Optothermal Properties of Fibers. VII. Optical Investigation of Annealed Nylon-6 Fibers

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ABSTRACT: The influence of annealing on the birefringence of nylon-6 fibers was examined. These fibers were found to show considerable optical anisotropy. Three independent techniques were used to study the optical anisotropy in these fibers. The first technique to determine the diameter of the fiber from the diffraction pattern was using a He—Ne laser beam. The second technique was the application of the Becke-line technique to study the effect of annealing on the skin layer of these fibers at different temperatures for a constant time of 5 h. The third is the application of a double-refracting interference microscope (the Pluta polarizing interference microscope). The double-beam technique is considered as the most suitable technique for determining the mean refractive indices and the double refraction of the annealed samples at different temperatures. (155 and 175°C and at two constant times 7 and 9 h). Microinterferograms and curves are given for illustration. © 1997 John Wiley & Sons, Inc. J Appl Polym Sci **65:** 1293–1306, 1997

INTRODUCTION

Studying the optical properties of fibers, such as refractive indices and birefringence, played an important role in describing the intermolecular arrangement within the fibers. The molecular mechanism responsible for the variation in the optical properties is clarified by simultaneous observation of the birefringence.[1,2] Birefringence depends on molecular orientation in polymer fibers as it contains contributions to the polarizabilities of all molecular units in the sample.[3]

Several studies have been reported on the effect of annealing on the structure of synthetic man-made and natural fibers.[4–12] Annealing was performed at a temperature below the melting point of the material and also may be performed with the ends of the sample free or fixed. In the former case, the sample shrinks, whereas in the latter, it retains its length but exerts measurable retractive forces on its fixed ends. Both effects increase with increasing annealing temperature.

In the present work, samples of nylon-6 fibers having different annealing conditions and allowed to cool down in the oven to room temperature (27 ± 1°C) were measured by two independent techniques: The first technique is the application of the Becke-line technique to study the effect of annealing on these fibers.[13] The second method deals with the application of two-beam interferometric technique.

THEORETICAL CONSIDERATION

A Pluta[14] polarizing interference microscope was used to determine the mean refractive indices and birefringence:

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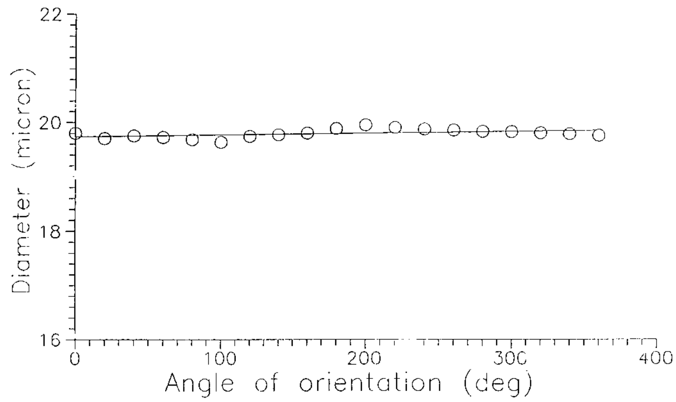


Figure 1 Fiber radius at different angular orientations for nylon-6 fibers.

$$n_a^{\parallel} = n_L + \frac{F^{\parallel}}{h}\frac{\lambda}{A} \quad (1)$$

$$n_a^{\perp} = n_L + \frac{F^{\perp}}{h}\frac{\lambda}{A} \quad (2)$$

$$\Delta n = \left(\frac{F^{\parallel} - F^{\perp}}{h}\right)\frac{\lambda}{A} = (F \cdot \lambda)/h \cdot A \quad (3)$$

where $n_a^{\parallel}$ and $n_a^{\perp}$ are the mean refractive indices of fiber for light vibrating parallel and perpendicular to the fiber axis, respectively, n_L, the refractive index of the immersion liquid; $F^{\parallel}$ and $F^{\perp}$, the area enclosed under the fringe shifts for parallel and perpendicular direction, respectively; h, the interference fringe spacing corresponding to the wavelength λ; and A, the mean cross-sectional area of the fiber. Also, Δn_a is the birefringence of the fiber and $F^{\parallel} - F^{\perp}$ is the area enclosed under the fringe shift when using the nonduplicated image. Hamza[15] used other equations to determine the mean refractive indices and the birefringence:

$$\therefore n_a^{\parallel} = n_L + \frac{dz^{\parallel}}{h}\frac{\lambda}{d} \quad (4)$$

$$n_a^{\perp} = n_L + \frac{dz^{\perp}}{h}\frac{\lambda}{d} \quad (5)$$

$$\Delta n_a = \left(\frac{dz^{\parallel} - dz^{\perp}}{h}\right)\frac{\lambda}{d} \quad (6)$$

where $dz^{\parallel}$ and $dz^{\perp}$ are the interference fringe shifts inside the fiber for light virbrating paralled and perpendicular to the fiber axis, respectively, and d is the diameter of the fiber. $dz^{\parallel} - dz^{\perp}$ is the interference fringe shift inside the fiber when using the nonduplicated image.

We calculated the isotropic refractive indices values for unannealed and annealed nylon-6 fibers by using the following equation[16]:

$$n_{\text{iso}} = \tfrac{1}{3}[n_a^{\parallel} + 2n_a^{\perp}] \quad (7)$$

The mean polarizabilities per unit volume $P^{\parallel}$ and $P^{\perp}$ for light vibrating parallel and perpendicular to the fiber axis were calculated from the

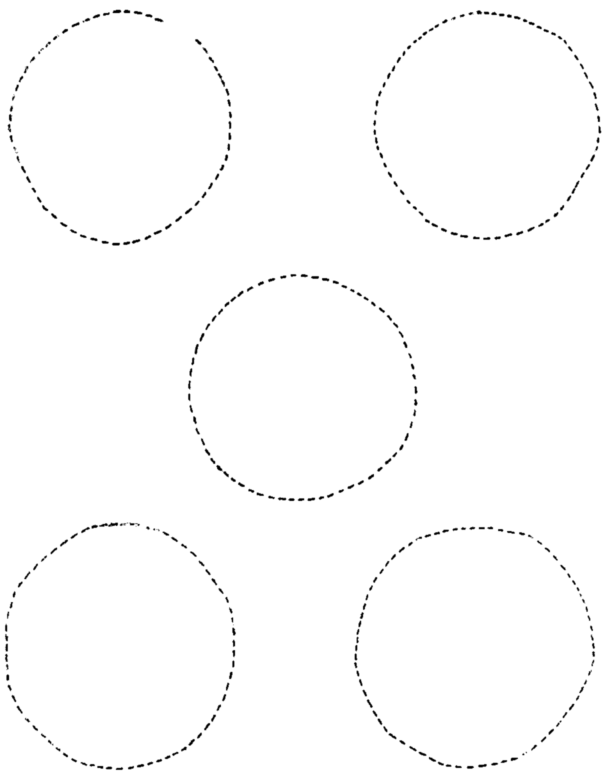


Figure 2 Cross-sectional view of nylon-6 fibers.

