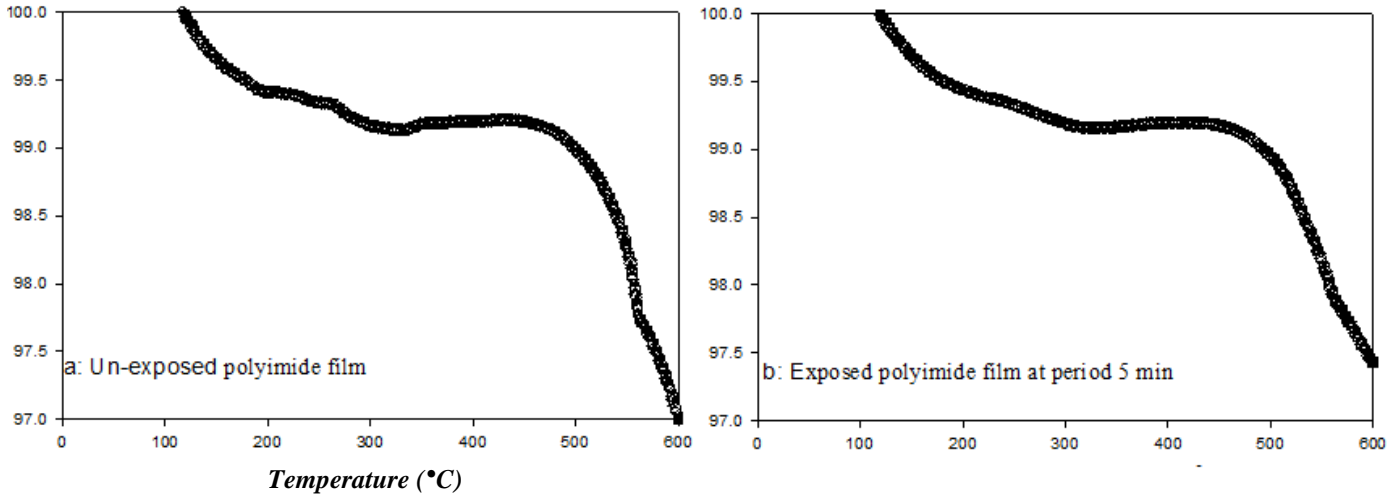


Figure 2. TGA curves for a-unexposed and b-exposed polyimide film with 5 min oxygen plasma.

materials have inherently good thermal stability and also due to the strong interaction/chemical bonding that exposed the polyimide film. These results suggest that molecules are bonding to the polyimide through reaction with H bonds and possibly through functionalities resulting from oxidative degradation of the polyimide film. The difference is mainly attributable to the different responses of the polymers to the evolution systems. One of the most important key parameters is the TGA of the optical device fabrication.

UV-Visible spectrophotometric measurements

Figure 3 shows the transmittance spectra, obtained at room temperature and near normal incidence for virgin film and different oxygen plasma exposure (1, 2, 3, 4 and 5 min) in the visible region. As can be seen from this Figure 3, the obtained films are characterized with high transmission greater than 60% for 600 nm depending on the film exposed by oxygen.

The transmittance decreases as the exposure time increases at wavelength $\lambda = 600$ nm and it can be positively concluded that the material is of highly absorbing nature. The absorption coefficient was calculated from transmittance measurements with the aid of the expression (Kiryong and John, 2002).

$$\alpha = \frac{2.303}{d} \log_{10}\left(\frac{1}{T}\right) \tag{3}$$

where d is the thickness of the film and T is the transmittance. If the sample faces are parallel, which is in most of the current case, multiple reflections have to be taken into account within the sample thickness. In this work, the polymer films are sufficiently thick compared to

the beam wavelength to neglect the interference phenomena. The air/polymer interface transmission T is expressed as functions of refractive index (n) and extinction coefficient (K) (Soldera et al., 200).

$$T = \frac{4n}{(n+1)^2 + k^2} \tag{4}$$

Once the measurements of value T is done, an iterative procedure to calculate n and K is initiated. When the absorption α is known, the extinction coefficient K can be found the relation $K = \alpha\lambda/4\pi$. It is found that the value of n decreases, while the value of K increases with the photon energy at different time exposure. Oxygen plasma exposure has effect on the spectral dependence of (n) on wavelength λ for films as shown in Figure 4. The high-frequency properties for virgin film and films exposure by oxygen plasma could be treated by using Cauch’s formula (Jenkins et al., 1985). It was found that the high-frequency dielectric constant ϵ_{∞} can be determined from the plots of $(n^2 - 1)^{-1}$ versus λ^{-2} as shown in Figure 5.

