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Optothermal properties of fibres

Part I *Optical behaviour of annealed polypropylene fibres as a function of the draw ratio*

A. A. HAMZA, I. M. FOUDA, T. Z. N. SOKKAR, M. M. SHAHIN, E. A. SEISA
Physics Department, Faculty of Science, Mansoura University, Mansoura, Egypt

Studies of the mechanical and optical properties of undrawn polypropylene fibres by annealing and drawing were performed. The optical properties and strain produced in polypropylene fibres at different conditions were measured interferometrically at room temperature. It was found that as the draw ratio of the fibre increased, its birefringence, Δn_a, increased at a constant rate, and then nearly levelled off. The refractive index, $n^{\parallel}$, and polarizability, $p^{\parallel}$, increased with different draw ratios; but for fibres annealed at 70 and 100 °C, there were no acceptable variations. For fibres annealed at 130 °C, $n^{\parallel}$ and $p^{\parallel}$ increased compared to those fibres annealed at 70 and 100 °C. An empirical formula has been suggested to explain the relationship between the cross-sectional area of polypropylene fibres with the draw ratio, and the constants of this formula have been determined. The effect of annealing on the refractive index profile of undrawn polypropylene fibres, before and after thermal treatment, was studied. The strain optical coefficient and the Poisson's ratio were calculated over different draw ratios. The results obtained clarify the effect of annealing time and temperature with different draw ratios on the optical behaviour of polypropylene fibres. Microinterferograms are given for illustration.

1. Introduction

Part of the modern trend in fibre research is to alter fibre properties. One of the methods for property modification involves the drawing process at different conditions. Synthetic fibres are produced from the melt by extrusion through fine holes, this is called spinning. The resulting thread has no desirable textile properties and has low birefringence. In order to turn it into a useful textile fibre it must be mechanically drawn out to make it thinner, stronger and, consequently, more birefringent. Thus, a distinction can be made between undrawn and drawn synthetic fibres. The values for the birefringence of these fibres have proven to be useful in the adjustment of the drawing process [1]. The phenomenon of drawing represents a series of variations upon a theme, or rather several themes, for there are so many relevant variables, including the temperature of drawing, its strain rate, initial morphology and molecular mass distribution, among others. Stretching at room temperature is known as cold draw and obviously concerns deformation of preexisting structures [2]. During the production of films, fibres and engineered parts, a polymer is exposed to varying degrees of heat and humidity. The processing conditions have a significant influence on such properties as the mechanical behaviour, dimensional stability, appearance and dye diffusion. The structural basis for these changes in the performances of the material can be studied by controlled annealing of the specimens. Several studies have been reported on the effect of annealing on the structure of synthetic and natural fibres [3–6]. Annealing implies heating a solid to temperatures approaching its melting point. This activates internal mobility and promotes greater stability by, for example, the elimination of stresses or defects in a general movement towards the thermodynamic equilibrium condition [2].

Annealing may be performed with the ends of the sample free or fixed. In the former case the sample shrinks; whereas in the latter case it retains its length, but exerts measurable retractive forces on its fixed ends. Both effects increase with increased annealing temperature [7].

2. Experimental procedure

The Pluta polarizing interference microscope [8, 9] was used in the present work to determine the refractive indices of drawn and undrawn fibres, where

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

and

$$\Delta n_a = \frac{dz}{h}\frac{\lambda}{t_f} \quad (2)$$

where $n_a^{\parallel}$ is the refractive index of fibre for light vibrating parallel (an analogous equation applies for light vibrating perpendicular to the fibre's axis), n_L is the refractive index of the immersion liquid, $dz^{\parallel}$ the fringe shift inside the fibre, t_f the fibre thickness, h

is the interference fringe spacing corresponding to the wavelength λ. Also, Δn_a is the birefringence of the fibre.

3. Results and discussion

3.1. Variation of birefringence before and after annealing

The Pluta microscope [8, 9] was used to determine directly the mean birefringence of undrawn polypropylene fibres before and after annealing at 70, 100 and 130 °C at a constant annealing time of 4 h with different draw ratios. The Pluta microscope was used in conjunction with a microstrain device [10]; a parallel beam of light was incident normally on the microscope stage and the slit diaphragm was parallel to the fibre axis to produce a non-duplicated image of the fibre.