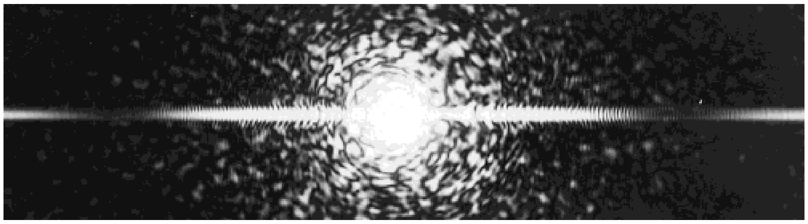


Plate 1 A Fraunhofer diffraction pattern of an unannealed nylon-6 fiber.

measured values of n_a'' and $n_a^{\perp}$ of the fiber using the Lorentz–Lorenz equation[17]:

$$\frac{(n''^2 - 1)}{(n''^2 + 2)} = \left(\frac{4}{3}\right)\pi \cdot P'' \qquad (8)$$

$$\frac{(n^{\perp 2} - 1)}{(n^{\perp 2} + 2)} = \left(\frac{4}{3}\right)\pi \cdot P^{\perp} \qquad (9)$$

EXPERIMENTAL PROCEDURES AND DISCUSSION

Annealing of Samples

Long bundles of nylon-6 fibers were loosely folded in a cocoon form with both ends free. Several samples, placed in special small glass bottles, were heated in an oven (Model WT, binder-type E53, Germany) at constant temperature. The temperatures ranged from 90 to 175°C with an error of ±2°C for different annealing times, ranging from 1 to 10 h. The samples were then left to cool in the oven to room temperature (27°C) with an error of ±1°C. Hence, for each annealing temperature, 10 samples were annealed at different durations.

Table I Skin Refractive Indices (R.I.) n_s'' and $n_s^{\perp}$ of Annealed Nylon-6 Fibers at Different Temperatures for Constant Time (5 h) by the Becke-line Method at 27°C, When Using Light of Wavelength 546. 1 nm

Temperature (°C)	R.I.		
	n_s''	$n_s^{\perp}$	$\Delta n_s \times 10^{+2}$
27	1.5769	1.5283	4.86
90	1.5769	1.5283	4.86
110	1.5791	1.5287	5.04
125	1.5820	1.5305	5.15
140	1.5850	1.5312	5.38
155	1.5879	1.5323	5.56
165	1.5921	1.5338	5.83
175	1.5948	1.5360	5.88

Values with accuracy ±0.0008.

Determination of the Diameter and Cross Section of Nylon-6 Fibers

The diffraction technique was used for determining the cross section of unannealed fibers. Ten samples of the fiber were chosen at random to be tested. Direct collimatted parallel light from a He—Ne laser source (λ = 632.8 nm) was directed at a normal incidence to the fiber axis. A Fraunhofer diffraction pattern was formed on a bright screen. The cross section of the fiber was scanned in 18 orientations, in steps of 20° about its axis. The positions of the minima are shown in Plate 1 for a sample of an unannealed nylon-6 fiber. The fiber diameter was calculated according to the following equation[18]:

$$d = \pm \lambda L / x \qquad (10)$$

where d is the diameter of the fiber; λ, the wavelength used; x, the distance from the center of the pattern to the first minima; and L, the distance between the fiber and the screen on which the pattern is produced.

Figures 1 and 2 illustrate some experimental results for the cross-sectional view of nylon-6 fiber. According to these figures, the cross-sectional view was perfectly circular in shape with a diameter of about 19.8 μm with an error of ±0.15 μm.

Determination of the Refractive Indices n_s'' And $n_s^{\perp}$ and Birefringence Δn_s of the Outer Layer of Nylon-6 Fibers Using Becke-Line Method

Using a monochromatic light of wavelength 546.1 nm, the immersion liquid was prepared by mixing different volumes of α-bromonaphthalene and liquid paraffin. For a mixture of two liquids 1 and 2, the refractive index of the mixture is $n = (n_1 v_1$

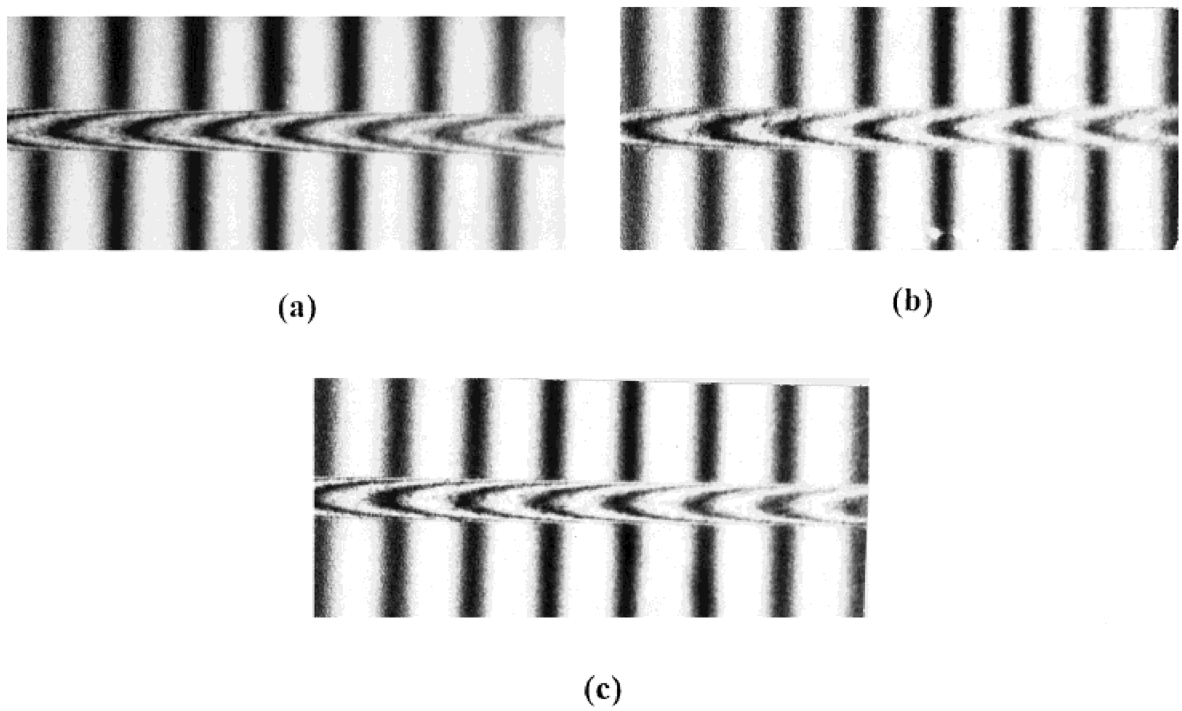


Plate 2 (a–c) Microinterferograms showing nonduplicated images by the two-beam interference microscope of nylon-6 fibers using monochromatic light of wavelength λ = 546.1 nm: (a) unannealed; (b) annealed for 7 h at 140°C; (c) annealed for 7 h at 155°C.

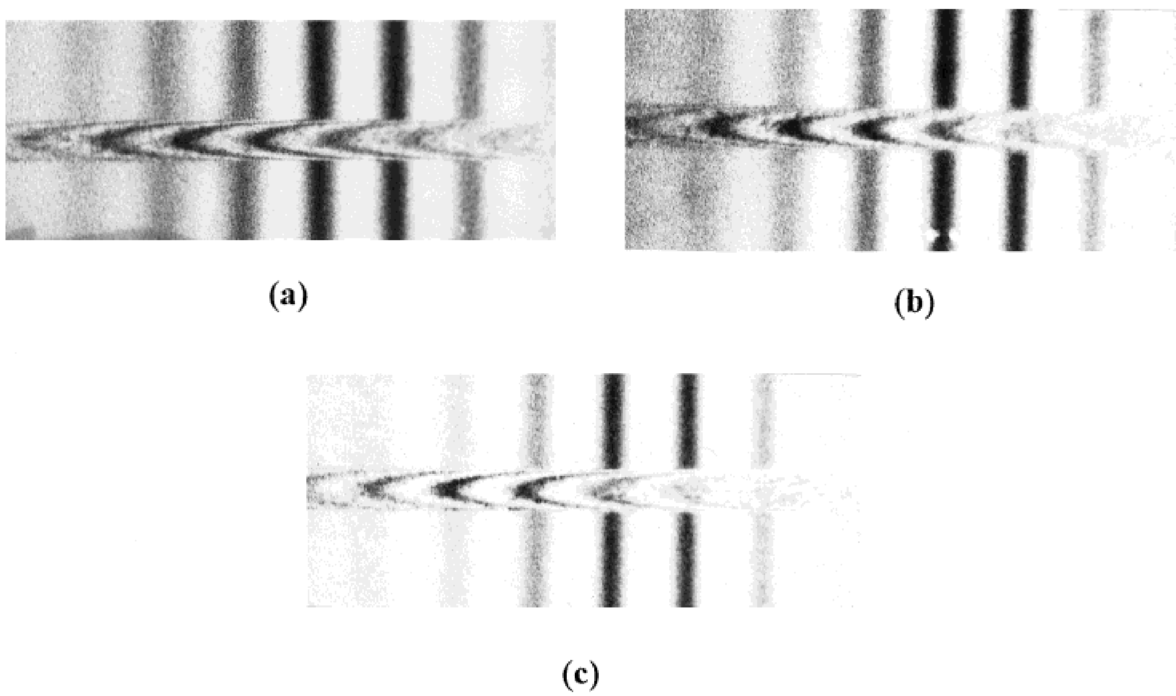


Plate 3 (a–c) Microinterferograms showing nonduplicated images by the two-beam interference microscope of nylon-6 fibers using white light of wavelength λ = 550 nm: (a) unannealed; (b) annealed for 7 h at 140°C; (c) annealed for 7 h at 155°C.

