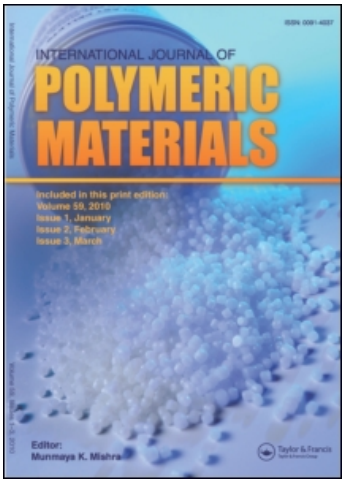

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γ-Irradiation Effects on Opto-Thermal and -Mechanical Properties of PET and PETG Fibers

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γ-Irradiation Effects on Opto-Thermal and -Mechanical Properties of PET and PETG Fibers

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Changes in opto-thermal and -mechanical properties of polyethylene terephthalate and glycol-modified polyethylene terephthalate (4 wt% of ethylene glycol) subjected to γ-irradiation at doses up to 30 Mrad and a dose rate of 138.8 rad/sec, were investigated by interferometry. Empirical formulae are suggested to correlate the opto-thermal and -mechanical properties of the γ-irradiated fibers with the applied dose. Microinterferograms and relationships between the parameters obtained are given for illustration.

Keywords: γ-radiation dose, birefringence, copolyester fiber, interferometry, mono-polyester, opto-mechanical, opto-thermal, refractive indices, structural parameters

INTRODUCTION

Radiation processing has been demonstrated to be a very effective means of improving end-use properties of various polymers on a large commercial scale. It is a well-established economical method of precisely modifying the properties of bulk polymer resins and formed polymer components. The reactions of crosslinking, degradation and grafting on polymers initiated by radiation have found many useful applications in plastic and rubber materials. Important properties of polymer materials, such as mechanical properties, thermal stability, chemical resistance, melt flow, processability and surface properties can be significantly improved by radiation processing [1].

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The different responses to radiation by different polymers are intrinsically related to the chemical structures of the polymers. Product improvements obtained by irradiation include increased tolerance to high temperature environments and overloaded conductors, fire retardation, increased abrasion resistance and tensile strength, reduction in cold flow, increased resistance to solvents and corrosive chemicals as well as some other important characteristics [2]. Irradiated wires are commonly used in automobiles, military vehicles, aircraft, spacecraft and many other applications where high performance is required.

There are several studies on the effect of radiation such as fast electron, gamma ray, and low-energy ions on poly(ethylene terephthalate) (PET). Buttafava et al. studied the effects of gamma irradiation on PET by positron annihilation lifetime spectroscopy at doses up to 1.1 MGy and a dose rate of 1.08 kGy/h [3]. Radiation-modified PET can be applied in many fields, including biomedicine as well as to improve adherence for metalized polymers [4]. It is known that 1.5 MGy produces noticeable changes in PET and severe damage is achieved at doses higher than 30 MGy [5].

The refractive indices and birefringence of a polymer are parameters sensitive to structure. Birefringence is a measure of the total molecular orientation of a polymer. Previous authors utilized interferometric and other tools to study the effect of irradiation on polymers [6–10]. Hamza et al. suggested the possibility of using refractive indices and birefringence changes in the fiber as radiation dose sensors [7–9].

In this paper, computer-aided fringe field interference technique is used to investigate the effect of γ-irradiation on the refractive indices, birefringence and refractive index profile of monopolyester (PET) and copolyester (PETG) fibers (polyethylene glycol-modified polyethylene terephthalate (4% wt polyethylene glycol). Both opto-thermal and -mechanical devices are attached to the two-beam polarizing interference microscope for studying the effect of γ-irradiation at different doses (5, 10, 15, 20, 25, and 30 Mrad) on the opto-thermal and -mechanical properties of irradiated PET and PETG fibers using the fringe field interference method. The refractive indices, birefringence, refractive index profile, and some structural parameters of irradiated PET and PETG fibers at different temperatures and draw ratios are determined for the given radiation dose.

EXPERIMENTAL

Optical Set-Up

Figure 1 shows the optical set-up for producing two-beam fringes in transmission, which is designed and described by Pluta [11]. The