The Cauch’s formula describes the contribution of free carriers and lattice modes. To obtain information about direct or indirect transitions, the optical band gap was determinate from analysis of the spectral dependence of the absorption near the fundamental absorption within the framework of an electron theory (Kumar et al., 2000). Thus, the electronic transition between valance and conduction band is given by

$$\alpha h\nu = A(h\nu - E_g)^s \tag{5}$$

Where s has discrete values like 1/2, 3/2, 2 or more

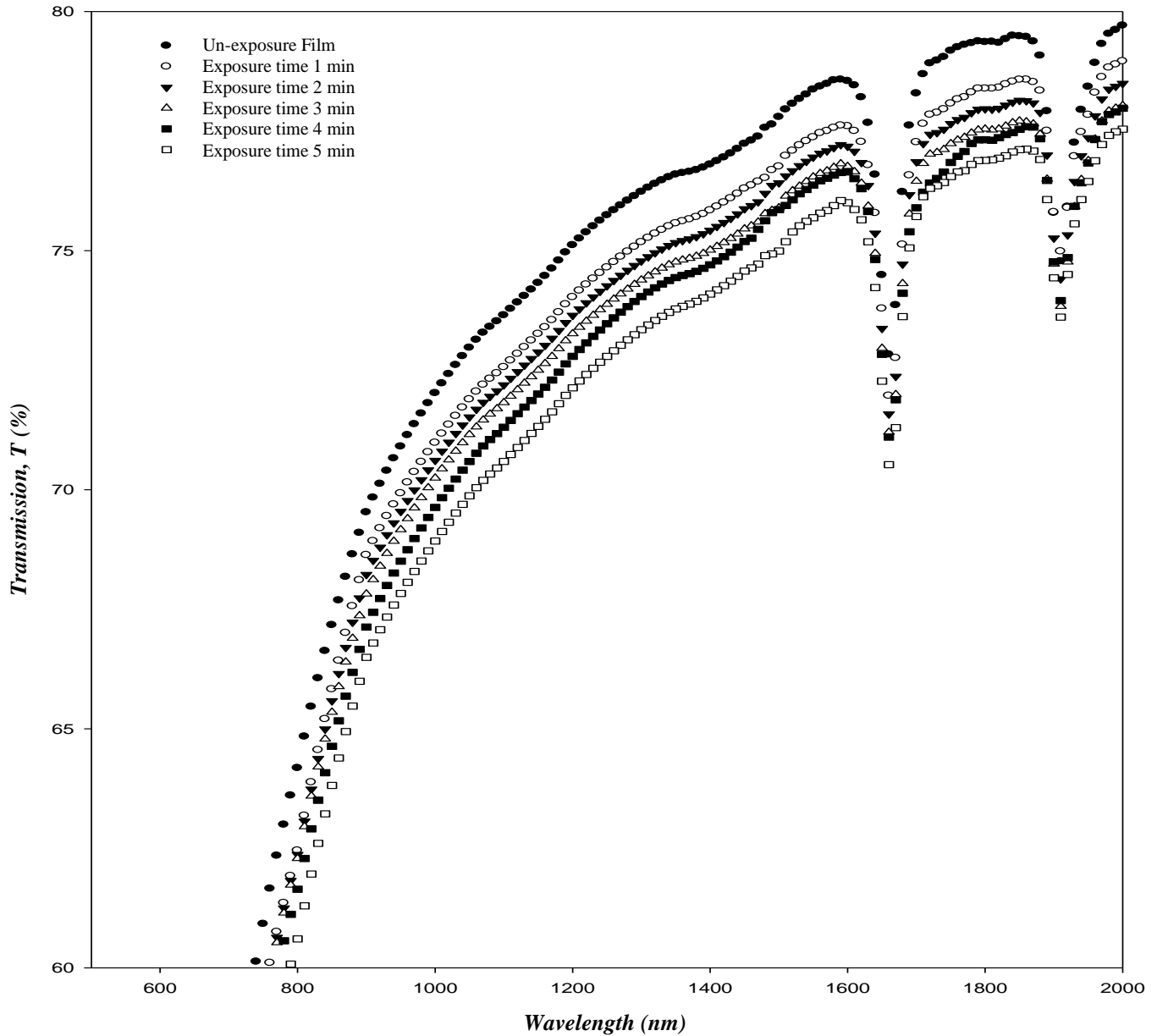


Figure 3. Transmission spectra for polyimide film with oxygen plasma exposure time.