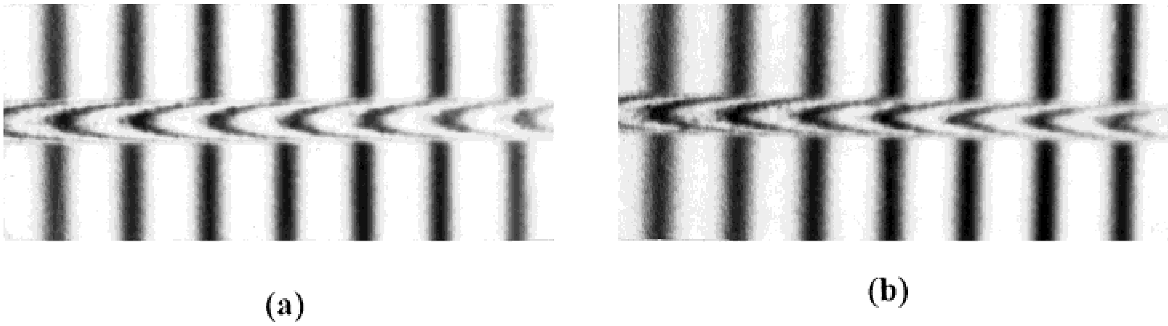


Plate 4 (a–b) Microinterferograms showing nonduplicated images by the two-beam interference microscope of nylon-6 fibers, using monochromatic light of wavelength $\lambda = 546.1$ nm, annealed for 9 h at (a) 110°C and (b) 165°C.

$+ n_2v_2)/(v_1 + v_2)$, where v_1 and v_2 are the volumes of the liquid 1 and 2, respectively. The results are given in Table I. In the same table, the birefringence Δn_s is calculated for a set of annealed nylon-6 fibers having different annealing temperatures at a constant time (5 h). It is well known that the Becke-line method cannot determine the refractive indices at two directions on the same fiber. Two determinations are necessary: one with the light vibrating parallel to the fiber axis and the other with the perpendicular. The optical effects produced by an immersed fiber do not depends solely upon the difference between the refracive indices of the fiber and the liquid but also upon the product of this difference and the fiber thickness. Also, the Becke refractive indices are not confined to the fiber surface but may occur at any point on the fiber radius.[19]

Interferometric Determination of the Birefringence and Refractive Indices of Nylon-6 Fibers

The totally duplicated image of the fiber obtained with the Pluta polarizing interference microscope was used to calculate the mean refractive indices $n_a^{\parallel}$ and $n_a^{\perp}$ of unannealed and annealed nylon-6 fibers. Throughout this experiment, the refractive index of the immersion liquid was $n_L = 1.5493$ at 27°C.

Plate 2(a) is a microinterferogram showing a nonduplicated image by the two-beam interfer-

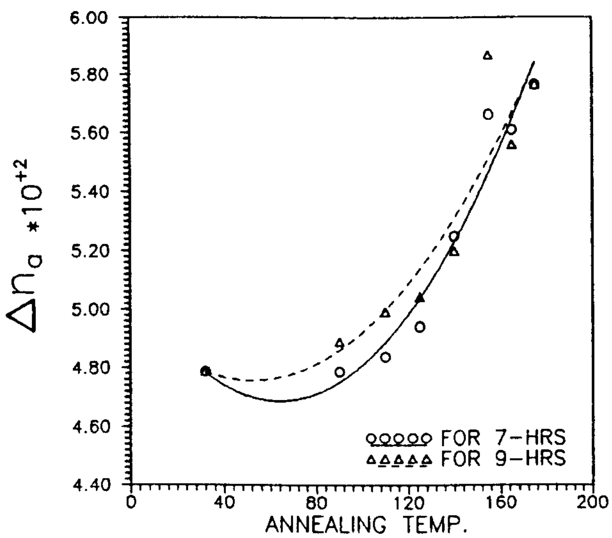


Figure 3 The variation of the mean birefringence Δn_a of nylon-6 fibers versus the annealing temperatures for 7 and 9 h duration, measured by Pluta micrscopy.

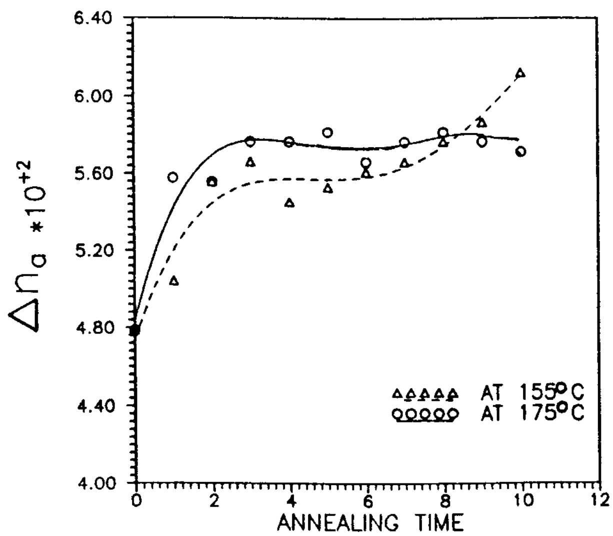
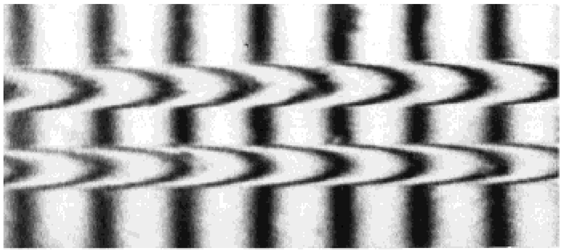
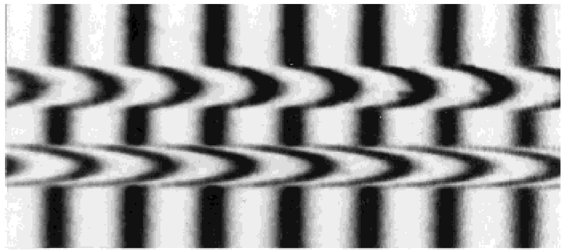


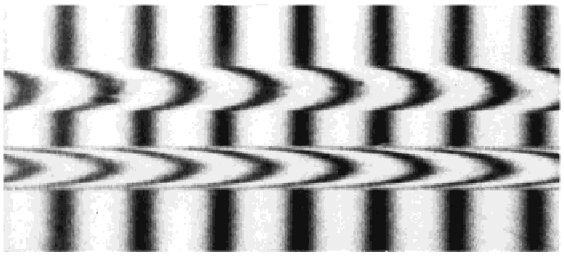
Figure 4 The relation between the mean birefringence of nylon-6 fibers and the annealing time for 155 and 175°C temperatures, measured by Pluta microscopy.