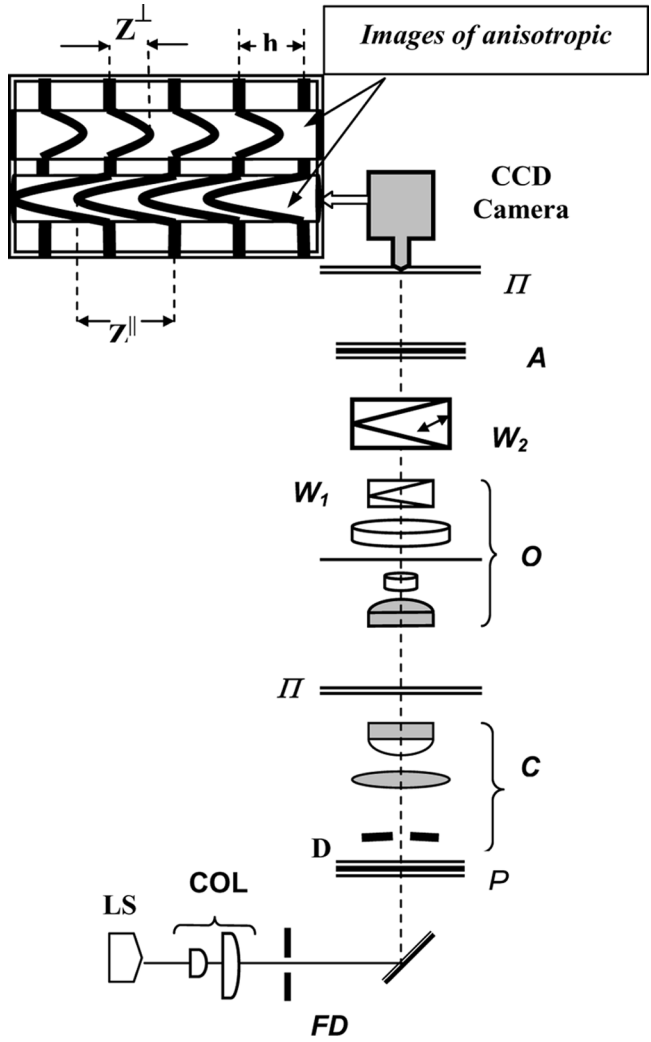


FIGURE 1 Schematic diagrams of experimental setup of the Pluta polarizing interference microscope: LS – light source, Col – collimator, FD – field diaphragm, P – polarizer, D – regular slit diaphragm, Π – object plane, W_1 – rotatable Wollaston prism, W_2 – tube Wollaston prism, A – analyzer, Π\ – image plane.

measuring technique consists of variable-wavefront shear birefracting microinterferometer (Pluta polarizing interference microscope Biolar PI) for transmitted light, CCD camera and PC computer. The output field of the microscope is scanned by CCD camera and the interference

image is automatically captured and processed. The Pluta polarizing interference microscope attached to an opto-thermal device is used to determine the refractive indices and birefringence of irradiated PET and PETG fibers at different temperatures [12]. Also, the Pluta polarizing interference microscope attached to an opto-mechanical device is used for determining the optical and structural parameters of stretched irradiated PET and PETG at different draw ratios [13].

Irradiation Procedure

Irradiation was carried out using a ^{60}Co source of 16134 Ci gamma cell located in the technology radiation center in Egypt. Samples were wrapped in a paper and exposed to doses from 5 Mrad up to 30 Mrad in air; the dose rate was maintained at 138.8 rad sec^{-1}.

Measurement of Optical Properties of the Irradiated PET and PETG Fibers

The refractive indices $n^{\parallel}$, $n^{\perp}$ and the birefringence of the irradiated monopolyester (PET) and copolyester (PETG) fibers with different radiation doses, 5, 10, 15, 20, 25, and 30 Mrad were determined using computer-aided Pluta polarizing interference microscope (Figure 1).

The irradiated PET and PETG fibers were immersed separately in two different liquids of refractive indices $n_L = 1.6490$ and $n_L = 1.5530$ at temperature 25°C when the light vibrates parallel and perpendicular to the fiber axis, respectively. Figures 2 and 3 display the microinterferograms of the differentially sheared images (parallel) of monopolyester (PET) and copolyester (PETG) fibers irradiated with different radiation doses. Monochromatic light of wavelength 546 nm was used. The upper and lower images represent the interference fringe displacement inside the fiber images for plane-polarized light vibrating parallel and perpendicular to the fiber axis, respectively. From these microinterferograms, one can notice that the change in the fringe deflection in the PET and PETG images increases with increasing radiation dose.

The fringe deflections (Z^i) in the duplicated images were measured automatically using a prepared software program. The mean directional refractive indices ($n^{\parallel}$and $n^{\perp}$) can be determined using a the following equation [11]:

$$n^i = n_L \pm \frac{Z^i}{h}\frac{\lambda}{d}, \tag{1}$$

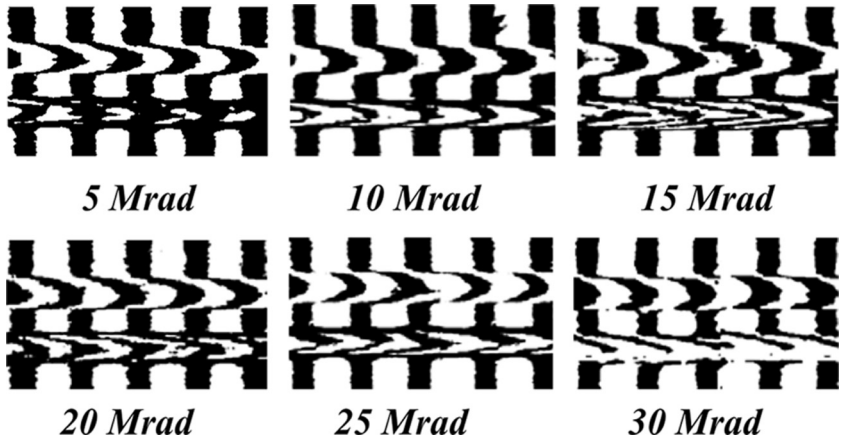


FIGURE 2 Variation of fringe deflection in upper image of the irradiated PET fiber as function of the radiation dose when the plane-polarized vibrating parallel to fiber axis, monochromatic light of wavelength 546 nm, $n_L = 1.553$ and temperature 25°C are used.

where i denotes the state of polarization of the light used (|| parallel or $\perp$ perpendicular to the fiber axis under study), n_L is the refractive index of the immersion liquid, d is the fiber thickness, h is the interfring-spacing, and Z^i is the fringe deflection in the fiber image.

