

Changes in Orientation Caused by UV Irradiation of Nylon 6 Fibers

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ABSTRACT: The effect of UV radiation on the structural parameters that relate to the optical properties of nylon 6 fibers are investigated using interferometry. When the optical parameters are known, we can use them to calculate various orientation functions given by Hermans and de Vries. The properties of concern are the surface reflectivity, transparency, stress optical coefficient, stress due to UV irradiation, optical configuration parameter, segment anisotropy, dielectric constant, and susceptibility. The number of molecules per unit volume, isotropic refractive index, polarizability of a monomer unit, specific refractivity of the di-

electric (along and across the fiber axis) and the form birefringence were obtained. The results are established by relationships designed to relate the calculated structural parameters to the time of exposure. The study demonstrates how the structural parameters change during exposure to UV radiation. Illustrations are given using graphs and microinterferograms. © 2003 Wiley Periodicals, Inc. *J Appl Polym Sci* 88: 3202–3211, 2003

Key words: UV radiation; nylon 6; segment anisotropy; orientation

INTRODUCTION

Light and penetrating radiation are important physical influences on polymers, which are capable of inducing chemical reactions in them. At short wavelengths (230–410 nm), the light quanta may detach fragments from the molecules or break up the macromolecules. Such changes are usually produced by light radiation with wavelengths ranging from 230 to 410 nm. Thus, light can greatly alter the physical and mechanical properties of the polymers. In the storage and service of polymeric articles, the action of light, heat, oxygen, and so forth may cause crosslinking of the macromolecules. This also detracts from the properties of a polymer by inducing brittleness and rigidity, and the ability to crystallize drops sharply.¹

Oriented polymers are produced industrially by extrusion of initially nonoriented fibers or films. The molecular mechanism responsible for the variation in the optical properties is clarified by observation of the birefringence. Birefringence depends on the molecular orientation in polymeric fibers because it contains a contribution from the polarizabilities of all molecular units in the sample.^{2,3} It is a measure of the total molecular orientation of a system and is an excellent property to use for the study of polycrystalline polymers.

Various techniques have been established to estimate the molecular orientation in polymer solids, in-

cluding X-ray diffraction,^{4–6} sonic velocity measurements,⁴ NMR Raman spectroscopy,^{6,7} UV–visible and IR absorption dichroism,⁸ birefringence,⁹ and fluorescence polarization measurements.¹⁰ The use of interferometric methods is of considerable importance, and they have been used in our laboratory in several studies.^{11,12}

Many authors have studied the effect of UV radiation on the physical and chemical properties of polymers in the form of fibers, films, and solutions.^{13–16} They report that the mechanical properties of irradiated fibers deteriorate with increasing exposure time.

The UV radiation from the sun is capable of exciting electrons to higher energy orbitals. Obviously, excitation will occur if the material of concern is capable of absorbing radiant energy and this in turn depends on the nature of the bonds and groups within the polymer. Deterioration in polymers is often caused by the joint effect of radiation from the sun together with oxygen.¹⁷

The present article focuses attention on the effect of the exposure of nylon 6 to UV for different times the density and optical parameters for determining essential industrial parameters are calculated. The stress optical coefficient, dielectric constant, susceptibility, various orientation functions given by Hermans and de Vries, and the surface reflectivity are calculated with other structural parameters.

THEORETICAL

The optical parameters obtained by two-beam interferometry and the density can be used to calculate the

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orientation functions, stress optical coefficient, configuration parameter, segment anisotropy, and other structural parameters via application of suitable well-known equations given elsewhere.³

The orientation function $f(\theta)$ due to Hermans is represented by a series of spherical harmonics (Fourier series) as follows:^{18,19}

$$f(\theta) = \sum_{m=0}^{\infty} (m + \frac{1}{2}) \langle f_m \rangle f_m(\theta) \tag{1}$$

where the parameters $\langle f_m \rangle$ are the average values (amplitudes). The odd components are all zero and the first three even components $f_2(\theta)$, $f_4(\theta)$, and $f_6(\theta)$ are derived from the above equation.

On the other hand, the $f(\theta)$ is developed according to the continuum theory of birefringence of oriented polymers,²⁰ where

$$f(\theta) = \frac{n_1^2 n_2^2}{n_1^2 + n_2^2} \frac{n_{\parallel} + n_{\perp}}{n_1 + n_2} f_{\Delta}(\theta) \tag{2}$$

where n_1 and n_2 are the refractive indices of the fully oriented fiber, using monochromatic light vibrating parallel and perpendicular to the fiber axis as previously determined²⁰ to be 1.627 and 1.555, respectively, for nylon 6 and n_{\parallel} and n_{\perp} are the measured refractive indices in the parallel and perpendicular directions, respectively. Also, Hermans and Platzek²¹ and Kratky,²² used eq. (2) in the following form:

$$f(\theta) = (1 + a)f_{\Delta}(\theta) - af_{\Delta}^2(\theta) \tag{3}$$

where $f_{\Delta}(\theta)$ is given by Ward²³ and a is constant for the polymeric material. The orientation function was determined according to the above equations, and the values obtained are slightly different.