Figure 1a–e shows microinterferograms of the non-duplicated image of undrawn polypropylene fibres, before annealing, with draw ratios of 1, 2, 3, 5 and 7, respectively. Monochromatic light of 546 nm wavelength was used. The refractive index of the immersion liquid was 1.5070 at 26 °C.

Figure 2a–g shows microinterferograms of the differentially sheared non-duplicated image of polypropylene

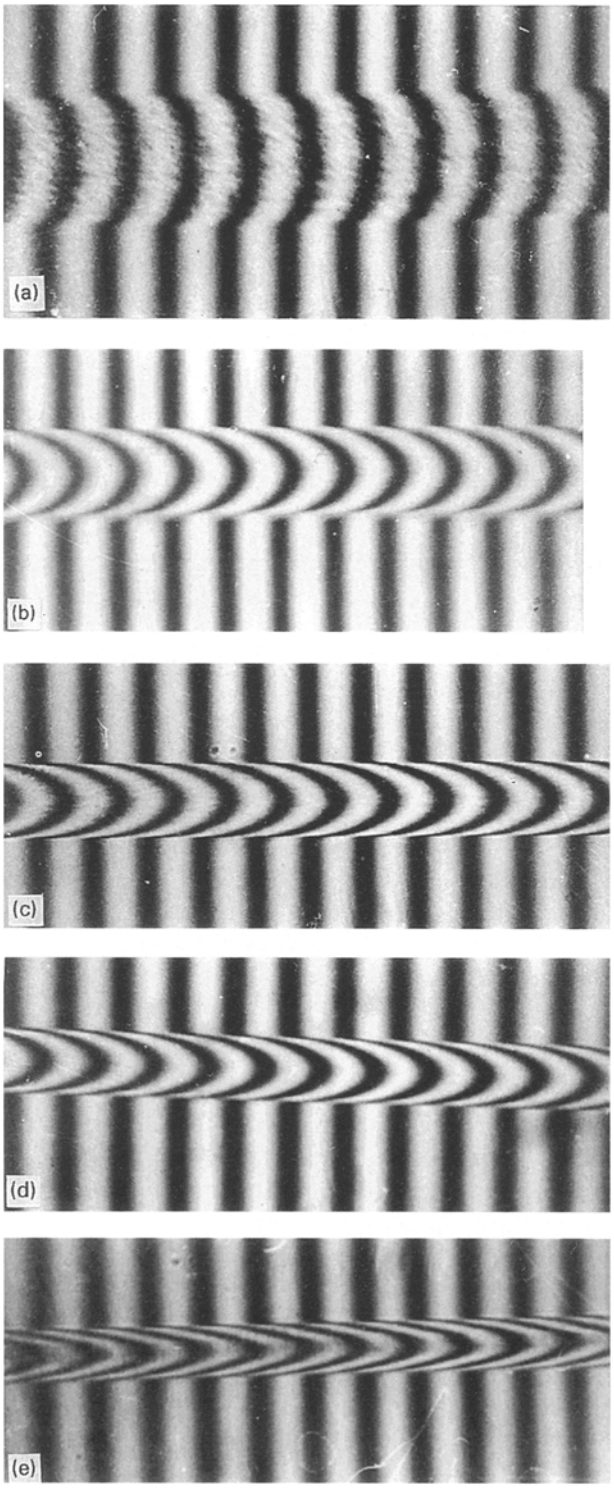

Figure 1 Differentially sheared (non-duplicated) image of undrawn polypropylene fibres before annealing, with draw ratios 1, 2, 3, 5 and 7, respectively.