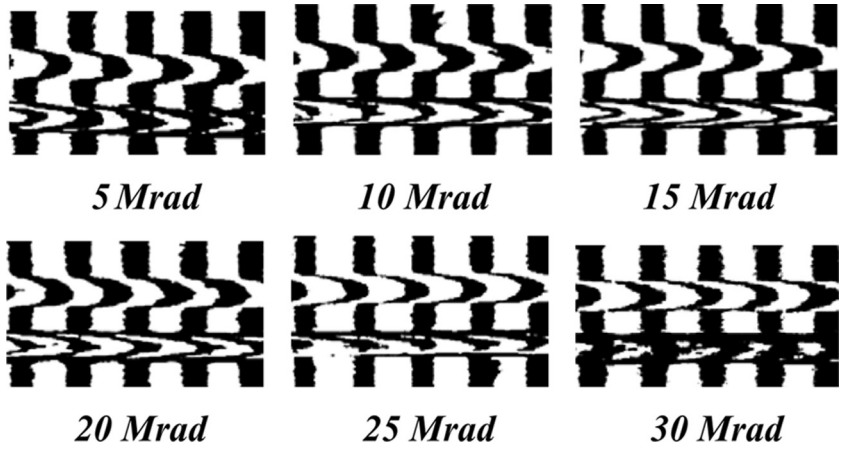


FIGURE 3 Variation of fringe deflection in upper image of the irradiated PETG fiber as function of the radiation dose when the plane-polarized vibrating parallel to fiber axis, monochromatic light of wavelength 546 nm, $n_L = 1.649$ and temperature 25°C are used.

Figure 4 illustrates the variation of the mean refractive indices ($n^{\parallel}$ and $n^{\perp}$) and the birefringence ($\Delta n = n^{\parallel} - n^{\perp}$) at different doses for the two irradiated samples PET and PETG at room temperature (25°C). Also, it shows that the mean refractive indices ($n^{\parallel}$ and $n^{\perp}$) and the birefringence of the irradiated PET and PETG increase linearly with the increasing radiation dose. This means that both the mean refractive indices and birefringence show linear dependence up to 30 Mrad. This agrees with the previous work [9]. An empirical formula is suggested for correlating the optical properties of fibers with the applied dose (ξ) as follows:

$$\eta = \sigma\xi + \eta_o, \tag{2}$$

where η denotes $n^{\parallel}$, $n^{\perp}$ and Δn, σ is a constant and η_o denotes $n_o{}^{\parallel}$, $n_o{}^{\perp}$ and Δn_o at radiation dose ($\xi = 0.0$).

The values of σ and η_o of PET and PETG fibers were calculated and the results are shown in Table 1. This table clarifies that the values of the mean refractive indices ($n_o{}^{\parallel}$ and $n_o{}^{\perp}$) and birefringence (Δn_o) at radiation dose ($\xi = 0.0$) approximately agree with the results obtained for non-irradiated PET and PETG fibers [12].

It is worth noting that the optical parameters of irradiated PETG are higher than those of irradiated PET fiber. This is because the irradiated copolyester chain is less regular than the irradiated monopolyester chain.

The birefringence is a measure of the total molecular orientation of a system. The birefringence of both irradiated PET and PETG fibers is increasing with increasing radiation dose. The rate of increase of the optical orientation function ($\langle f(\theta) \rangle$) of both irradiated PET and PETG fiber as a function of radiation dose (ξ) can be controlled by the suggested empirical formulae: $\langle f(\theta) \rangle = 1.2715 \times 10^{-4} \quad \xi + 0.7054$ and $= 5.5137 \times 10^{-4} \ \xi + 0.5647$, respectively. Such behavior indicates that irradiation enhances the anisotropic nature of both monopolyester and copolyester fibers. The density of irradiated PET and PETG fibers was calculated as a function of radiation dose and the results are shown in Table 2. It is clear that the density increased with increasing dosage. The effect of γ-radiation is that the polyester molecule generates free radicals, which react with other molecules to crosslink and increase the density.

From the obtained suggested empirical formula, one can notice that the rate of variation of measured parameters of irradiated PETG fiber is higher than that of irradiated PET fiber. This is due to the irradiated monopolyester (PET) fiber being of higher crystallinity than the copolyester (PETG).