depending on whether the transition is direct or indirect and allowed or forbidden. In the equation 5, A is constant, h is Planck's constant, E_g the optical band gap and $h\nu$ the energy photon. Plotting $(\alpha h\nu)^{1/2}$ as a function of the photon energy $h\nu$ for the curves (un-exposed and exposed films) is shown in Figure 6. This linear region characterizes an optical gap E_g according to Equation 5. The linear region in Figure 6 is used to obtain the values of optical band gaps (namely E_g). The values of E_g for all studied films are calculated and listed in Table 4. It is

clear that the Polyimide film with thickness (0.160 mm) shows an energy gap E_g . And also, all films revealed an additional energy gap E_g at higher wavelength due to the decreased grain size of the higher time exposure films and also results in the decrease in crystalline nature which in turns decrease the band gap. Where, the peaks in UV-Vis region have generally interpreted in terms of $\pi \rightarrow \pi^*$ transition between bonding and antibonding molecular orbital. Assuming that the frequency is relatively, the real part ϵ' and imaginary part ϵ'' . Parts of the complex dielectric constant can be written according

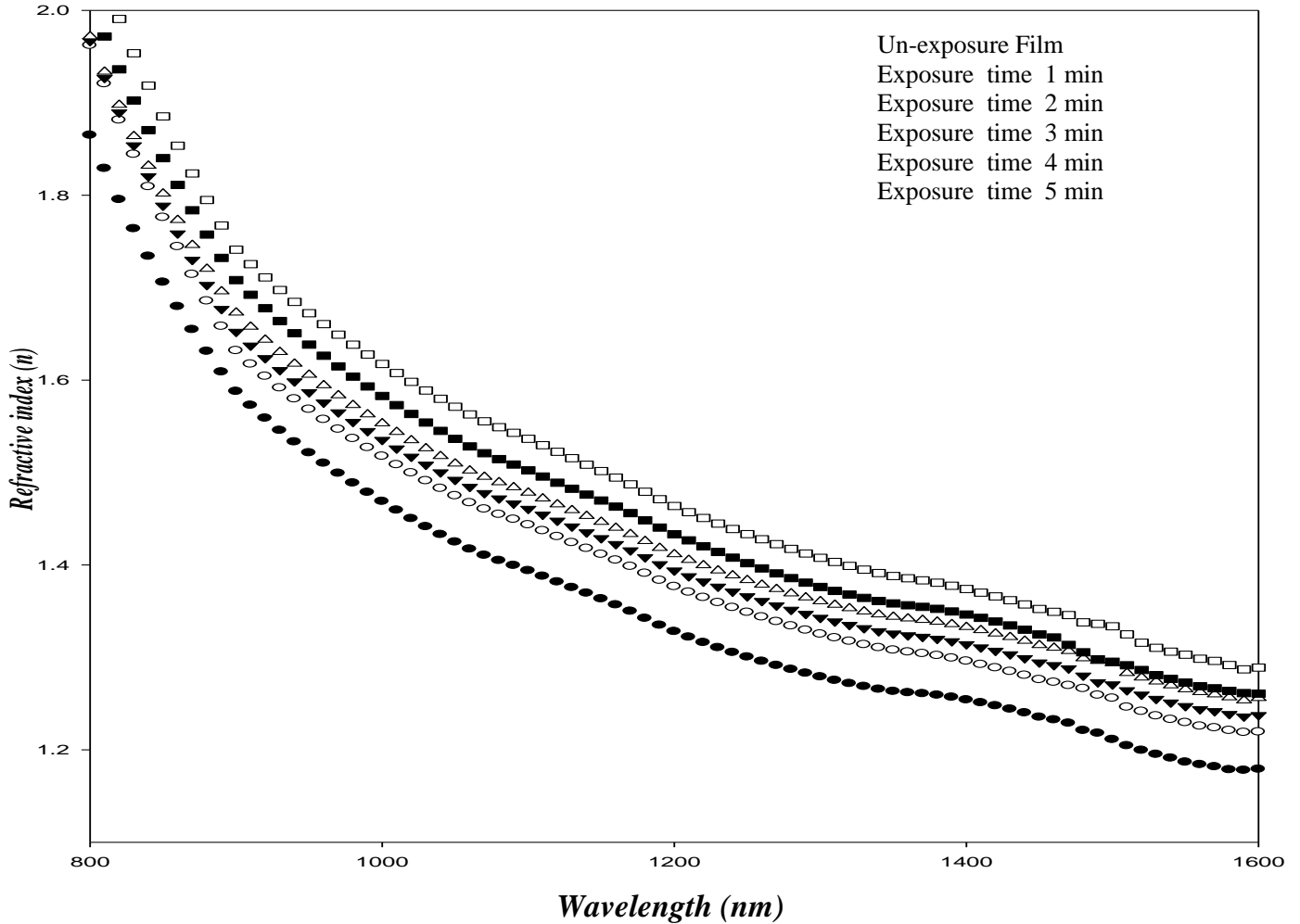


Figure 4. Wavelength dependence of the refractive index n with oxygen plasma exposure time for polyimide film.