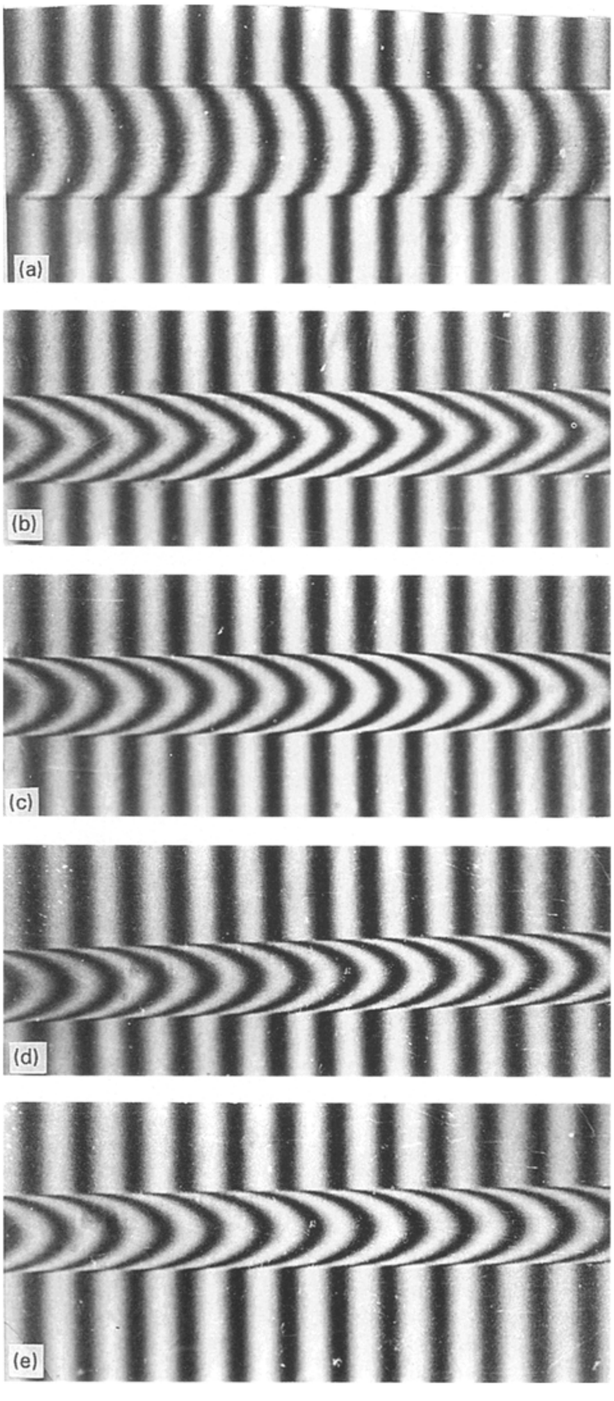

Figure 2 Differentially sheared (non-duplicated) image of undrawn polypropylene fibres annealed at 100 °C for an annealing time of 4 h, with draw ratios of 1, 2, 3, 4, 5, 7 and 8, respectively.

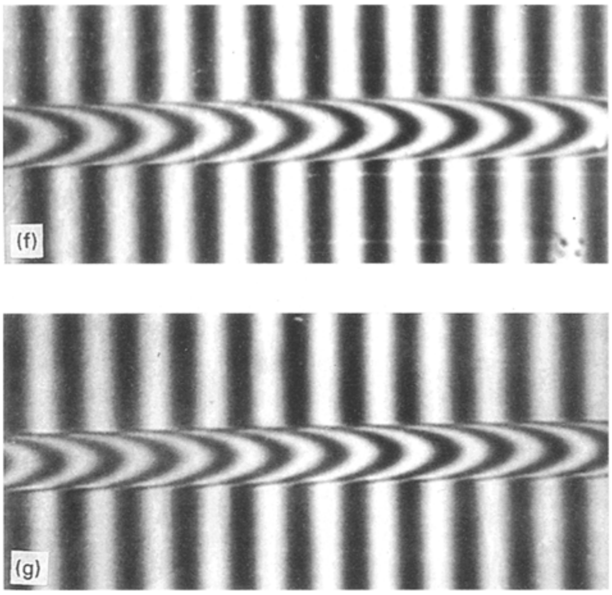

Figure 2 (continued).