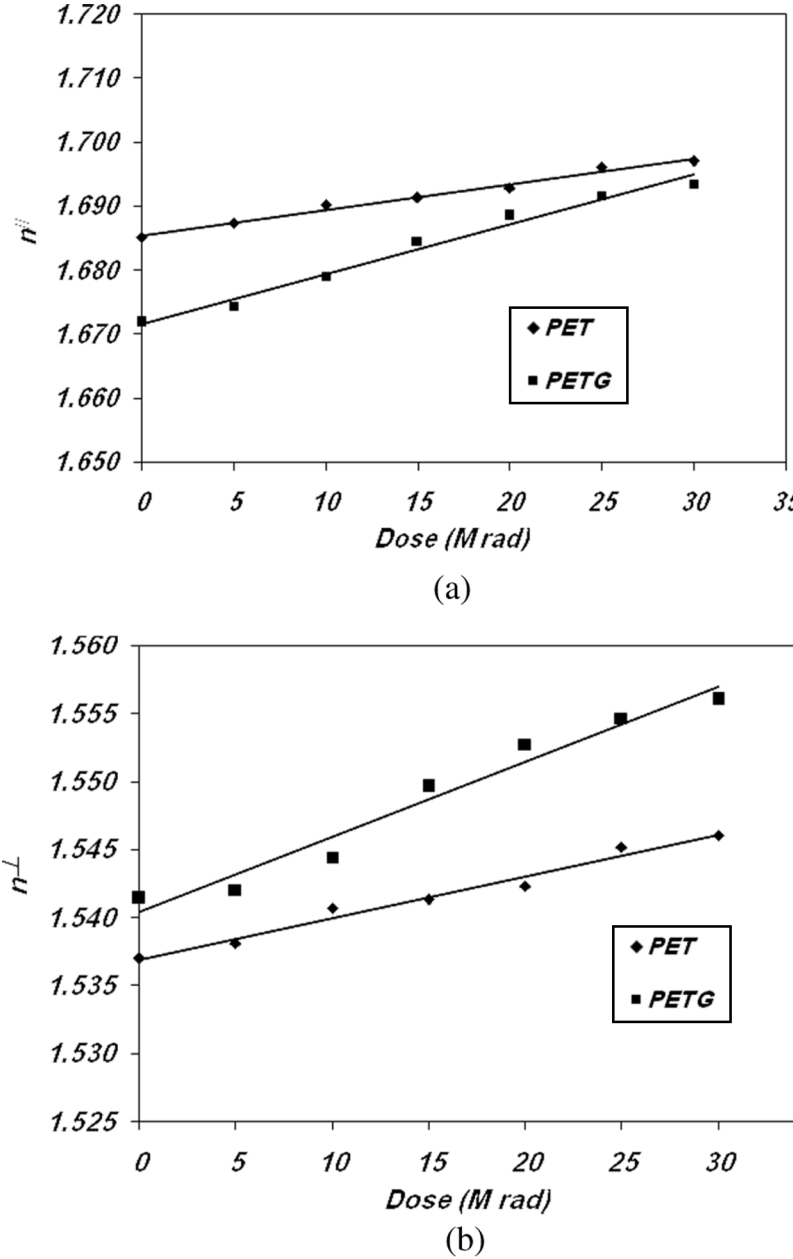


FIGURE 4 The variation of the mean refractive index as function of radiation dose for the plane-polarized light vibrating (a) parallel and (b) perpendicular to both the PET and PETG fibers axes.

TABLE 1 The Constants of the Suggested Formula (2) for γ-Irradiated PET and PETG Fibers

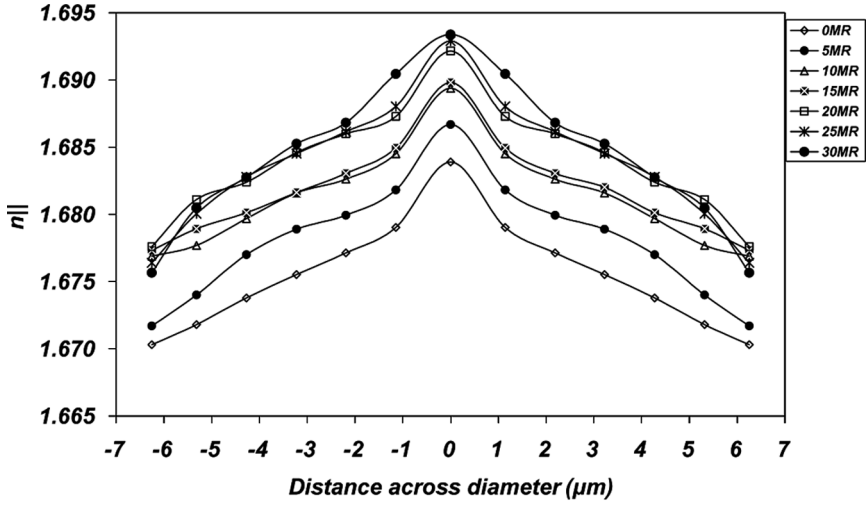
	PET fiber			PETG fiber		
Constant	$n^{\parallel}$	$n^{\perp}$	Δn	$n^{\parallel}$	$n^{\perp}$	Δn
σ	4×10^{-4}	3×10^{-4}	9×10^{-5}	8×10^{-4}	6×10^{-4}	2×10^{-4}
η_o	1.6855	1.5369	0.1485	1.6717	1.5404	0.1313

The Effect of γ-Irradiation on the Refractive Index Profile of the PET and PETG Fibers

The fiber cross-section is assumed to be divided into very small circular zones. Each one can be considered to have a constant refractive index. We can increase the accuracy of the measured refractive index profile by increasing the number of circular zones. The accuracy of this method is also increased by using an immersion liquid whose refractive index is very close to the fiber skin. The refractive index of each layer can be calculated, and consequently the refractive index profiles for $n^{\parallel}$and $n^{\perp}$ are measured experimentally using a computer program based on the zonal approximation method [14]. Figure 5 illustrates the refractive index profile of monopolyester (PET) and copolyester (PETG) fibers at different doses at constant temperature (45°C) when the plane-polarized light vibrating parallel to fiber axis. These profiles confirm the previous results. It is obvious that, upon increasing the radiation dose of the PET and PETG fibers, the refractive index profiles increase. The variation of the refractive index profiles of both irradiated PET and PETG fibers has the same behavior but the values of the refractive index profile of irradiated PET fiber is larger than that of irradiated PETG fiber.

TABLE 2 The Variation of the Density of γ-Irradiated PET and PETG Fiber with Radiation Dose

	Density g/cm^3	
Dose (M rad)	PET	PETG
0	1.3590	1.3561
5	1.3617	1.3585
10	1.3667	1.3644
15	1.3743	1.3682
20	1.3807	1.3704
25	1.3850	1.3760
30	1.3880	1.3777



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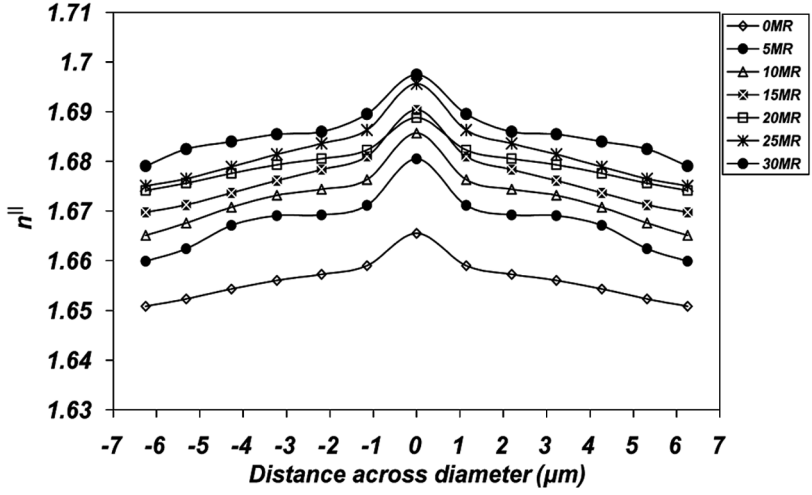