to the following relations (Wakkad et al., 2000).

$$\epsilon'' = n^2 - K^2 = \epsilon_\infty - \frac{e^2}{4\pi^2 C^2 \epsilon_0} \frac{N}{m^*} \lambda^2 \tag{6}$$

and

$$\epsilon'' = 2nK = \frac{\epsilon_\infty \omega_p^2}{8\pi^2 C^3 \tau} \lambda^3 \tag{7}$$

where

$$\omega_p = \left[\frac{e^2 N}{\epsilon_0 \epsilon_\infty m^*} \right]^{1/2} \tag{8}$$

Where ω_p is the plasma resonance frequency for one kind of the free carriers, ϵ_∞ is the high frequency dielectric

constant, e is the electronic charge, C is the velocity of light, ϵ_0 is the free space dielectric constant, N/m^* is the ratio of free carrier concentration (N) to the free carrier effective mass (m^*) and τ is the optical relaxation time.

Equations (6) to (8) were employed to determine the parameters ϵ_∞ , ω_p and N/m^* . The values ω_p and N/m^* decreased by increasing oxygen plasma exposure time from 1 up to 5 min as shown in Figure 7. On the other hand, the shift of plasma edge towards higher frequency in the present case could be attributed to the relatively higher value of carrier concentration as mentioned before. This could be accounted for by the generation of excess electronic delocalized states.

The refractive index and the single oscillator parameters were calculated and discussed in terms of the Wemple-DiDmenico model (Wemple, 1973). That the parameters E_o , E_d are calculated by know oscillator energy E_o which is independent of the scale of ϵ and is consequently an “average” energy gap, where as E_d depends on the scale of ϵ and thus serves as an