Average optical orientation

The average overall orientation²⁴ (F_{av}) was calculated from birefringence measurements on individual fibers. The F_{av} was calculated from the following equation:

$$F_{av} = \left[\frac{2\Delta n}{(\Delta n_c^o + \Delta n_a^o)} \right] \tag{4}$$

where the denominator is composed of the intrinsic birefringence of crystals and the ideal amorphous birefringence: $\Delta n_c^o = 0.089$,²⁴ $\Delta n_a^o = 0.078$,²⁴ and $\Delta n_{max}^o = 0.072$.²⁵

Crystallinity equation

The degree of crystallinity (χ) was determined by the relation²⁶

$$\chi = \left(\frac{\rho - \rho_a}{\rho_c - \rho_a} \right) \tag{5}$$

where ρ_c and ρ_a are the respective crystalline density and noncrystalline density ($\rho_c = 1.235 \times 10^3$ kg/m³ and $\rho_a = 1.084 \times 10^3$ kg/m³) as given elsewhere.^{27,28}

The experimental density of the fiber was estimated in an average form by a resonance technique discussed previously.²⁹

Evaluation of form birefringence

The total birefringence for a fiber is the sum of three contributions,

$$\Delta n = \chi_c \Delta n_c^o + (1 - \chi_c) \Delta n_a^o + \Delta n_f \tag{6}$$

where Δn_c^o is the birefringence per unit volume of a crystalline material; Δn_a^o is the birefringence per unit volume for an amorphous material, Δn_f is the form birefringence, and χ_c is the volume fraction of crystalline regions.³⁰

Transparency

When light is incident perpendicularly on an optically homogeneous sample of fibers, the well-known Fresnel equation³² reduces to the following expression for the refractivity factor (R_o):

$$R_o = \left(\frac{\bar{n} - 1}{\bar{n} + 2} \right)^2 \tag{7}$$

The internal transmittance (τ_i) can be given from the following equation (transmittance, transparency):³¹

$$\tau_i = 1 - R_o \tag{8}$$

Determination of dielectric constant and dielectric susceptibility

The dielectric constant (D_{\perp}) measured radially is different from (D_{\perp}) measured axially. The fiber has two different dielectric constants (i.e., it is anisotropic with respect to its dielectric):³³

$$D_{\parallel} = \frac{1 - 2(n_{\parallel}^2 - 1/n_{\parallel}^2 + 2)}{1 - (n_{\parallel}^2 - 1/n_{\parallel}^2 + 2)} \tag{9}$$

An analogous equation holds for D_{\perp} . A fiber's dielectric properties vary according to the degree of orientation.

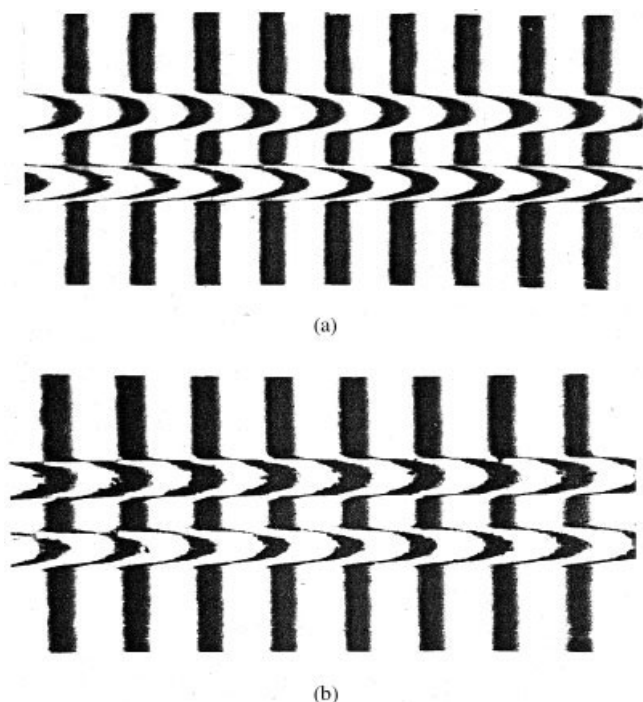


Figure 1 The relationship between the optical orientation functions $f_4(\theta)$ and $f_6(\theta)$ and the exposure time.