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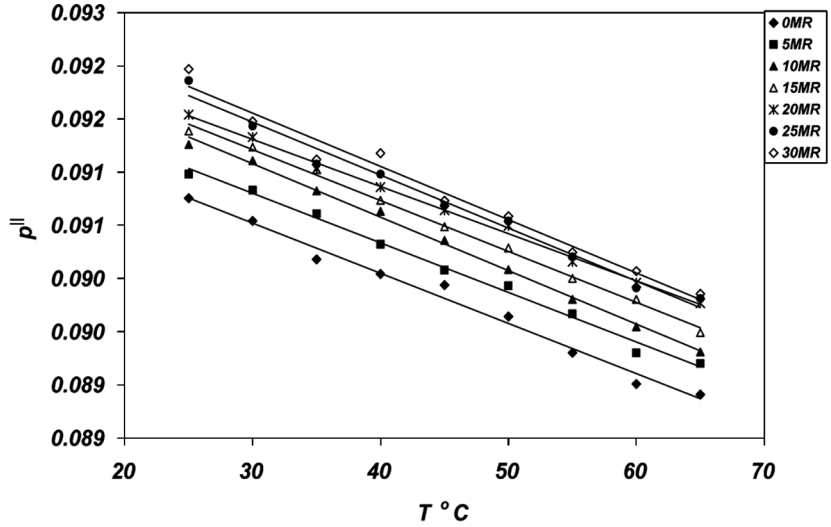
FIGURE 5 The changes of refractive index profile of (a) PET and (b) PETG fibers at different radiation doses when the plane polarized light vibrating parallel to the fiber axis.



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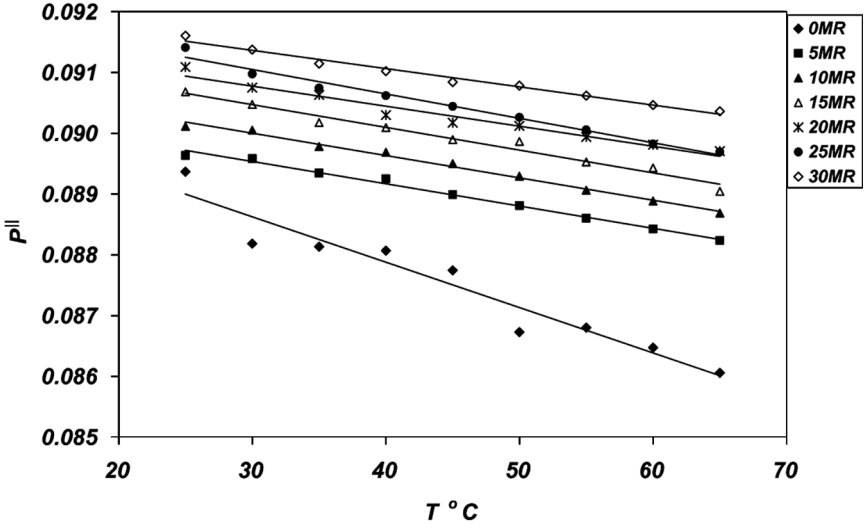


(b)

FIGURE 6 The relation between the molecular polarizability ($p^{\parallel}$) of irradiated (a) PET and (b) PETG fibers with different doses as a function of temperature.

Opto-Thermal Properties of Irradiated PET and PETG Fiber

The Pluta polarizing interference microscope is attached with an opto-thermal device for changing the temperature. The influence of the temperature on the optical and the structural properties of the irradiated PET and PETG fibers has been studied. It was found that, upon increasing the temperature, the optical and the structure parameters of irradiated PET and PETG fibers linearly decreased for the given radiation dose. The value of polarizability per unit volume parallel ($p^{\parallel}$) and perpendicular ($p^{\perp}$) to the fiber axis were calculated using the Lorentz-Lorenz equation [15]:

$$P^{i} = \left(\frac{3}{4\pi}\right)\left(\frac{n_i^2 - 1}{n_i^2 + 2}\right) \tag{3}$$

Figure 6 displays the effect of temperature on the value of polarizability per unit volume ($p^{\parallel}$) of irradiated PET and PETG fibers for a given radiation dose. The slope values of the variation of optical and structure parameters of irradiated PET and PETG fibers with the temperature at different radiation doses are experimentally determined and the results are shown in Tables 3 and 4, respectively. It is clear that the decreasing rate of optical and structure parameters of irradiated PET fiber is different from those of irradiated PETG. Also, the decreasing rate of these parameters of PET and PETG is different for each radiation dose.

The variation of the mean refractive indices of irradiated PET and PETG fibers as a function of radiation dose at different temperatures (25, 30, 35, 40, 45, 50, 55, 60 and 65°C) was studied. It was observed that the mean refractive indices and birefringence of irradiated PET and PETG fibers nonlinearly increase with increasing the radiation dose for the given temperature. The experimental results up to $\xi = 30$ Mrad approximately agree with the following empirical formula:

$$Y = a\xi^2 + b\xi + c, \tag{4}$$

where, Y denotes $n^{\parallel}$, $n^{\perp}$ and Δn and a, b, and c are constants.

These constants of the above suggested empirical formula, which relates the optical parameters of irradiated PET and PETG fibers to the applied radiation dose at temperature 45°C, were determined as shown in Table 5.

It is worth concluding that the optical and structural parameters of irradiated PET and PETG fibers are higher in comparison than those of non-irradiated PET and PETG fibers when the influence of the temperature is studied.