(a)



(b)



(c)

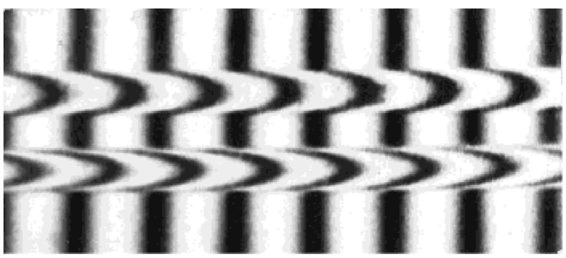
Plate 5 (a–c) Microinterferograms showing totally duplicated images using monochromatic light of wavelength $\lambda = 546.1$ nm, by the two-beam interference microscope of nylon-6 fibers: (a) unannealed; (b) annealed for 7 h at 140°C; (c) annealed for 7 h at 165°C.

ence microscope of a normal nylon-6 fiber. Plates 2(b) and 2(c) are microinterferograms showing nonduplicated images by the two-beam interference microscope of nylon-6 fibers annealed for 7 h at different annealing temperatures: 140 and 155°C, respectively. A monochromatic light of wavelength $\lambda = 546.1$ nm was used.

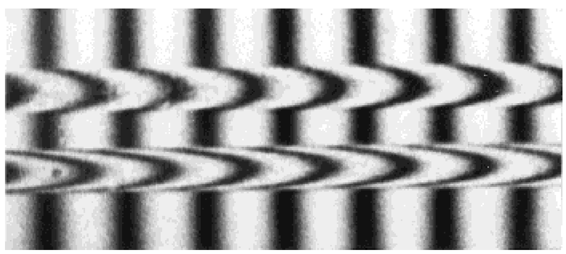
For comparison, another nonduplicated image by the two-beam interference microscope of a normal fiber appears in Plate 3(a). Plates 3(b) and 3(c) are microinterferograms showing nonduplicated images of nylon-6 fibers annealed for 7 h at different annealing temperatures 140 and 155°C, respectively. White light ($\lambda = 550$ nm) was used for this series.

Microinterferograms of nonduplicated images of the fibers annealed for 9 h at different annealing temperatures, 110 and 165°C, appear in Plates 4(a) and 4(b). Monochromatic light ($\lambda = 546.1$ nm) was used.

The mean distance of the fringe shift ($dz'' - dz^{\perp}$) and the interfering spacing h were deter-



(a)



(b)

Plate 6 (a, b) Micorinterferograms showing totally duplicated images using monochromatic light of wavelength $\lambda = 546.1$ nm, by the two-beam interference microscope, of nylon-6 fibers annealed at 155°C for (a) 4 h and (b) 10 h.

Table II Mean Birefringence Values of Nylon-6 Fibers at Various Annealing Temperatures for 7 h Corresponding to the Wavelengths 546.1 nm and 550 nm Using One Birefringent Prism and That Corresponding to the Wavelength 546.1 nm Using Two Birefringent Prisms, Measured by Pluta Microscopy at 27°C

	Applying Two Birefringent Prisms					Applying One Birefringent Prism			
	$\lambda = 546.1$ nm					$\lambda = 546.1$ nm		$\lambda = 550$ nm	
Annealing Temp (°C)	dz'' (mm)	$dz^{\perp}$ (mm)	n''_a	$n^{\perp}_a$	Δn_a	$dz'' - dz^{\perp}$ (mm)	Δn_a	$dz'' - dz^{\perp}$ (mm)	Δn_a
27	13	10.25	1.576	1.528	0.048	23.25	0.048	22.75	0.047
90	13	10.25	1.576	1.528	0.048	23.25	0.048	22.75	0.047
110	14	10	1.578	1.529	0.049	23.5	0.048	23	0.048
125	14.5	10	1.579	1.529	0.050	24	0.049	23.75	0.049
140	18.25	9	1.587	1.531	0.056	25.5	0.052	25	0.052
155	19.5	8	1.589	1.533	0.057	27.5	0.057	27.25	0.056
165	20.25	7	1.591	1.535	0.056	27.25	0.056	27	0.056
175	22.5	5.5	1.596	1.538	0.058	28	0.058	27.75	0.058

$n_L = 1.5493$, $h = 13.4$ mm, and magnification is 375. The error in n'' and $n^{\perp}$ is 2×10^{-3}.

mined from the microinterferograms for all the samples. The mean diameter d of the nylon-6 fibers was determined from the diffraction pattern of a single fiber using a He—Ne laser beam.

The mean birefringence Δn_a was determined directly using eq. (6) and the results are exhibited in Figures 3 and 4. Figure 3 shows the relationship between Δn_a and the various annealing temperatures for the constant annealing time of 7 and 9 h, using monochromatic light ($\lambda = 546.1$ nm). The change of Δn_a corresponding to the different intervals of annealing time at the constant annealing temperatures 155 and 175°C is shown in Figure 4.

The Variable Double-Refraction Interference Microscope for Determining the Mean Refractive Indices of Nylon-6 Fibers

A Pluta microscope was used in a different manner for determining the mean refractive indices n''_a, $n^{\perp}_a$ and the mean birefringence Δn_a for the same groups of samples of nylon-6 fibers mentioned above. Plate 5(a) shows a microinterferogram of totally duplicated images of an unannealed nylon-6 fiber in the parallel direction (upper image) and in the perpendicular direction (lower image). Microinterferograms of the totally duplicated images of nylon-6 fibers annealed for

Table III Comparitive Results of the Mean Refractive Indices and Birefringence of Nylon-6 Fibers Annealed for 9 h at Different Temperatures According to Eqs. (4)–(6) and (1)–(3), When Using Light of Wavelength 546.1 nm at 27°C by Pluta Microscopy

	Applying Eqs. (4)–(6)					Applying Eqs. (1)–(3)				
Annealing Temp (°C)	dz'' (mm)	$dz^{\perp}$ (mm)	n''_a	$n^{\perp}_a$	Δn_a	F'' (mm)^2	$F^{\perp}$ (mm)^2	n''_a	$n^{\perp}_a$	Δn_a
27	13	10.25	1.576	1.528	0.048	75	60	1.576	1.528	0.048
90	13.5	10.25	1.577	1.528	0.049	79	59	1.577	1.529	0.049
110	15	10	1.580	1.529	0.051	84	59	1.579	1.529	0.051
125	16.5	9.5	1.583	1.530	0.054	95	56	1.583	1.530	0.053
140	18.5	8.75	1.587	1.531	0.056	108	51	1.587	1.531	0.056
155	20	7.75	1.590	1.533	0.057	116	46	1.590	1.533	0.057
165	20	7.5	1.590	1.534	0.057	118	44	1.591	1.534	0.057
175	23	5	1.597	1.539	0.058	133	30	1.596	1.539	0.058

$n_L = 1.5493$, $h = 13.4$ mm, and magnification is 375. Values with accuracy $\pm 2 \times 10^{-3}$.