The dielectric susceptibility (η) is related to D by the well-known equation³⁴

$$\eta = \frac{D - 1}{4\pi} \quad (10)$$

As the refractive index of a polymer depends on the total polarizability of the molecules, the polarizability of the dielectric (α^{\parallel}) is given by³⁵

$$\frac{n_{\parallel}^2 - 1}{n_{\parallel}^2 + 2} = \frac{N_m \alpha^{\parallel}}{3\psi_o} \quad (11)$$

with an analogous formula for n_{\perp} , where Ψ_o is the permittivity of free space ($8.85 \times 10^{-12} \text{ Fm}^{-1}$). The specific refractivity of the isotropic dielectric (ϵ^{\parallel} , m^3/kg) is proportional to the ρ (kg/m^3) of the medium due to the De Vries equations.²⁰

$$\epsilon^{\parallel} = \frac{(n_{\parallel}^2 - 1)\rho^{-1}}{n_{\parallel}^2 + 2 + S(n_{\parallel}^2 - 1)} \quad (12a)$$

$$\epsilon^{\perp} = \frac{(n_{\perp}^2 - 1)\rho^{-1}}{n_{\perp}^2 + 2 - S(n_{\perp}^2 - 1)} \quad (12b)$$

where S is the anisotropy index, which equals -0.48 for nylon 6.²⁰

Stress optical coefficient

The stress optical coefficient C_s , which equals $\Delta n/\sigma$, is used to determine the segment anisotropy (γ_s) from the following equation:³⁶

$$C_s = \frac{\gamma_s}{90\psi_o KT} \frac{(\bar{n}^2 + 2)^2}{\bar{n}} \quad (13)$$

where γ_s is the segment anisotropy, K is the Boltzmann constant, and T is the absolute temperature.

Optical configuration parameter

The optical configuration parameter³⁷ ($\Delta\alpha$) is related to the C_s by the following equation:

$$\Delta\alpha = \frac{(45KTC_s/2\pi)\bar{n}}{(\bar{n}^2 + 2)^2} \quad (14)$$

where $\Delta\alpha = \alpha_{\parallel} - \alpha_{\perp}$.

The total number (N) of molecules per unit volume is determined from the birefringence Δn and the difference in the polarizabilities of the macromolecules ($P^{\parallel} - P^{\perp}$) as follows:³⁸

$$N = \frac{\bar{n}\Delta n}{2\pi} \left[\frac{(\bar{n}^2 + 2)}{3} \right]^{-2} (P^{\parallel} - P^{\perp})^{-1} \quad (15)$$

where P^{\parallel} and P^{\perp} are the polarizabilities per unit volume, which can be calculated using the equation used in our previous publication.³⁹

TABLE I
Values of Exposure Time, Birefringence, Crystallinity, Three Orientation Functions, Constant a , and Surface Reflectivity

Exposure time (h)	$\Delta n_a \times 10^{-3}$	χ (%)	$f_2(\theta)$	$f_4(\theta)$	$f_6(\theta)$	a	$R_o \times 10^{-2}$
0	51.3	20.80	0.712	0.202	17.31	0.69	4.61
48	53.7	31.45	0.746	0.278	18.51	0.69	4.63
144	54.9	30.46	0.762	0.318	19.14	0.68	4.65
240	56.3	29.80	0.782	0.365	19.89	0.68	4.67

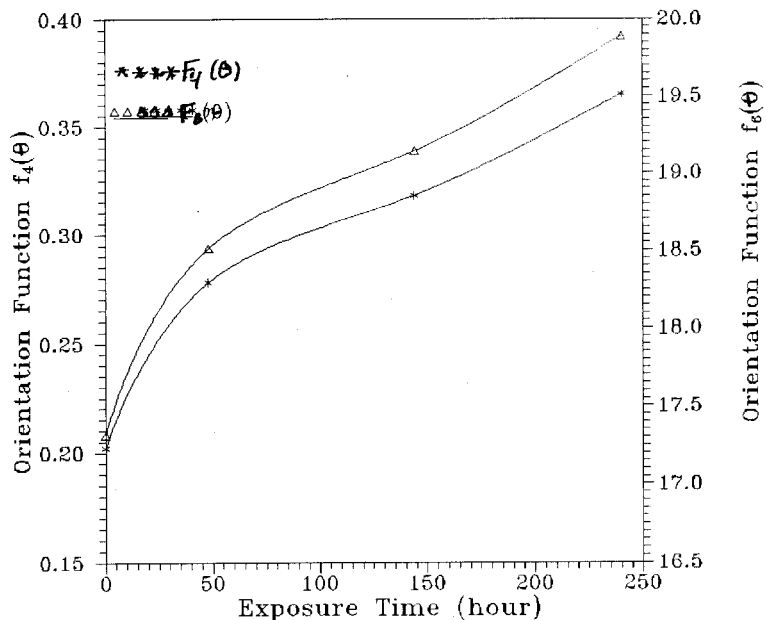


Figure 2 The relationship between the optical orientation functions $f_4(\theta)$ and $f_6(\theta)$ and the birefringence.