TABLE 3 Thermal Coefficient of Optical and Structural Parameters of γ-Irradiated PET at Different Radiation Doses

Radiation dose (M rad)	$dn^{\parallel}/dT \times 10^{-4}$	$dn^{\perp}/dT \times 10^{-4}$	$d\Delta n/dT \times 10^{-4}$	$dP^{\parallel}/dT \times 10^{-5}$	$dP^{\perp}/dT \times 10^{-5}$	$d\langle f(\theta)\rangle/dT \times 10^{-4}$
0	−4.543	−3.258	−1.284	−4.716	−3.789	−3.178
5	−4.502	−3.228	−1.408	−4.663	−3.750	−3.705
10	−4.860	−3.453	−1.408	−5.026	−3.847	−3.142
15	−4.634	−3.258	−1.376	−4.785	−3.464	−3.486
20	−4.302	−3.298	−1.091	−4.437	−3.305	−3.308
25	−4.477	−3.308	−1.169	−4.984	−3.511	−2.905
30	−4.416	−3.440	−0.966	−5.006	−3.661	−2.721

TABLE 4 Thermal Coefficient of Optical and Structural Parameters of γ-Irradiated PETG at Different Radiation Doses

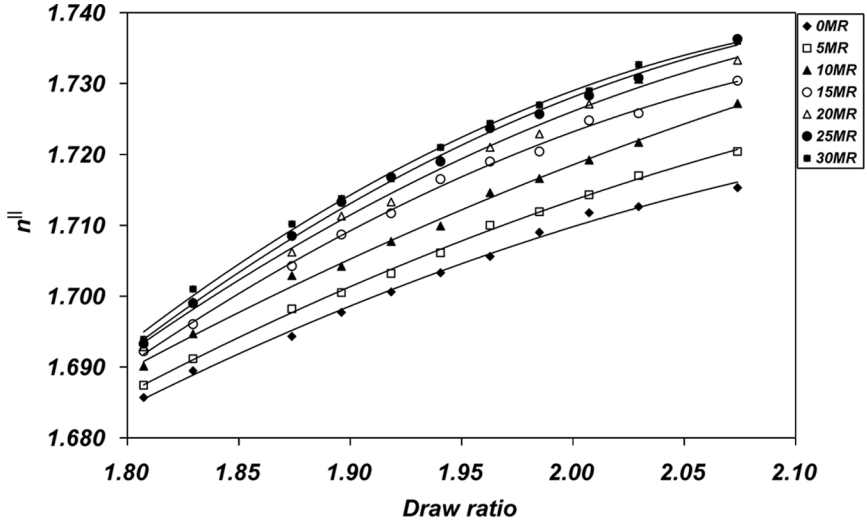
Radiation dose (M rad)	$dn^{\parallel}/dT \times 10^{-4}$	$dn^{\perp}/dT \times 10^{-4}$	$d\Delta n/dT \times 10^{-5}$	$dP^{\parallel}/dT \times 10^{-5}$	$dP^{\perp}/dT \times 10^{-5}$	$df(\theta)/dT \times 10^{-4}$
0	−7.070	−4.017	−31.38	−7.464	−4.665	−3.018
5	−3.510	−3.394	−10.66	−3.667	−3.933	−3.288
10	−3.533	−3.570	−9.212	−3.678	−4.130	−3.161
15	−3.607	−3.480	−1.266	−3.741	−4.010	−2.848
20	−3.198	−3.078	−1.198	−3.305	−3.537	−3.278
25	−3.894	−3.394	−2.833	−4.021	−3.895	−3.447
30	−2.917	−2.688	−2.291	−3.002	−3.069	−3.853

TABLE 5 The Constants of the Suggested Empirical Formula Which Relate the Optical Parameters of Irradiated PET and PETG Fibers to the Applied Radiation Dose (at Temperature 45°C)

	PET			PETG		
Constant	$n^{\parallel}$	$n^{\perp}$	Δn	$n^{\parallel}$	$n^{\perp}$	Δn
a	-7×10^{-6}	-3×10^{-6}	-2×10^{-6}	-3×10^{-5}	-4×10^{-6}	-2×10^{-5}
b	0.0005	0.0005	0.0002	0.0017	0.0008	0.0008
c	1.677	1.5292	0.1475	1.6582	1.5307	0.1261

Measurement of Optical and Structural Parameters of Stretched γ-Irradiated PET and PETG Fibers

The Pluta polarizing interference microscope attached to an opto-mechanical device was used to determine the optical and structural



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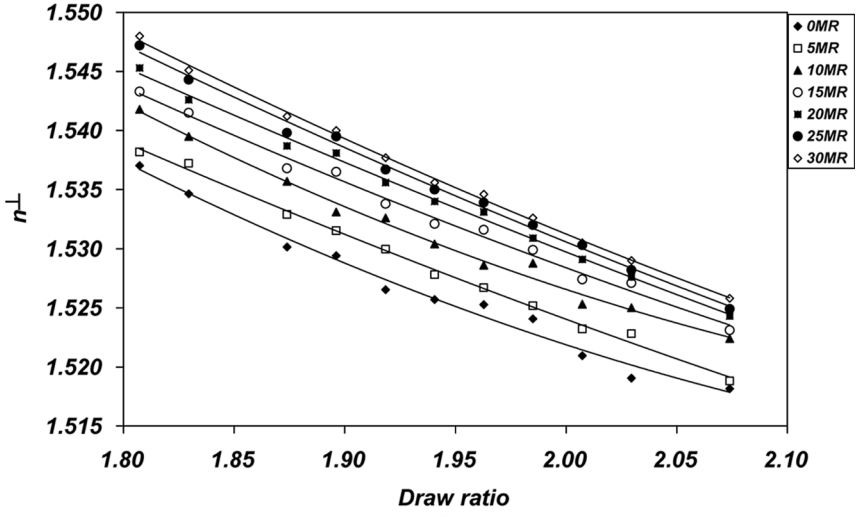


(b)

FIGURE 7 The variation of the mean refractive index for plane-polarized vibrating (a) parallel and (b) perpendicular to the axis of irradiated PET fibers with different doses as a function of draw ratio.