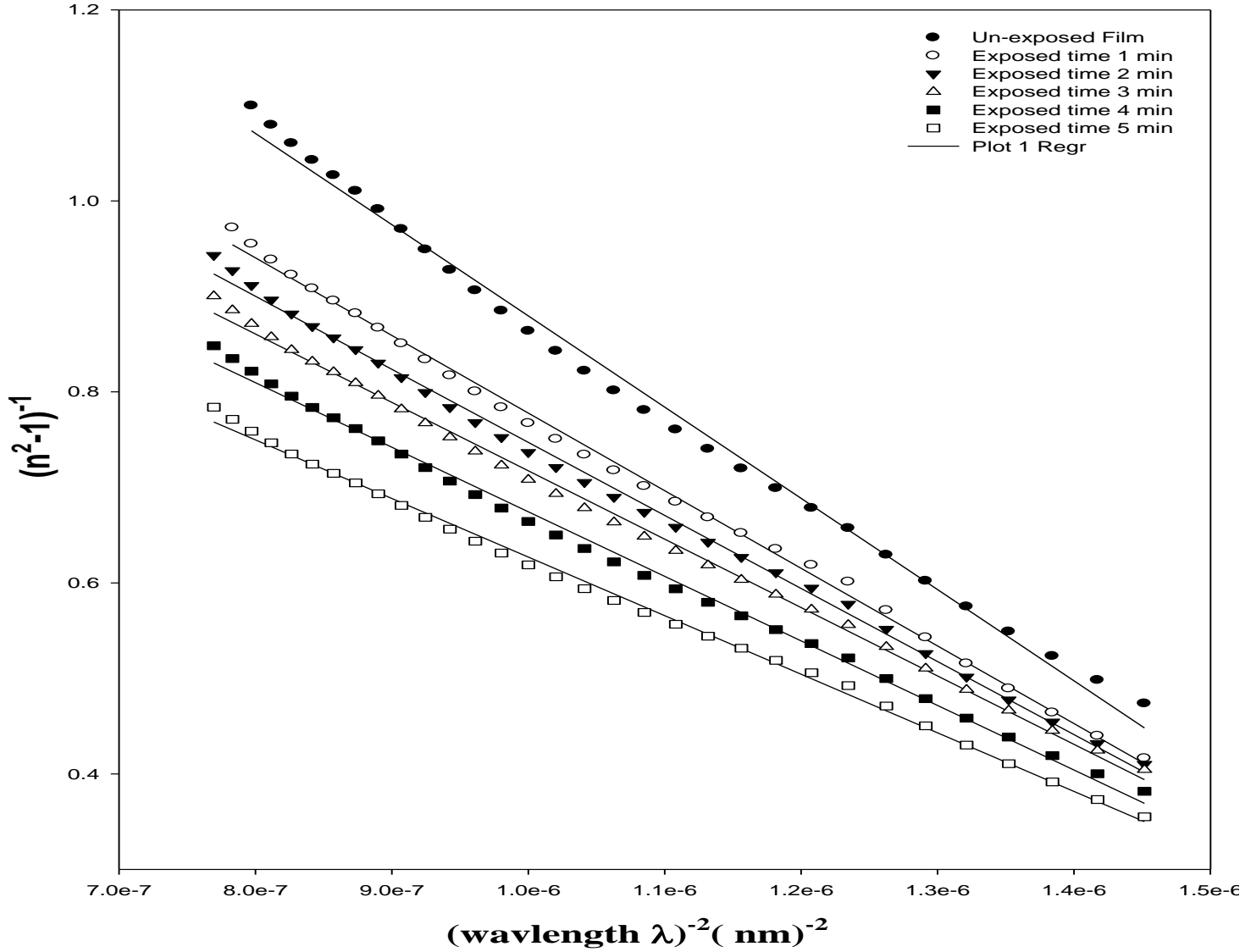


Figure 5. Plots $(n^2 - 1)^{-1}$ against square wavelength (λ^{-2}) with oxygen plasma exposure time for polyimide film.

interband strength parameters. Since the M_0 and M_d moments are involved most heavily near the interband absorption edge.

The dispersion of refractive index below the interband absorption edge has been obtained according to the single oscillator model (Wemple, 1973; Wemple and Didomenico, 1971).

$$n^2(E) = 1 + \left[\frac{E_0 E_d}{E_0^2 - E^2} \right] \quad (9)$$

where the parameters E_0 , E_d are the single-oscillator energy and dispersive energy respectively. By plotting $(n^2 - 1)^{-1}$ versus E^2 and fitting a straight line as shown in Figure 8, E_0 and E_d are determined directly from the

gradient, $(E_0, E_d)^{-1}$ and the intercept (E_0 / E_d) , on the vertical axis. Therefore, in order to account for the trend of E_d , it is suggested that the observed increase in E_d is with increasing exposed time is primarily a crosslinking. It is shown that a peculiarity for E_0 , E_d . On the basis of the above-mentioned model the single-oscillator parameters E_0 , E_d are related to the imaginary part, ϵ'' of the complex dielectric constant, the M_{-1} , M_{-3} moments of polyimide can be derived from the relations (Yakuphanoglu et al., 2004)

$$E_0^2 = \frac{M_{-1}}{M_{-3}} \text{ and } E_d^2 = \frac{M_{-1}^3}{M_d} \quad (10)$$

So that the higher energy interband transitions that influence M_{-1} have little influence on E_d . However, within