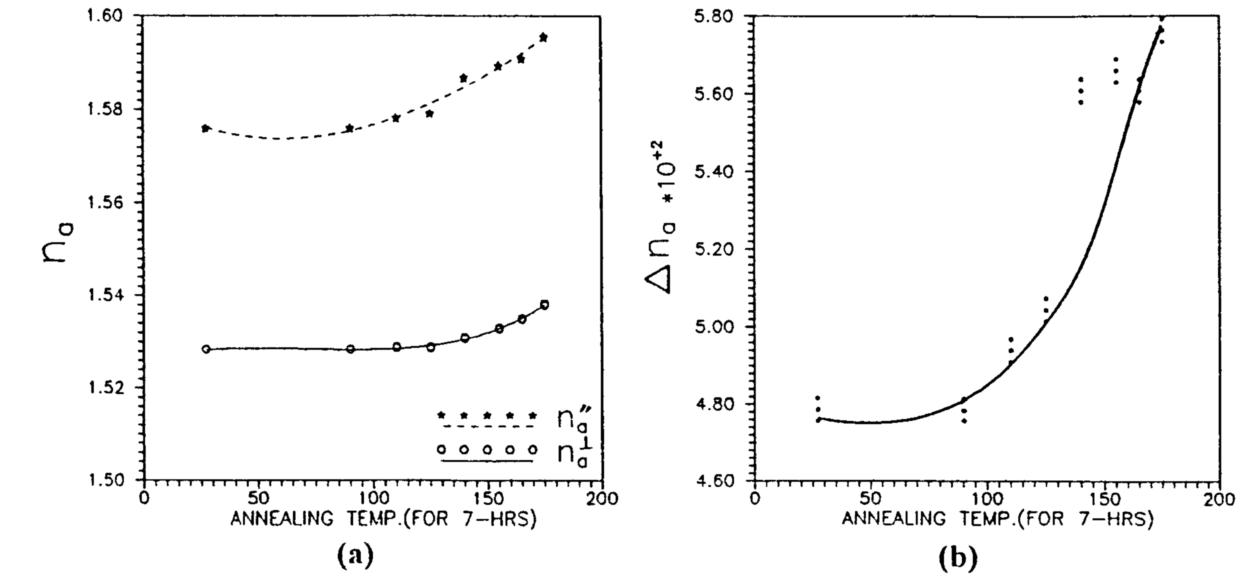


Figure 5 The variation of the mean refractive indices and birefringence of nylon-6 fibers with the annealing temperatures for 7 h, measured by Pluta microscopy: (a) n_a'' and $n_a^{\perp}$; (b) birefringence Δn_a.

7 h at different annealing temperatures, 140 and 165°C, respectively, are shown in Plates 5(b) and 5(c). Microinterferograms of nylon-6 fibers annealed at a constant temperature of 155°C for different intervals of time, 4 and 10 h, respectively, are shown in Plates 6(a) and 6(b).

In the present work, the refractive index of the immersion liquid was $n_L = 1.5493$ at 27°C and monochromatic light of wavelength $\lambda = 546.1$ nm were used. The mean distance of the fringe shifts for parallel and perpendicular directions, dz'' and $dz^{\perp}$, the mean area enclosed under the fringe shifts for parallel and perpendicular directions F'' and $F^{\perp}$, and the interfering spacing h were determined from the microinterferograms. The fiber thickness d was determined from the Fraunhofer

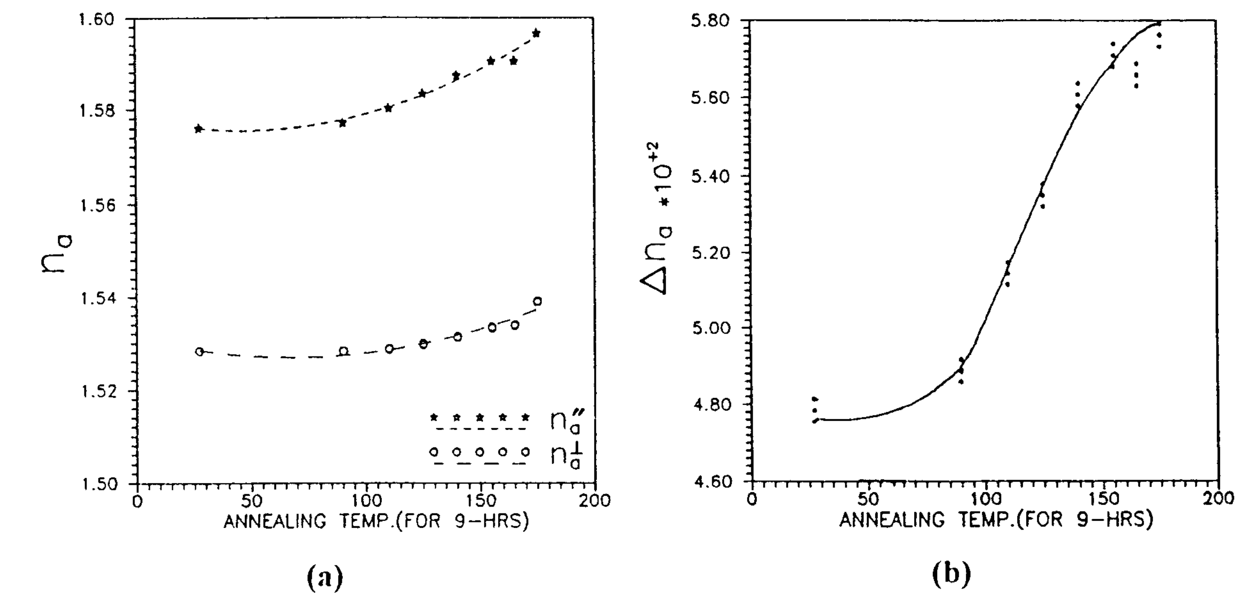
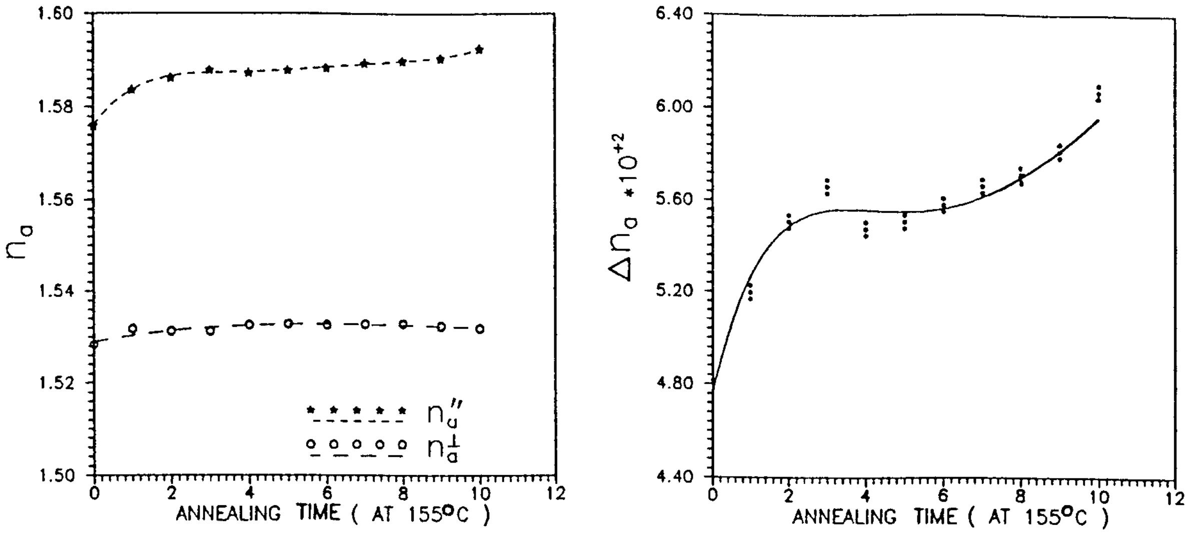


Figure 6 The change in the mean refractive indices and birefringence of nylon-6 fibers versus the annealing temperatures for 9 h measured by Pluta microscopy: (a) n_a'' and $n_a^{\perp}$; (b) birefringence Δn_a.