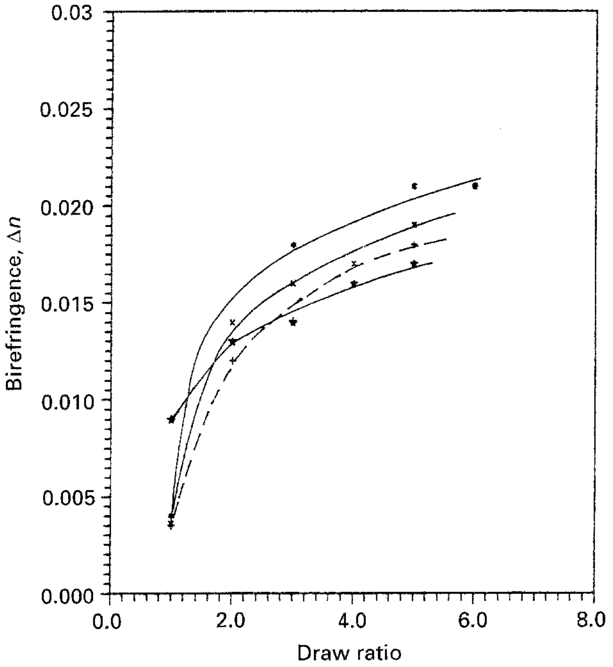

Figure 3 Relationship between the mean birefringence (direct) of undrawn polypropylene fibres, before (*) and after annealing at (×) 70, (+) 100 and (★) 130 °C at a constant annealing time of 4 h as a function of draw ratios.

TABLE I Values of the parameters which characterize the deformation process relating the birefringence with the draw ratio

Temperature of polypropylene fibre (°C)	Constant		
	m	*b*	*c*
Room temperature	0.005 547	0.478 46	0.008 371
Annealed			
70	0.031 582	− 1.055 73	0.046 854
100	0.027 622	2.186 63	− 0.000 304
130	0.025 119	− 1.142 98	0.022 657

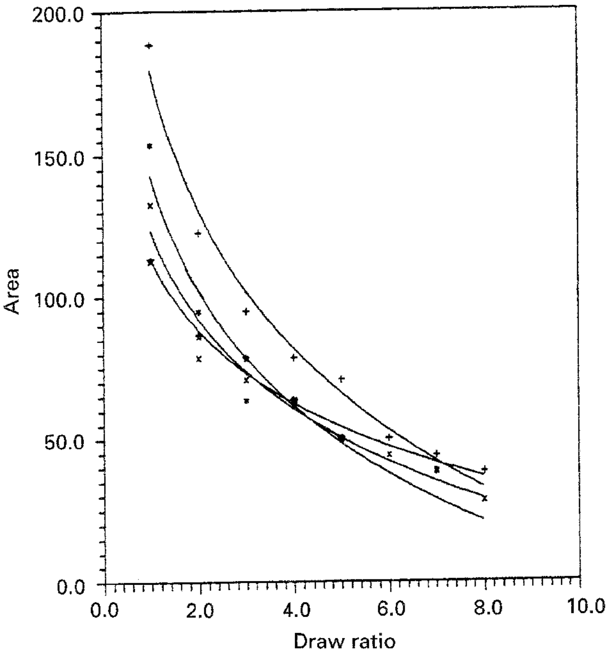

Figure 4 Relationship between the mean cross-sectional area of the fibre before (*) and after annealing at (×) 70, (+) 100 and (★) 130 °C at a constant annealing time of 4 h, as a function of draw ratios.

TABLE II Values of the parameters which characterize the deformation process relating the cross-sectional area with the draw ratio

Temperature of polypropylene fibre (°C)	Constant		
	γ	β	α
Room Temperature	− 0.418 564	− 0.7273	116.959 53
Annealed			
70	− 40.3586	0.108 879	55.061 017
100	− 36.2431	0.341 849	100.056 08
130	− 91.6071	− 0.633 459	21.320 213 4

TABLE III Poisson's ratio, μ, and strain optical coefficient, C_ε, of polypropylene fibre at various temperatures