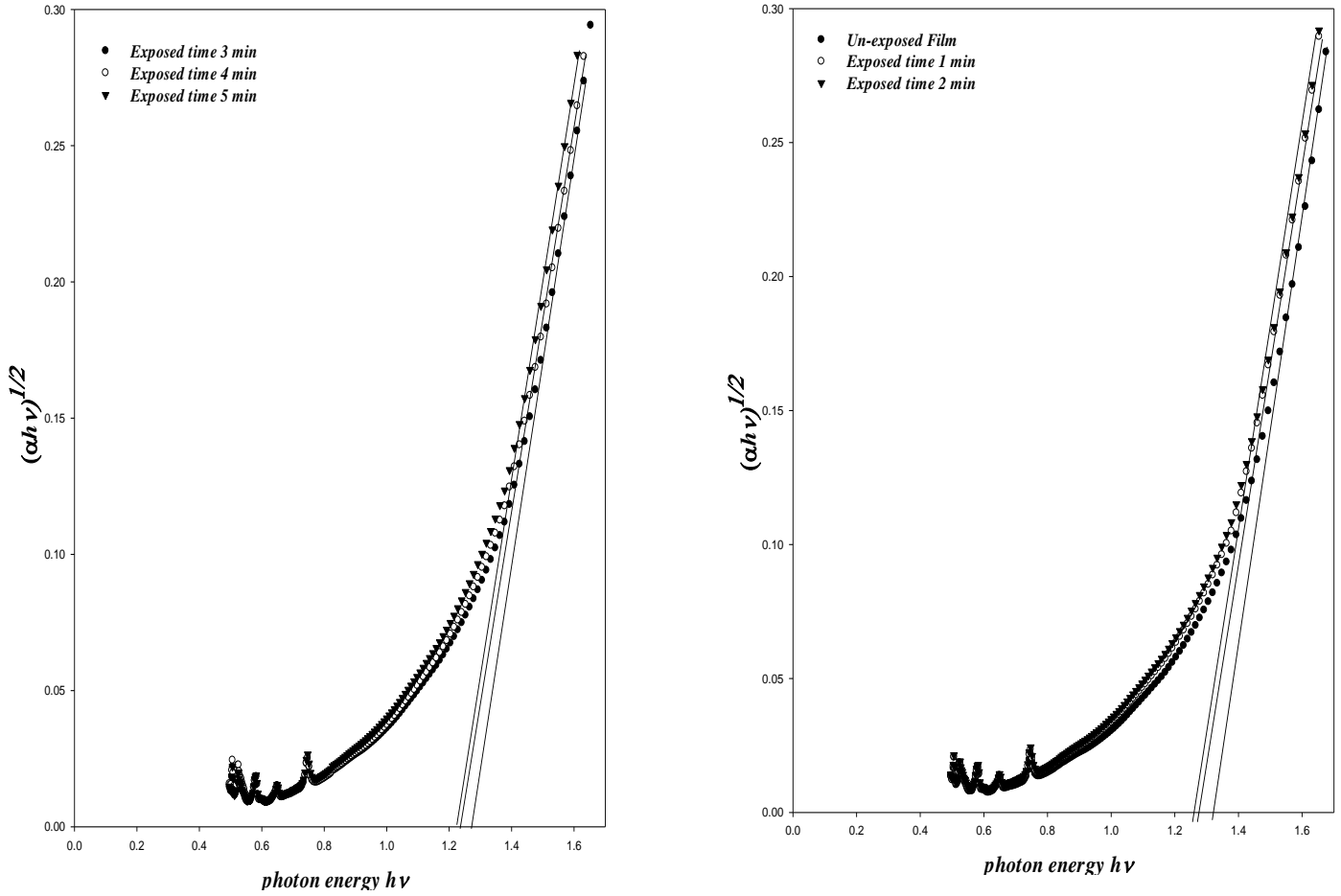


Figure 6. The optical absorption coefficient edge plotted as $(\alpha h\nu)^{1/2}$ versus photon energy ($h\nu$) for un-irradiated and irradiated polyimide films with oxygen plasma exposure time.

Table 4. The optical band gap E_g for PI as a function of plasma exposure time.

Films	ϵ_∞	$\omega_p (10^{12}/s)$	$N/m^* (10^{17}/cm^3)$	$E_g (eV)$
Virgin Film	1.836	9.831	6.911	1.328
Exposed 1 min	1.591	9.739	5.879	1.281
Exposed 2 min	1.512	9.691	5.534	1.266
Exposed 3 min	1.434	9.649	5.181	1.260
Exposed 4 min	1.351	9.639	4.890	1.241
Exposed 5 min	1.241	9.581	4.438	1.219

the confines of the constant conductivity model the equation 10 (M_{-1} / M_{-3}) relates E_d to the sum-rule moments M_{-3} and M_{-1} . As a result, the dispersion energy may depend upon the detailed charge distribution within each unit cell, consequently, would then be closely related to chemical bonding may lie within a nearly localized orbital theory. Also, it was observed from Table 5 that E_0 was increased while E_d was increased with the increasing plasma oxygen exposure time. Therefore, it is

suggested that the observed in E_d may be accounted for exposure sensitivity trend induced by increasing plasma oxygen exposure time.

Conclusions

1) XRD obtained for virgin and polyimide films were exposure by oxygen plasma as confirmed by the semi