Isotropic refractive index

Because most macromolecular crystals are birefringent, an appropriate average isotropic refractive index⁴⁰ (n_{iso}) is determined from the following equation:

$$n_{iso} = (n_{\perp}^2 n_{\parallel})^{1/3} \tag{16}$$

where n_{\perp} and n_{\parallel} were defined before.

EXPERIMENTAL

Sample preparation

The irradiation of the nylon 6 fibers (T1392 nylon, 1400 dtex 2/0; ICI polyamide nylon 6) was carried out using a UV source (OMO, OCL-1, no. 660018; $\lambda \cong 350$ nm) over a period of 48–288 h at room temperature ($30 \pm 2^\circ\text{C}$). The samples were fixed on glass rods and put in front of the source.

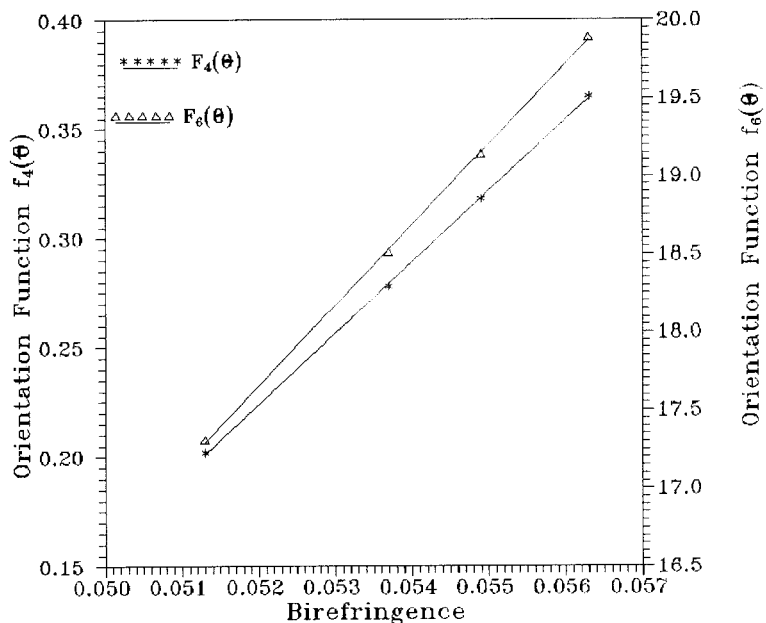


Figure 3 The relationship between the optical orientation functions $f_4(\theta)$ and $f_6(\theta)$ and the birefringence.

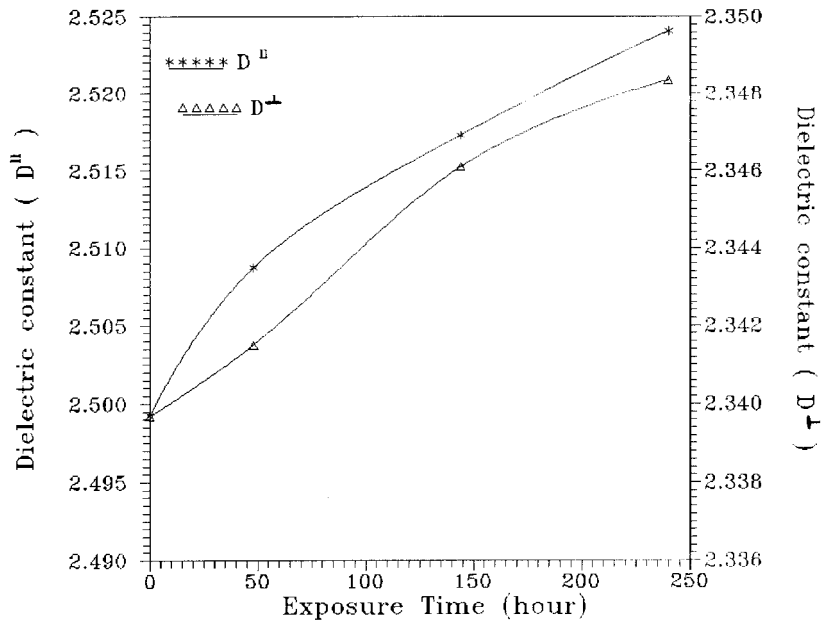


Figure 4 The dielectric constant along and across the fiber axis D^{\parallel} and D^{\perp} and their relationship with the exposure time.

Then the samples were stored in a cool place before use.

The structural changes in the nylon 6 fibers induced by UV radiation and their correlation to the optical properties and density has been investigated using a double-beam polarizing interference microscope.⁴¹

Measurements of transverse sectional area for fibers

The measurements of the transverse sectional area for the nylon 6 fibers showed that the cross section of the

nylon 6 fiber seen by high power optical microscopy is perfectly circular.