Temperature (°C)	Draw ratio	Poisson's ratio, μ	Strain optical coefficient C_ε
Room temp	2.0	0.214	0.0090
	3.0	0.179	0.0050
	5.0	0.107	0.0150
	7.0	0.083	0.0040
70	2.0	0.231	0.0104
	3.0	0.135	0.0020
	4.0	0.103	0.0010
	5.0	0.096	0.0020
	6.0	0.085	0.0030
	7.0	0.077	0.0010
	8.0	0.077	0.0040
100	2.0	0.194	0.0085
	3.0	0.145	0.0020
	4.0	0.118	0.0020
	5.0	0.097	0.0020
	6.0	0.097	0.0030
	7.0	0.086	0.0020
	8.0	0.078	0.0040
130	2.0	0.125	0.0040
	3.0	0.083	0.0010
	4.0	0.083	0.0020
	5.0	0.083	0.0010

fibres annealed at 100 °C, for an annealing time of 4 h with draw ratios of 1, 2, 3, 4, 5, 7 and 8, respectively. Monochromatic light of 546 nm wavelength was used. The refractive index of the immersion liquid was 1.5070 at 26 °C. Using these microinterferograms and Equation 2 the mean birefringence at different draw ratios are calculated.

Figure 3 shows the general behaviour of the direct birefringence of undrawn polypropylene fibres, before and after annealing at 70, 100 and 130 °C at a constant annealing time of 4 h with different draw ratios (monochromatic light of 546 nm wavelength was used). It is clear that the birefringence, Δn, of polypropylene fibres increased with draw ratio, R, according to the following empirical relation in differential form [1]

$$\frac{\mathrm{d}\Delta n}{\mathrm{d}\ln R} = m + b\,\Delta n \tag{3}$$

Integrating Equation 3

$$\Delta n = \frac{C}{b}R^{b} - \frac{m}{b} \tag{4}$$

where m, b and C are parameters which characterize the deformation process, their values are calculated for polypropylene fibre before and after annealing at 70, 100 and 130 °C at a constant annealing time of 4 h. The results are given in Table I, where m indicates the initial slope of the birefringence's natural extension