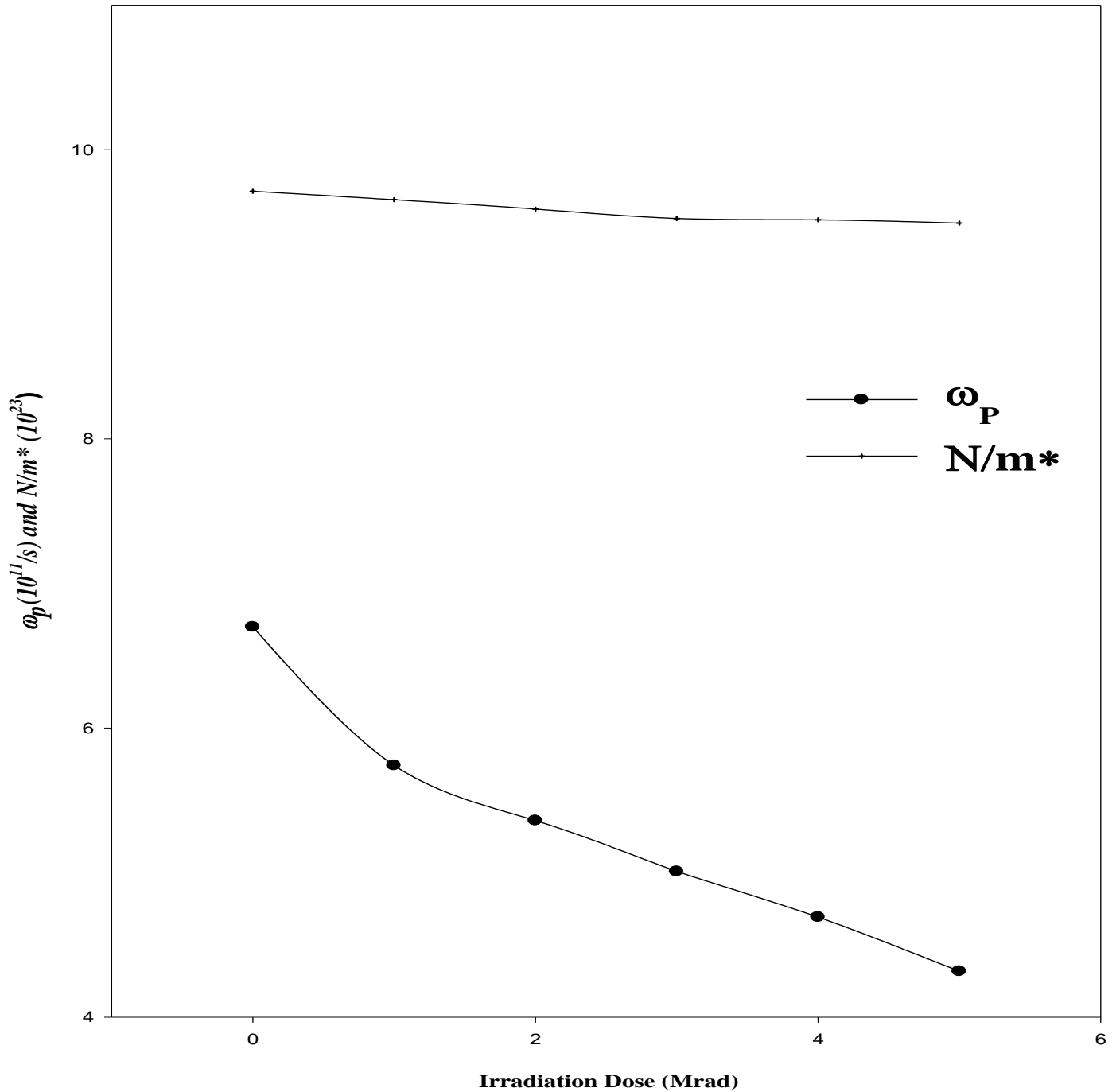


Figure 7. Variation of ω_p (plasma resonance frequency) with oxygen plasma exposure time for polyimide film.

crystalline nature for different films.

2) The intensity of these peaks for the exposed films is smallest than that of virgin film, indicating that the crystallinity becomes little obvious by increasing molecular weight.

3) The TGA curves of the virgin polyimide and exposure films at various time showed that the thermal stability

increased with increasing time exposure.

4) Oxygen plasma exposure of polyimide films in the different time range 1 to 5 min has been investigated as a potential technique of refractive index modulating optical elements. There was an increase in the refractive index via increasing plasma exposure in the dispersion energy.