RESULTS

Application of two-beam interferometry

Figure 1 is an example of the interferograms of two-beam interferometry from the duplicated image position. Plane polarized light with a 546-nm wavelength vibrating parallel and perpendicular to the fiber axis and a liquid refractive index (n_L) of 1.5502 at 30°C are used. The values of the Δn and ρ are given in Table I.

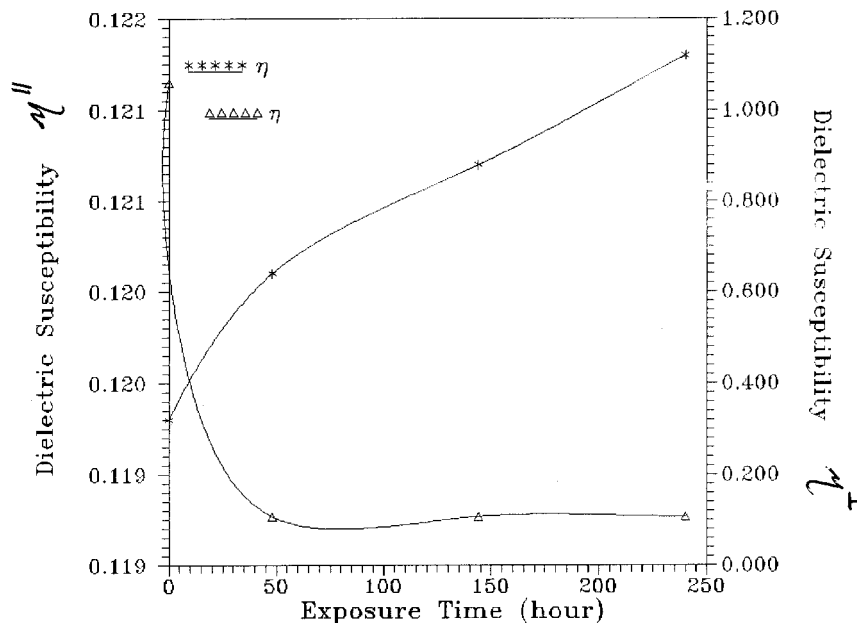


Figure 5 The dielectric susceptibility along and across the fiber axis η^{\parallel} and η^{\perp} and their relationship with the exposure time.

TABLE II
Values of Exposure Time, Dielectric Constant, Optical Configuration Parameter, Specific Refractivity of Dielectric Along and Across Fiber Axis, Segment Anisotropy, Stress Optical Coefficient, Stress, and Isotropic Refractive Index

Exposure time (h)	Δn_f	$\Delta\alpha \times 10^{-34}$ (C/m ²)	$\epsilon^{\parallel} \times 10^{-2}$ (cm ³ /g)	$\epsilon^{\perp} \times 10^{-2}$ (cm ³ /g)	$\gamma_s \times 10^{-44}$ (s ⁴ A ⁴ kg ⁻¹)	$C_s \times 10^{-14}$ (Pa) ⁻¹	$\sigma \times 10^{11}$ (Pa)	n_{iso}
0	-0.398	1.07	34.9	23.69	1.19	4.45	11.53	1.547
48	-0.381	1.12	35.2	23.75	1.24	4.67	11.50	1.548
144	-0.358	1.15	35.4	23.87	1.28	4.80	11.44	1.550
240	-0.349	1.19	35.4	23.91	1.32	4.96	11.35	1.551

Evaluating molecular orientation

The optical orientation functions given by Hermans $f_2(\theta)$, $f_4(\theta)$, and $f_6(\theta)$ were calculated and their values are given in Table I. The optical birefringence gives a direct measure of these parameters on the basis of the aggregate model and the calculated values for these orientation functions are useful in predicting the optical anisotropy present in nylon 6 fibers. Figure 2 shows the relationships between the exposure time and the optical orientation functions $f_4(\theta)$ and $f_6(\theta)$. The $f_4(\theta)$ and $f_6(\theta)$ values were drawn as a function of the birefringence as in Figure 3. The constant a is determined and its values are given in Table I. In addition, the surface reflectivity is also determined and found to be slightly increased with increasing the time of exposure as in Table I.

The dielectric constants (D) in the parallel and perpendicular directions are plotted as a function of the exposure time in Figure 4. The dielectric susceptibilities (η) determined from eq. (10) for parallel and perpendicular cases was drawn as a function of the exposure time as in Figure 5. The calculated values of the

form birefringence (Δn_f) at different times of exposure are given in Table II. The polarizabilities of the dielectric along (α^{\parallel}) and (α^{\perp}) the fiber axis and across were determined and plotted versus the exposure time as in Figure 6, where they increase as the exposure time increases. The difference in the polarizabilities α^{\parallel} and α^{\perp} , which are sometimes called the optical configuration parameter ($\Delta\alpha$), increase with the exposure time as in Table II. The specific refractivity of the isotropic dielectrics (ϵ^{\parallel} and ϵ^{\perp}) were also determined from eq. (12). The values of ϵ^{\parallel} and ϵ^{\perp} are reported in Table II. They increase with increasing time of exposure.