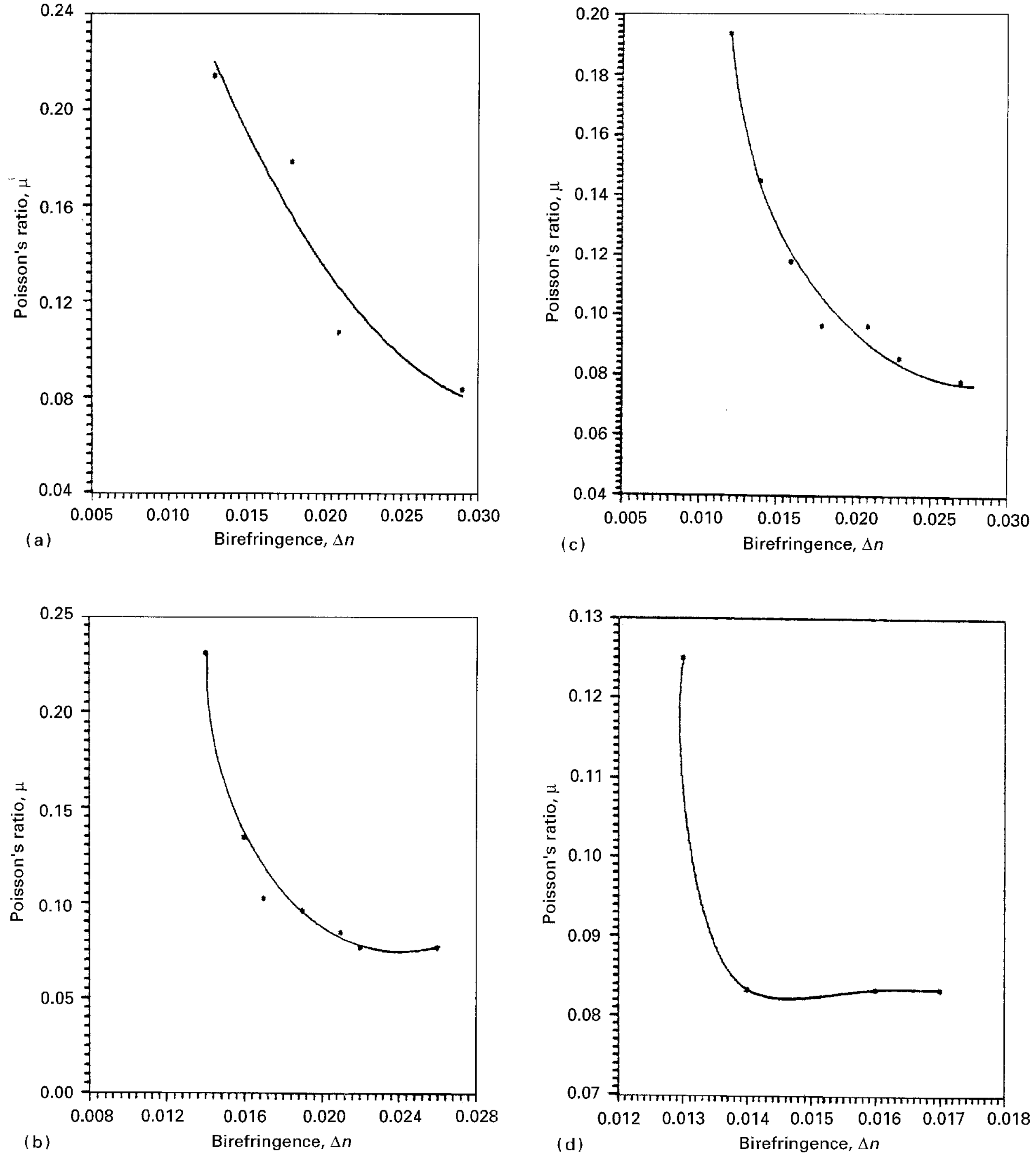

Figure 5 Birefringence variations of polypropylene fibres against Poisson's ratio at different temperatures: (a) room temperature, (b) 70 °C, (c) 100 °C, and (d) 130 °C.