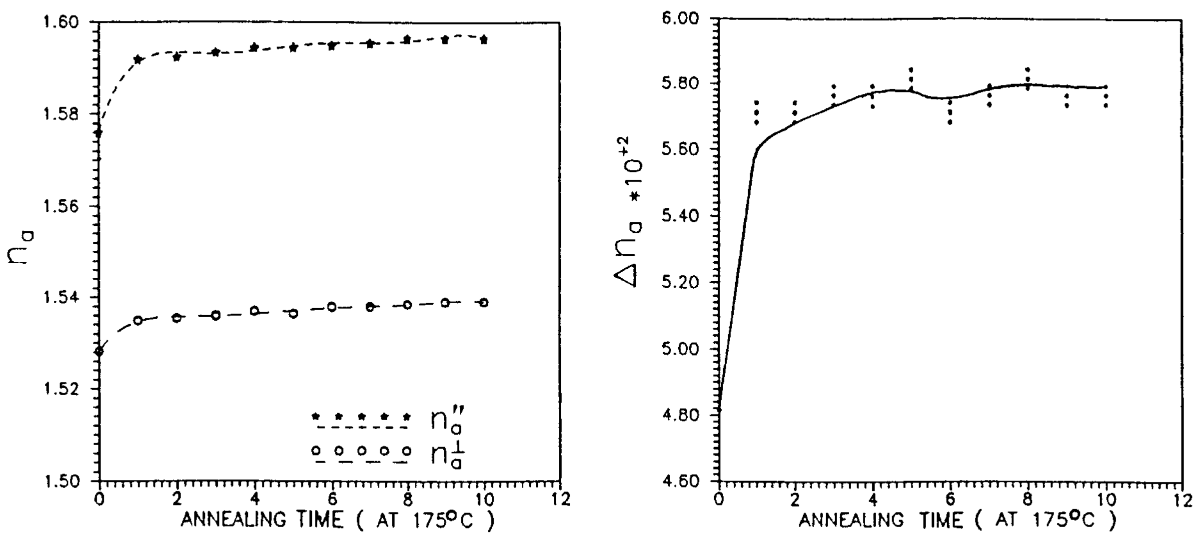


(a) (b)

Figure 7 The variation of the mean refractive indices and birefringence of nylon-6 fibers with the annealing time at 155°C, measured by Pluta microscopy: (a) n_a'' and $n_a^{\perp}$; (b) birefringence Δn_a.

diffraction pattern of a single fiber. The mean refractive indices n_a'' and $n_a^{\perp}$ were calculated using eqs. (4) and (5), whereas the mean birefringence was determined using eq. (6).

The mean birefringence values of nylon-6 fibers at various annealing temperatures for the constant annealing time of 7 h corresponding to the wavelengths 546.1 and 550 nm using one birefringent prism and that corresponding to the wavelength 546.1 nm using two birefringent prisms are given in Table II. From the obtained data, we observe that the results obtained by applying the



(a) (b)

Figure 8 The change in the mean refractive indices and birefringence of nylon-6 fibers versus the annealing time at 175°C, measured by Pluta microscopy: (a) n_a'' and $n_a^{\perp}$; (b) birefringence Δn_a.

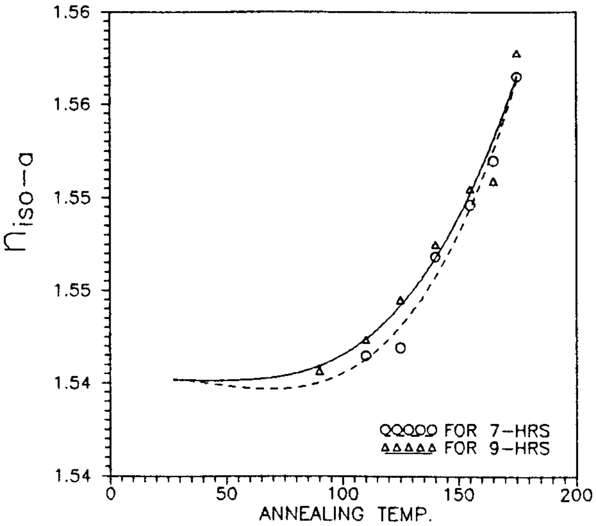


Figure 9 The variation of the mean isotropic refractive indices of nylon-6 fibers versus the annealing temperatures for 7 and 9 h duration by Pluta microscopy.

two different wavelengths did not exhibit significant disagreements.

The mean refractive indices $n_a^{\parallel}$, $n_a^{\perp}$, and Δn_a of nylon-6 fibers annealed for 9 h at different temperatures obtained by eqs. (1)–(3) and that obtained by eqs. (4)–(6) using the Pluta microscope and monochromatic light of wavelength 546.1 nm are given in Table III. By comparison, the results obtained by applying the different equations exhibit a remarkable agreement.

Figures 5 and 6 show the variations of $n_a^{\parallel}$, $n_a^{\perp}$, and Δn_a with the different annealing temperatures at the constant annealing time of 7 and 9 h, respectively. At the constant annealing temperatures of 155 and 175°C, the behavior of $n_a^{\parallel}$, $n_a^{\perp}$, and Δn_a for the different intervals of annealing time are depicted in Figures 7 and 8, respectively.

Determination of the Isotropic Refractive Indices of Nylon-6 Fibers

The obtained values of $n_a^{\parallel}$ and $n_a^{\perp}$ using the variable double-refracting interference microscope were used to determine the mean isotropic refractive indices values $n_{\text{iso-}a}$ of nylon-6 fibers according to eq. (7). Figure 9 shows the variation of $n_{\text{iso-}a}$ with the different annealing temperatures at the constant annealing times of 7 and 9 h, respectively. At the constant annealing temperatures of 155 and 175°C, the behavior of $n_{\text{iso-}a}$ for the different intervals of annealing time appear in Figure 10, respectively.

Determination of the Polarizability per Unit Volume of Nylon-6 Fibers

The experimental values of the refractive indices were utilized to obtain the polarizability per unit volume using Lorentz–Lorenz eqs. (8) and (9). Figures 11–14 give the behavior of $P_a^{\parallel}$ and $P_a^{\perp}$ for different conditions of annealing.

DISCUSSION

From the previous data and the corresponding figures obtained in this work, several remarks can be made: The variation in $n_a^{\parallel}$ and $P_a^{\parallel}$, during the thermal treatment, is greater than in $n_a^{\perp}$ and $P_a^{\perp}$, although the general trend is almost similar. This indicates that the reorientation of nylon-6 fibers is stronger in the parallel direction than in the perpendicular direction, i.e., Δn_a and ΔP_a are positive. This has been suggested in the literature and some experiments with γ-radiation have been reported.[20,21]

At the constant annealing temperature of 155°C, for the first 2 h of annealing, $n_a^{\parallel}$, $n_a^{\perp}$, and Δn_a as well as $P_a^{\parallel}$, $P_a^{\perp}$, ΔP_a, and $n_{\text{iso-}a}$ exhibited the same linear behavior. At the annealing tem-

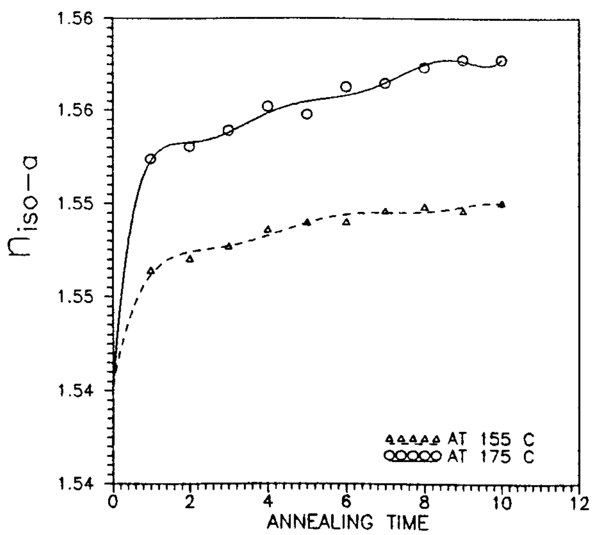


Figure 10 The relation between the mean isotropic refractive indices of nylon-6 fibers and the annealing time for 155 and 175°C temperatures by Pluta microscopy.