5) Single oscillator energies in the polyimide films were significantly influenced by Oxygen plasma effects. It was

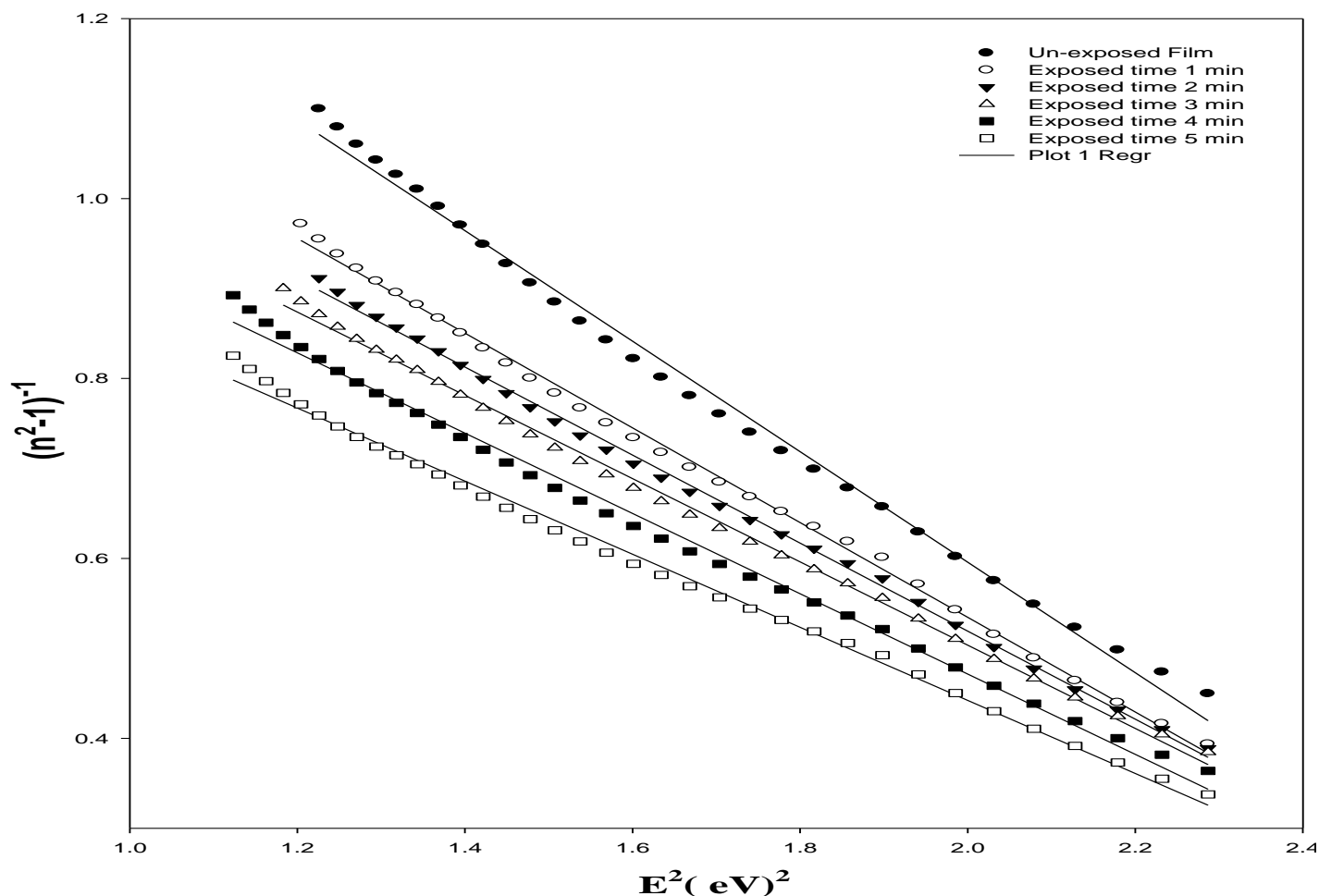


Figure 8. Plots $(n^2 - 1)^{-1}$ against square photon energy (E^2) with oxygen plasma exposure time for polyimide film.

Table 5. The single-oscillator energy and dispersive energy parameters for PI as a function of plasma exposure time.

Films	E_o	E_d	M_{-1}	M_{-3}
Virgin Film	1.723	0.945	0.548	0.185
Exposed 1 min	1.736	1.094	0.631	0.209
Exposed 2 min	1.750	1.168	0.668	0.218
Exposed 3 min	1.757	1.229	0.699	0.227
Exposed 4 min	1.758	1.274	0.725	0.235
Exposed 5 min	1.765	1.395	0.790	0.254

an increase in the dispersion energy and discussed in terms of the Wemple-DiDmenico model.

6) High frequency dielectric constant (ϵ_{∞}) and N/m^* ratio of free carrier concentration (N) to the free carrier effective mass (m^*) are significantly affected by the change with increasing plasma exposure time.

7) The effect of Oxygen plasma exposure on the optical band gap obtained. Moreover, there was a decrease in the optical energy gap with increasing exposure time up

to 5 min.

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Conflict of Interest

The authors have not declared any conflict of interests.

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