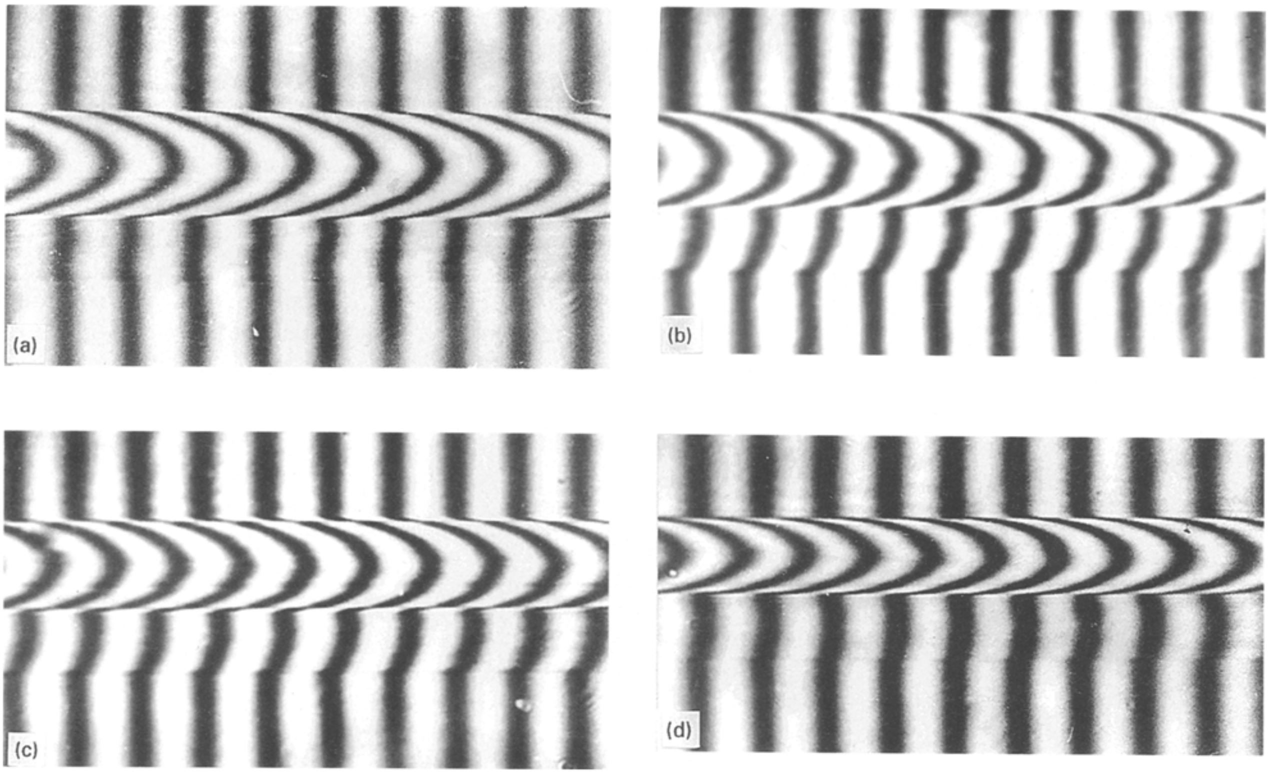

Figure 6 Totally duplicated image of undrawn polypropylene fibre before thermal treatment, with draw ratios of 2, 3, 5 and 6, respectively.

curve, if the drawing starts from the isotropic state. m can assume positive or negative values according to the birefringence sign in the fibre.

The different values of b express [11] the difference in interaction between the chain element, accordingly they become progressively better orientated with respect to the fibre axis.

Figure 4 shows the mean cross-sectional area of polypropylene fibres, before and after annealing at 70, 100 and 130 °C at a constant annealing time of 4 h, with different draw ratios. From this figure, the cross-sectional area, A, of the fibre decreases with increasing draw ratio, R according to the suggested empirical formula [12]

$$A = \frac{\alpha}{\beta} R^{-\beta} + \frac{\gamma}{\beta} \qquad (5)$$

where α, β and γ are parameters, Table II, which characterize the deformation process.

The differential form of Equation 5 reads

$$\frac{\mathrm{d}A}{\mathrm{d}\ln R} = \gamma - \beta A \qquad (6)$$

for the polypropylene fibre before and after annealing at 70, 100 and 130 °C at a constant annealing time of 4 h. The values of the parameters are given in Table II.

On straining, the fibre becomes thinner and this change in radius, r, can be related to the change in length, l, via the logarithmic Poisson's ratio, μ [12], as defined by

$$\frac{\mathrm{d}r}{r} = -\mu \frac{\mathrm{d}l}{l} \qquad (7)$$

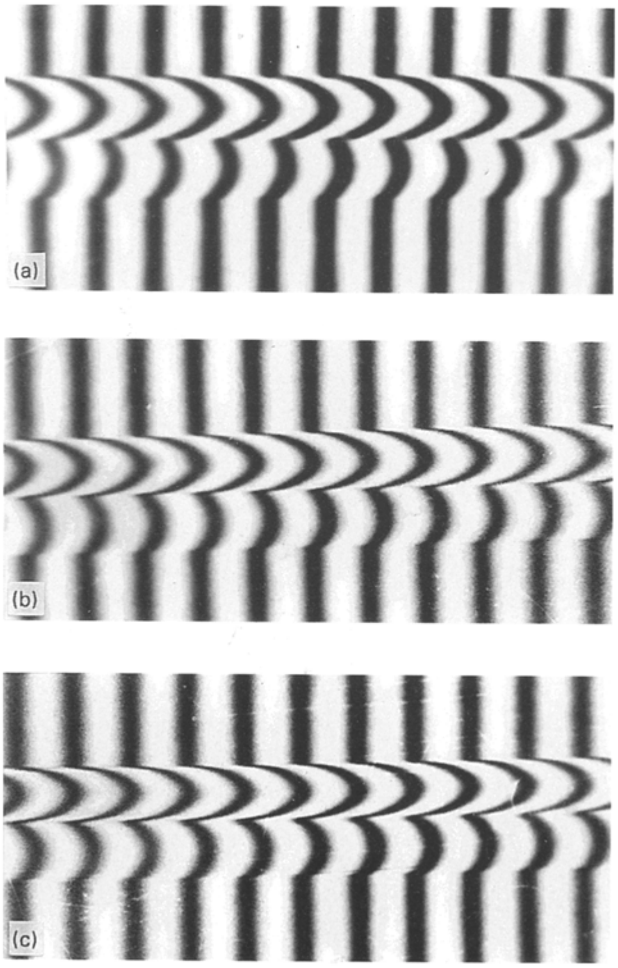

Figure 7 Totally duplicated image of undrawn polypropylene fibre, annealed at 100 °C for an annealing time of 4 h, with draw ratios of 4, 5 and 6, respectively.