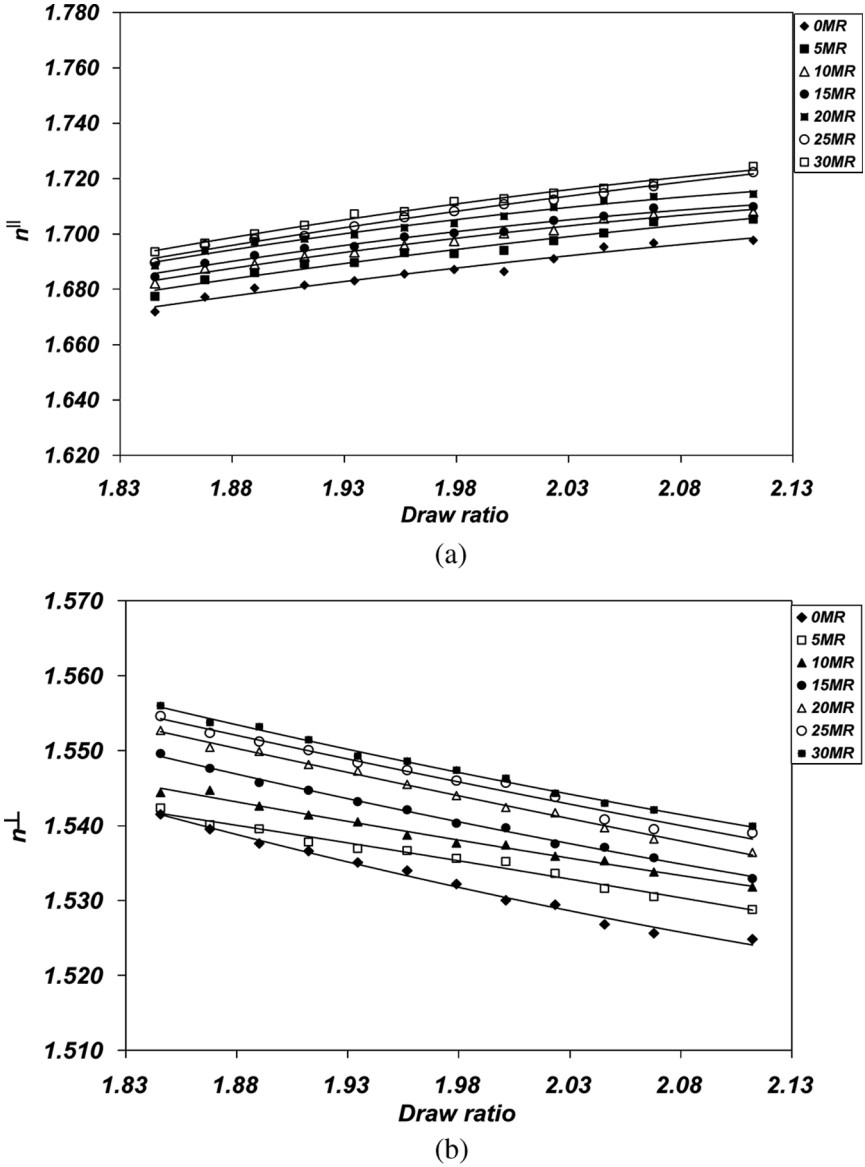


FIGURE 8 The variation of the mean refractive index for plane-polarized vibrating (a) parallel and (b) perpendicular to the axis of irradiated PETG fibers with different doses as a function of draw ratio.

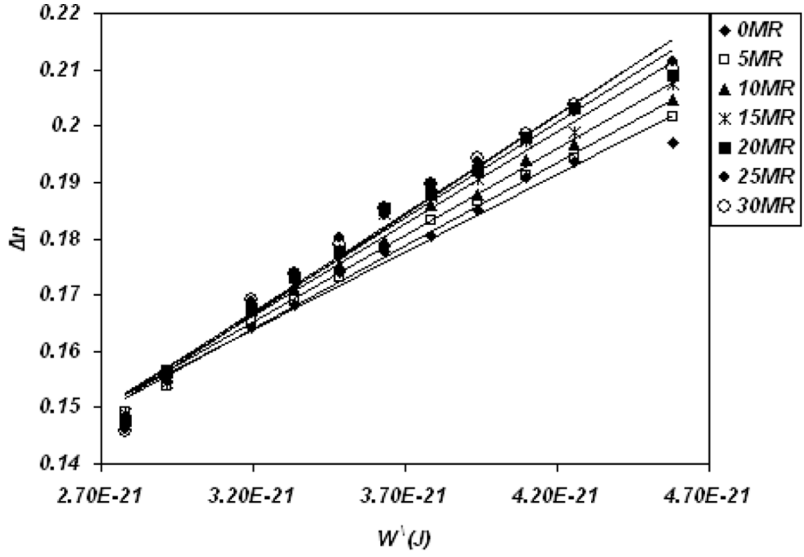
parameters of stretched irradiated PET and PETG at different draw ratios. Having determined the fringe displacement as a function of draw ratio for a given radiation dose, the mean refractive indices ($n^{\parallel}$ and $n^{\perp}$) and the birefringence ($\Delta n = n^{\parallel} - n^{\perp}$) were calculated using Eq. (1). Figures 7 and 8 show the variation of the refractive indices ($n^{\parallel}$ and $n^{\perp}$) of the γ-irradiated PET and PETG fibers at different radiation doses as a function of draw ratio at room temperature. Monochromatic light of wavelength 546 nm was used.