In addition, the γ_s , C_s , σ , N , and n_{iso} are also calculated at different times of exposure. The resulting data for them are given in Table II. The parameters γ_s , C_s , and n_{iso} increase with increasing dose whereas the σ decreases. The γ_s and C_s parameters are plotted against the time of exposure in Figure 7. The N value is constant (1.00). The $\Delta\alpha$ and C_s are plotted against the birefringence in Figure 8. The F_{av} was calculated from eq. (4) and plotted against the time of exposure in Figure 9. The τ_i and R_o are calculated from eqs. (7)

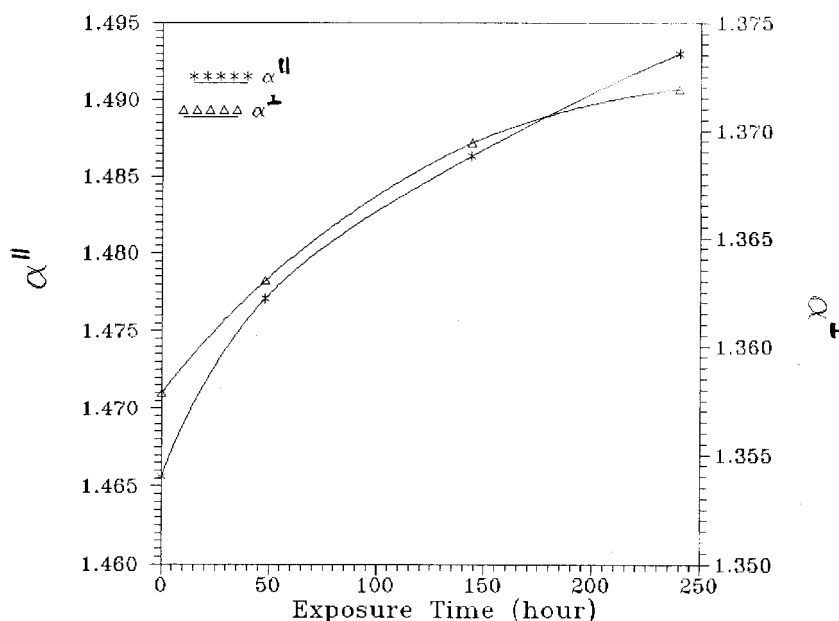


Figure 6 The polarizability of the dielectric along and across the fiber axis α^{\parallel} and α^{\perp} and their relationship with the exposure time.

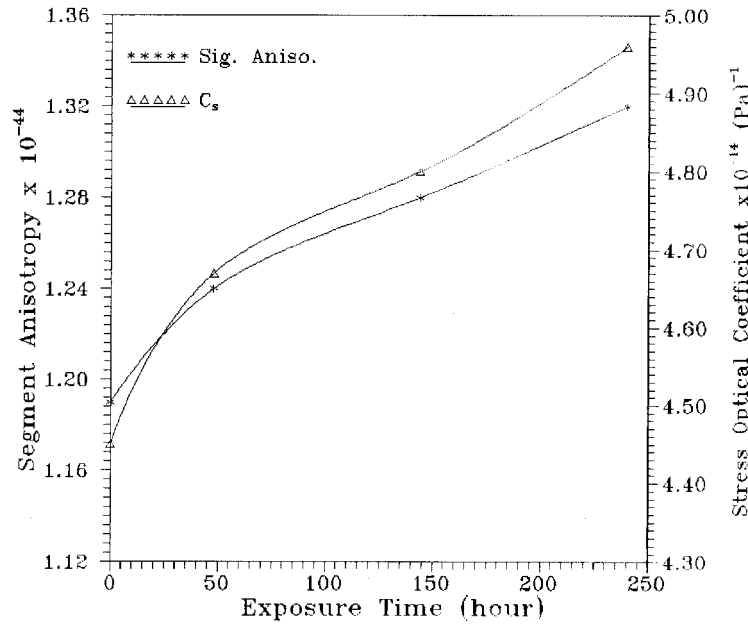


Figure 7 The segment anisotropy γ_s and stress optical coefficient C_s versus the exposure time.

and (8) and plotted against the time of exposure in Figure 10. Figure 11 shows the relationships between the exposure time and crystallinity.