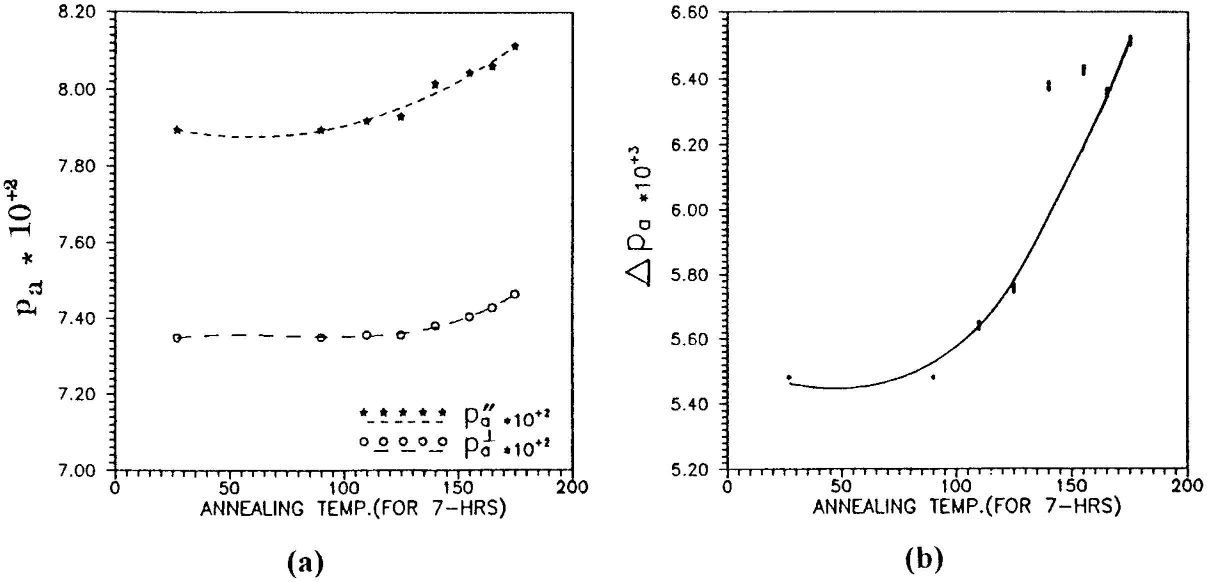


Figure 11 The variation of the mean polarizabilities per unit volume and ΔP_a with the annealing temperatures for 7 h, measured by Pluta microscopy for (a) P_a'' and $P_a^{\perp}$ and (b) ΔP_a.

perature of 175°C, the change in the above parameters was abrupt during the first hour.

Between 2 and 8 h of annealing at the constant temperature of 155°C, as well as in the range of 1–8 h at the annealing temperature of 175°C, the change in the above parameters was considerably small. This probably indicates an adaptation of the macromolecular structure of nylon-6 fibers to the ambient temperature. It was observed that at 155°C, when the fibers were annealed for 10 h, the corresponding values of the birefringence and ΔP were maximum.

At the constant annealing time of durations 7

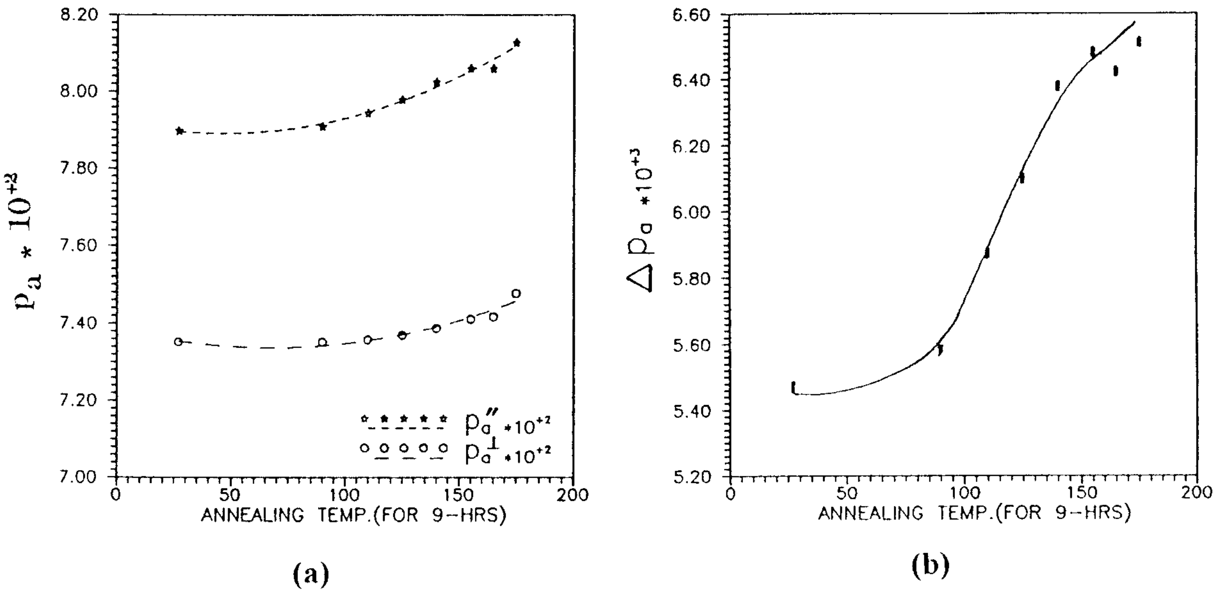
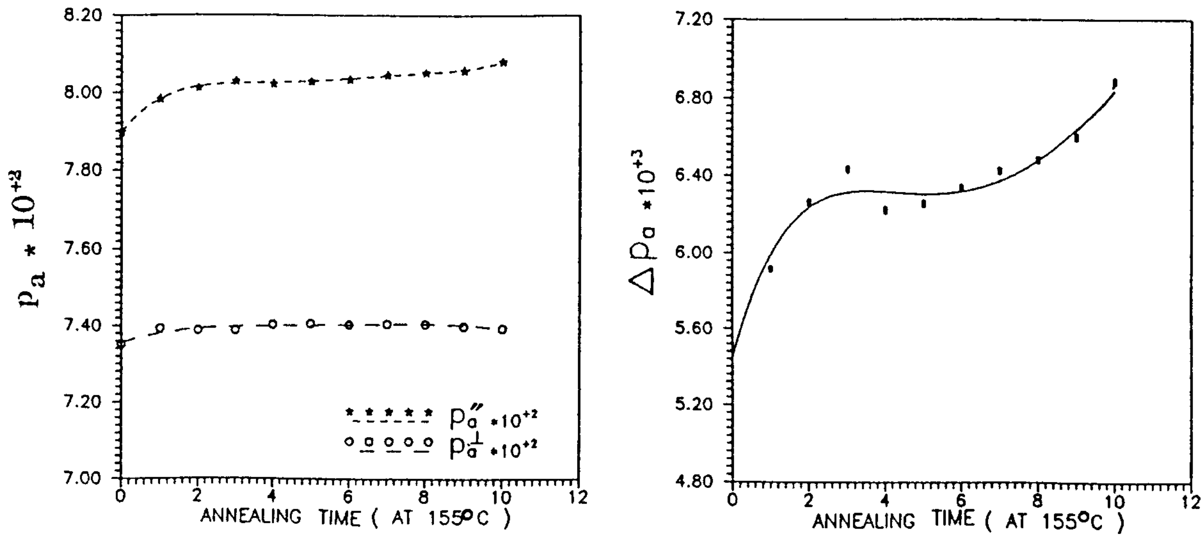


Figure 12 The change in the mean polarizabilities per unit volume and ΔP_a versus the annealing temperatures for 9 h, measured by Pluta microscopy for (a) P_a'' and $P_a^{\perp}$ and (b) ΔP_a.