These figures indicate that when the irradiated PET and PETG fibers are stretched and the draw ratio increases, the refractive index increases for plane-polarized light vibrating parallel to the fiber axis ($n^{\parallel}$) of the irradiated fibers. In contrast, the refractive index for plane-polarized light vibrating perpendicular to the fiber axis ($n^{\perp}$) decreases. The rate of increasing $n^{\parallel}$ and decreasing $n^{\perp}$ of irradiated PET fiber is different from that of irradiated PETG fiber. As expected, the values of the mean refractive indices and the birefringence ($\Delta n = n^{\parallel} - n^{\perp}$) of irradiated PET and PETG fibers are higher in comparison to those of non-irradiated PET and PETG fibers when the stretching process is applied. This is because the irradiation enhances the anisotropic nature of the given polymer.

The change of the mean refractive indices ($n^{\parallel}$ and $n^{\perp}$) and the birefringence (Δn) of irradiated PET and PETG fibers was studied as a function of radiation dose at room temperature and different draw ratios. It was found that the mean refractive indices ($n^{\parallel}$ and $n^{\perp}$) and the birefringence (Δn) increase with increasing doses according to the empirical formula (4). The constants of this suggested empirical formula, which relate the optical parameters of irradiated PET and PETG fibers to the applied radiation dose at draw ratios 1.9406 and 1.9790, respectively, were determined, as shown in Table 6.

TABLE 6 The Constants of the Suggested Empirical Formula Which Relate the Optical Parameters of Irradiated PET and PETG Fibers to the Applied Radiation Dose at Draw ratios 1.9406 and 1.9790, Respectively

	PET			PETG		
Constant	$n^{\parallel}$	$n^{\perp}$	Δn	$n^{\parallel}$	$n^{\perp}$	Δn
a	-2×10^{-6}	-7×10^{-6}	-8×10^{-6}	-5×10^{-6}	-4×10^{-6}	-1×10^{-6}
b	0.0011	0.0006	0.0005	0.0009	0.0006	0.0003
c	1.7022	1.5255	0.1767	1.6877	1.5332	0.1548



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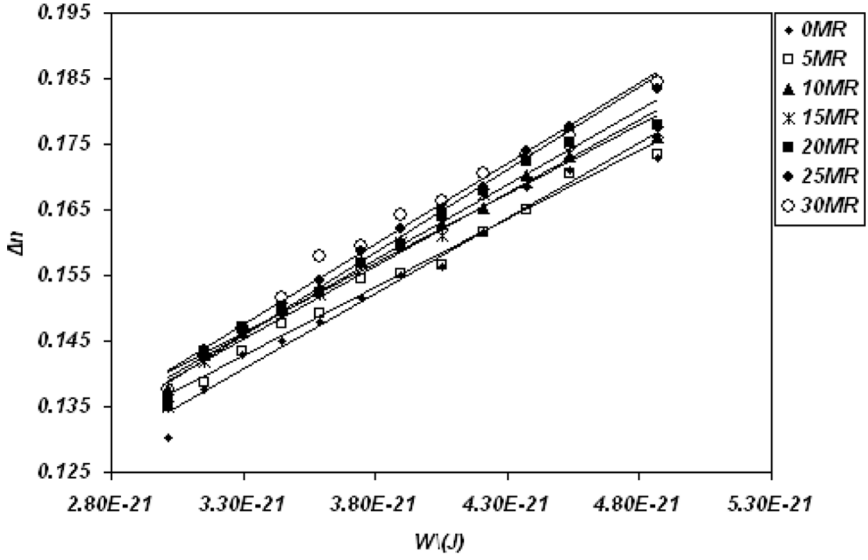

(b)

FIGURE 9 The relation between the average work-per-chain $\dot{W}$ and the birefringence Δn for irradiated PET (a) and PETG (b) fibers at room temperature.

The average work $\grave{W}$ for a collection of chains depends on the distribution of chain-end distances. It can be determined using the following equation [16]:

$$W = \frac{3K_BT}{2}\left[\frac{1}{3}(D^2 - D^{-1}) + (D^{-1} - 1)\right], \quad (5)$$

where, K_B is the Boltzmann's constant, D is the drawn ratio of the fiber and T is the absolute temperature.

It was found that the average work-per-chain ($\grave{W}$) of irradiated PET and PETG fibers increases with the radiation dose increase, as shown in Figure 9.

CONCLUSIONS

The most important results of this study can be summarized as:

- When γ-radiation interacts with the polymeric textile fiber, its energy is absorbed by this fiber and active species such as free radicals are produced. This results in increasing the optical and structural parameters. Therefore, the values of optical and structural parameters of γ-irradiated PET and PETG fibers are higher in comparison to those of non-irradiated PET and PETG fibers when the stretching and thermal processes are applied.
- γ-irradiation improves the anisotropic nature of the polymeric textile fiber.
- At room temperature the optical and structural parameters of the irradiated PET and PETG linearly increase with increasing the γ-radiation dose up to 30 Mrad.
- The values of optical and structural parameters of irradiated copolyester (PETG) fiber are less than those of irradiated monopolyester (PET) fiber. This confirmed that the irradiated monopolyester (PET) fiber is more highly crystalline than the irradiated copolyester (PETG).
- The variation rates of optical and structural parameters of irradiated PET fiber are different from those of irradiated PETG fiber. Also, the variation rates of these parameters are different for each radiation dose.
- Empirical formulae are suggested for correlating the optical properties of fibers with the applied dose.
- These formulae could be used as bases for constructing a simple method for γ-irradiation dosimetry at room temperature (Eq. (2)) and above the room temperature up to 65°C (Eq. (3)).
- Finally, radiation processing is a practical and economical method for modifying the physical and chemical properties of polymeric materials.

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