DISCUSSION

Nylon 6 is a suitable polymer for detailed comparative studies of molecular alignment because it is available in a number of distinctly different physical states and it can be readily fabricated into transparent sheets, which are good enough for optical measurements. The

degree of orientation and the related parameters could vary according to the fiber history during manufacturer and subsequent processing operations. In addition, UV irradiation enhances the anisotropic properties of fibers by changing these parameters. The variation in the orientation of the chains caused by increasing the time of irradiation increases the birefringence and therefore the various optical orientation functions increase, as predicted from eqs. (1), (2), and (3). Also, the change in the optical parameters may be due to alterations in the electrical properties arising

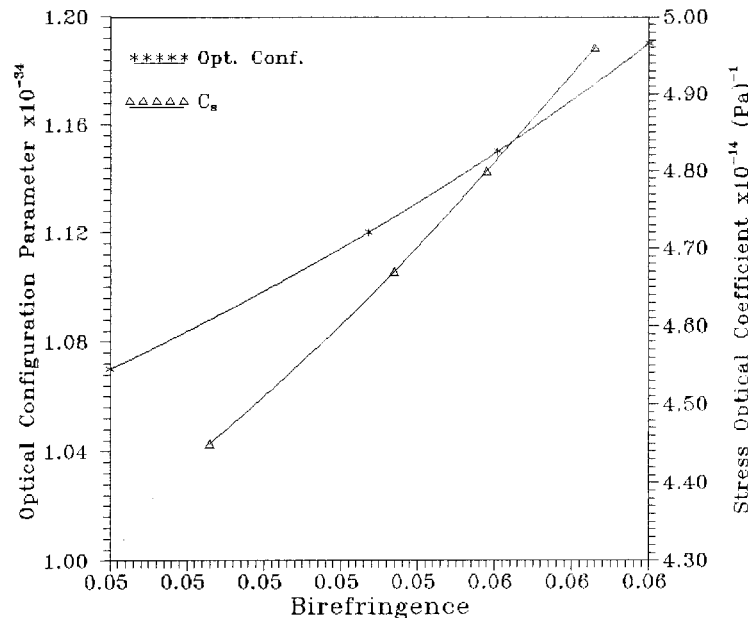


Figure 8 The optical configuration parameter $\Delta\alpha$ and stress optical coefficient C_s and their relationship with the birefringence.

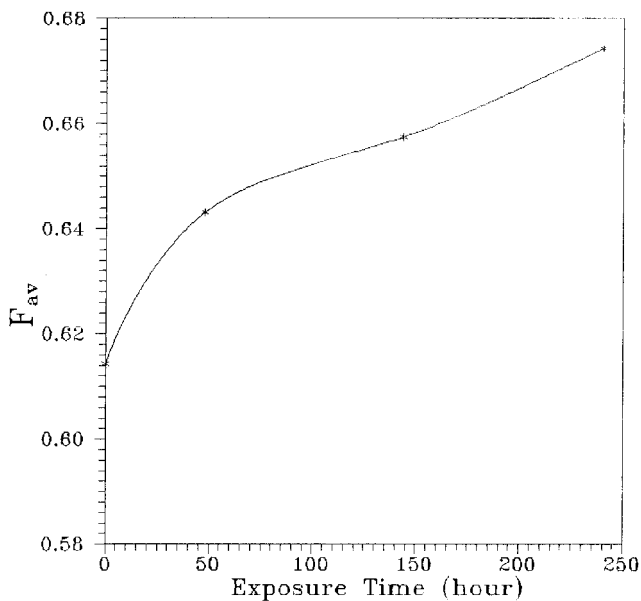


Figure 9 The average orientation F_{av} and the time of exposure.

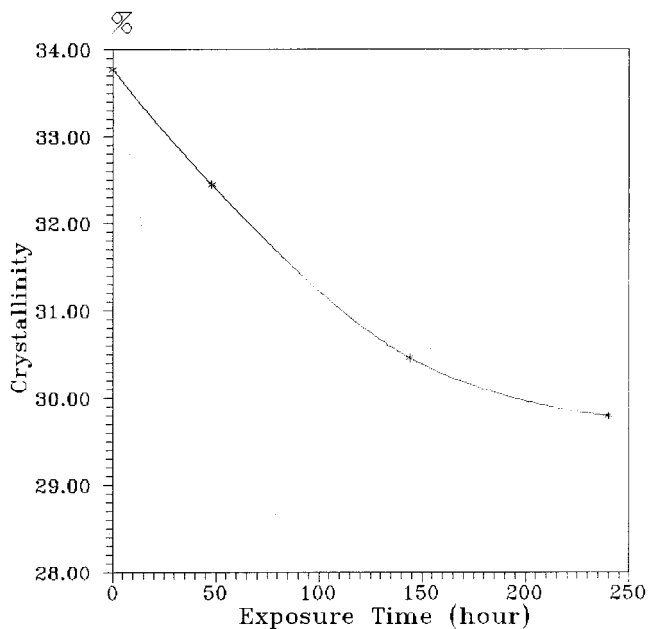


Figure 11 The crystallinity and time of exposure.

from the existing space charges and the residual electric field in the fibers after preparation.⁴²

The form birefringence is usually neglected. It is evident that the birefringence yields reliable information about the orientation in single-phase systems, but additional information is required when two-phase systems are involved.^{29,43} If the sign of the birefringence in the spherulite is positive, this means that the layer refractive index lies along the radial direction; if the sign is negative, then the larger index lies at right angles to this along the tangential direction. The values obtained for the present form birefringence follow

the negative sign; the form birefringence will in theory fall to zero when the refractive index difference between the phases is reduced to zero. Thus, UV radiation for nylon 6 changes its internal structural parameters.