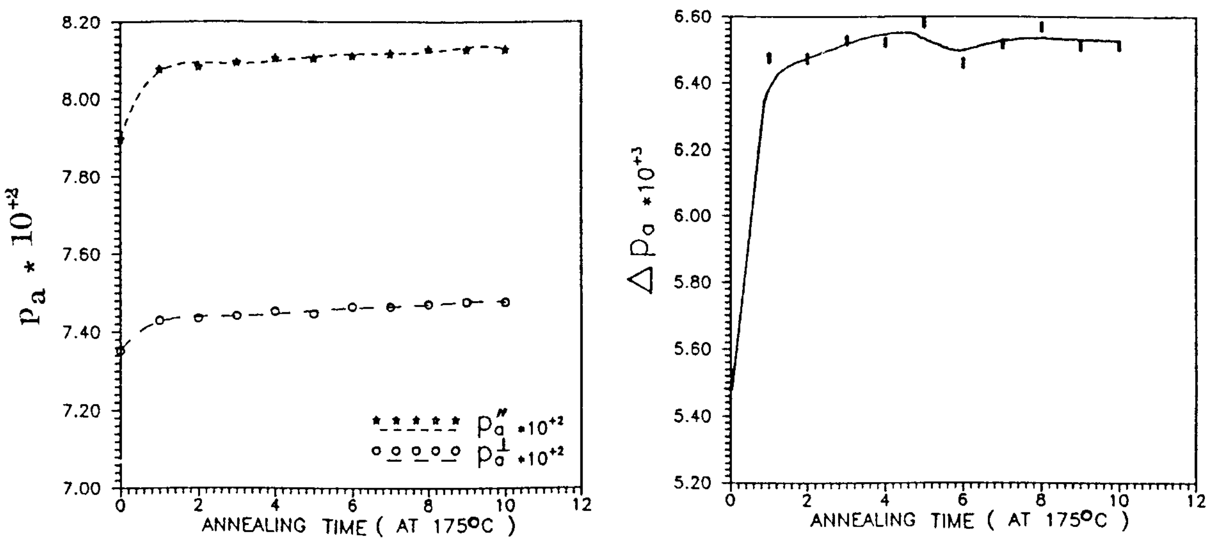


(a) (b)

Figure 13 The variation of the mean polarizabilities per unit volume and ΔP_a with the annealing time from 1 to 10 h at 155°C, measured by Pluta microscopy for (a) P''_a and $P^{\perp}_a$ and (b) ΔP_a.

and 9 h, while increasing the temperature from 90 to 175°C, the behavior of the mean refractive indices, birefringence, polarizabilities per unit volume, and isotropic refractive indices was approximately in agreement with the exponential behavior in its increase.

It was observed visually that the color of the nylon-6 fibers suffer an unnoticeable change when the annealing temperature range was 90–125°C, although the time duration reached 10 h. On the other hand, when annealing took place at high temperatures (155–175°C), a variation in the color accompanied the increase of annealing time duration. The hue changed between light to dark



(a) (b)

Figure 14 The change in the mean polarizabilities per unit volume and ΔP_a versus the annealing time from 1 to 10 h at 175°C, measured by Pluta microscopy for (a) P''_a and $P^{\perp}_a$ and (b) ΔP_a.

ivory in the lower range of temperatures and between light to dark beige at 175°C.

From the present results and the practical importance of the measurements carried out and described in this work which provide acceptable data for the optothermal parameters, the values of the obtained optical parameters are a direct consequence of the isothermal treatment of material. Consequently, the reorientation of polymers may occur not only during fabrication but also during isothermal treatments. The two-beam interference proved to be useful in clarifying the mechanism responsible for the optical behavior of nylon-6 fibers at different annealing temperatures and times.

CONCLUSION

In conclusion, it has been seen that microinterferometric techniques are useful in clarifying the annealing process. From the measurements carried out in the present work to investigate the change in optical properties due to the annealing process for nylon-6 fibers, the following conclusions may be drawn:

1. The annealing study for nylon-6 fiber revealed continuous changes for $n_a^{\prime\prime}$, $n_a^{\perp}$, and Δn_a, which means continuous changes of cyrstalline parameters with increased annealing temperatures and time (see Figs. 3–8).
2. As the nylon-6 fiber is an anisotropic material, the direction dependence of the refractive indices exhibits unequal behavior in different directions. Clearly, this behavior is proof that the structure of the fiber is different along and across its axis. The arrangement of the linear molecules of which it is composed is different in the parallel direction than in the perpendicular direction.
3. The density ρ of the samples was expected to vary due to thermal annealing according to the mobility of the molecules and the mass redistributions of the sample, as $\{[(n_{\text{iso}} - 1)/\rho] = K\}$.
4. The variation of isotropic refractive indices is related to the changes in the specific volume, degree of order of orientation, and mass redistribution of the sample due to different annealing conditions.
5. Under certain conditions, when the fibers were annealed for 10 h at 155°C, maximum values of birefringence (0.061) and $\Delta P (6.89 \times 10^{-3})$ were obtained.
6. The visual color of nylon-6 fibers is changed remarkably due to different annealing conditions. Thus, the changes occurring in nylon-6 are expected.
7. The observed changes in the color of nylon-6 fibers can be related to the thermooxidative degradation process.[10]
8. The annealing process affects other physical properties (swelling, electrical, melting temperature, etc.) of nylon-6. This is expected due to changes in the cohesive forces between adjacent molecules and needs further study.

One can conclude from the above results and considerations that new reorientation appeared due to thermal treatment which promotes a new rearrangement of the structure of the thermally treated polymer.

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REFERENCES

1. R. S. Stein and A. V. Tobolsky, *Text. Res. J.,* **18,** 302 (1948).
2. A. Utso and R. S. Stein, *J. Polym. Sci.,* **3,** 49 (1964).
3. A. A. Hamza, I. M. Fouda, K. A. El-Farahaty, and E. A. Seisa, *Polym. Test.,* **10,** 83 (1991).
4. H. W. Wyckoff, *J. Polym. Sci.,* **62,** 83 (1962).
5. N. S. Murthy, H. Minor, and A. J. Latis, *Macromol. Sci.-Phys. B,* **26**(4), 427 (1987).
6. I. M. Fouda and M. M. El-Tonsy, *J. Mater. Sci.,* **25,** 4752 (1990).
7. I. M. Fouda, M. M. El-Tonsy, and M. A. Shaban, *J. Mater. Sci.,* **26,** 5085 (1991).
8. R. S. Stein, *J. Polym. Sci.,* **7,** 1021 (1969).
9. E. A. Zachariades and S. R. Porter, *The Strength and Stiffness of Polymers,* Marcel Dekker, New York, Basel, 1983, p. 121.
10. A. A. Hamza, M. M. El-Tonsy, I. M. Fouda, and A. M. El-Saied, *J. Appl. Polym. Sci.,* **57,** 265 (1995).
11. I. M. Fouda, M. M. El-Nicklawy, E. M. Naser, and R. M. El-Agamy, *J. Appl. Polym. Sci.,* **60,** 1247 (1996).

12. I. M. Fouda, E. A. Seisa, and K. A. El-Farahaty, *Polym. Test.,* **15,** 3 (1996).
13. A. N. J. Heyn, *Fiber Microscopy,* Interscience, New York, 1954, p. 298.
14. M. Pluta, *Opt. Acta,* **18,** 661 (1971).
15. A. A. Hamza, *J. Microsc.,* **142,** 35 (1986).
16. H. Hannes, *Kolloid Z. Z. Fur Polym.,* **250,** 765 (1972).
17. I. M. Ward, *Structure and Properties of Oriented Polymers,* Applied Science, London, 1975, p. 57.
18. S. M. Curry and A. L. Schawlow, *Am. J. Phys.,* **42,** 12 (1974).
19. J. H. Mclean, *Text. Res. J.,* **35,** 242 (1965).
20. A. A. Hamza, A. M. Chander, and M. A. Mabrouk, *Radiat. Phys. Chem.,* **33,** 231 (1989).
21. N. Barakat, H. A. El-Hennawi, M. M. El-Oker, and F. Sharaf, *J. Phys. D Appl. Phys.,* **2,** 786 (1989).