The crystalline regions in the polymer are affected by radiation. One can expect an increase in some of the structural parameters such as the surface reflectivity, optical configuration parameter, specific refractivity of the dielectric along and across the fiber axis, segment anisotropy, and stress optical coefficient with increas-

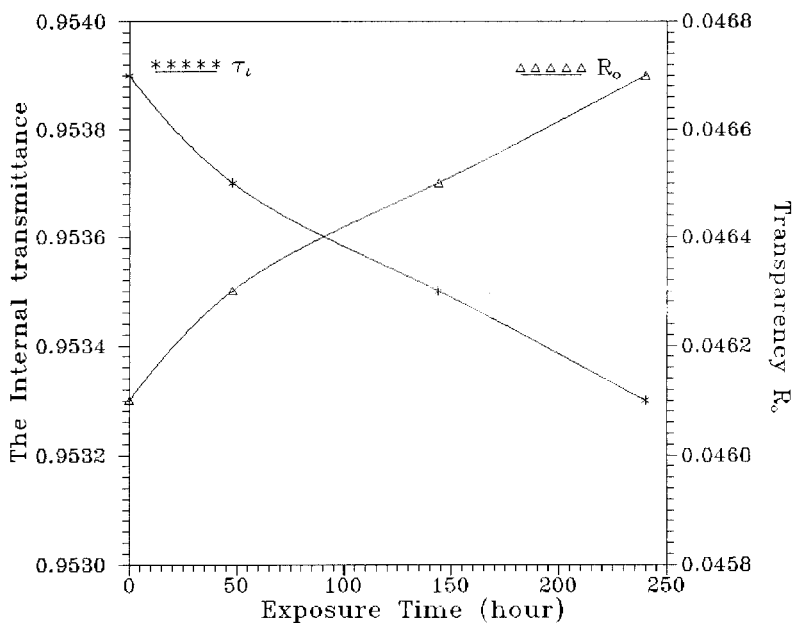


Figure 10 The internal transmittance τ_i and reflectivity factor R_o versus the time of exposure.

ing time of radiation. Such an effect was observed previously, and it was demonstrated by changes in the polymer density and the crystallinity parameters.

The radiation process is used to vary the degree of orientation and other physical properties in polymeric materials. Studying the radiation properties of nylon 6 fibers is designed to establish a connection between orientation and any other structural parameters that relate to the end use. Radiation increases the energy of the polymer and promotes branching and crosslinking reactions, thus producing thermoplastics of greater rigidity.⁴⁴

On the other hand, UV light and penetrating radiation are important kinds of physical influences on polymers that are capable of inducing chemical reactions in them. When a polymer is exposed to ionizing radiation, physical and chemical changes occur.⁴⁵ The degree to which the radiation affects the polymer depends on the chemical composition, general morphology, free energy state of the polymer, and the radiation dose applied (time, value, temperature, etc.). Such effects also cause the disordered crosslinking of the macromolecules and the formation of branched and crosslinked structures.⁴⁶

Investigations in the field of irradiation of polymers in the presence of air led to a marked oxidation of the polymer. Some investigators reported that the weight of the polymer increased after irradiation in air, whereas gas evaluation caused a decrease in the weight if irradiation occurred *in vacuo*.⁴⁷

The oxidation process of polyamides is also a chain reaction and begins at the hydrogen in the α positions relative to the NH group.⁴⁸ The color changes that occur in the presence of air are generally due to oxidation.

CONCLUSIONS

1. Most of the investigated structural parameters were affected by UV irradiation. Increasing the exposure time of irradiation led to an increase in the optical orientation functions. The principal optical parameters are suitable for evaluating the dielectric constant, the dielectric susceptibility, and the surface reflectivity in irradiated nylon 6 fibers.
2. The polarizability of a monomer unit, specific refractivity of the dielectric along and across the fiber axis, the optical configuration parameter, and the stress optical coefficient increase as the time of irradiation increases.
3. The number of molecules per unit volume is unaffected by the time of irradiation.
4. The density and crystallinity decreased with the increase of the time of radiation for nylon 6 fiber (Table I).

5. Color changes in irradiated nylon 6 were observed (R_o and τ_i , Figure 10), which were due to the oxygen chemical reaction.

Thus, the influence of radiation on the physical properties of polymers differs widely, depending on whether the polymer crosslinks or degrades. Radiation degradation always causes a steady decrease in most of the valuable properties of polymers and needs further study.

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