The values of μ at different temperatures are given in Table III. The strain optical coefficient, C_ε, is defined as

$$C_\varepsilon = \frac{d\Delta n}{d\varepsilon} \qquad (8)$$

where ε is the strain and was determined for different strain ranges at different annealing temperatures. The results are given in Table III.

Figure 5a–d gives the relationship between the direct birefringence of polypropylene fibre, before and after annealing at 70, 100 and 130 °C, respectively, at a constant annealing time of 4 h with Poisson's ratio.

The Pluta microscope was used, with the microstrain device elsewhere [10], to determine the variation of the mean refractive index of drawn and undrawn fibres. The mean refractive index of the undrawn polypropylene fibres was determined before and after annealing at 70, 100 and 130 °C at a constant annealing time of 4 h with different draw ratios.

Figure 6a–d shows microinterferograms of totally duplicated images of undrawn polypropylene fibres before thermal treatment, with draw ratios of 2, 3, 5 and 6, respectively. Monochromatic light of 546 nm wavelength was used, and the refractive index of the immersion liquid was 1.5070 at 26 °C.

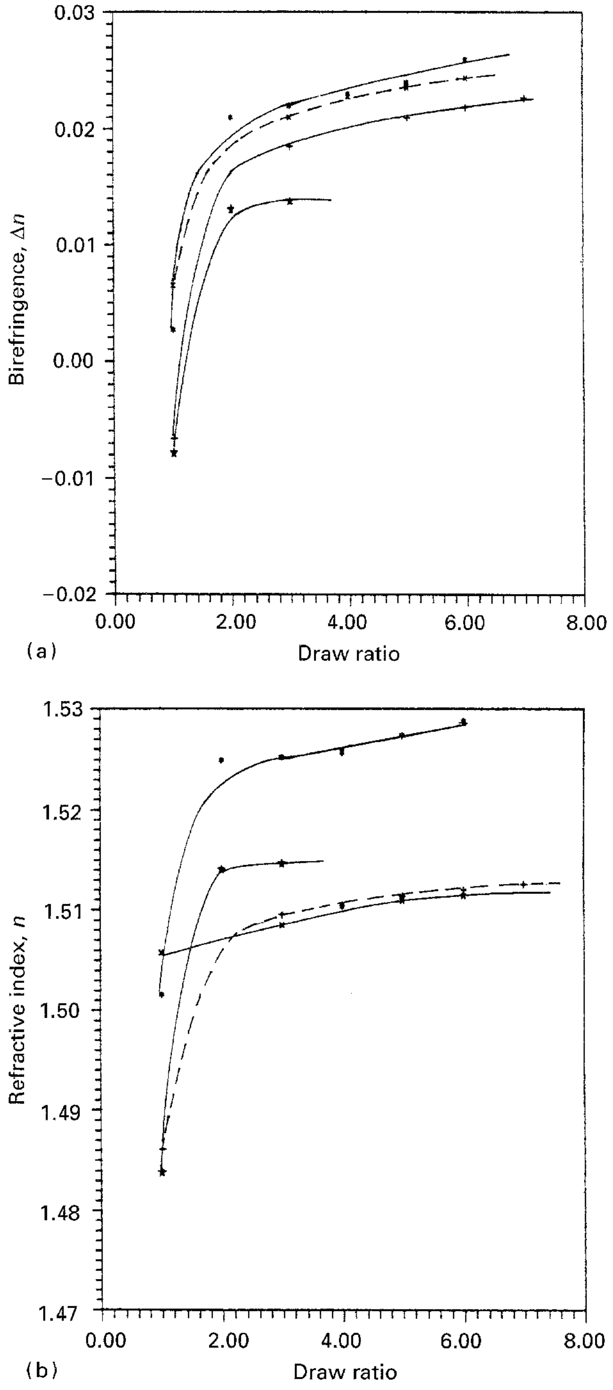

Figure 8 (a) Mean birefringence (indirect), Δn, of undrawn polypropylene fibre and (b) mean refractive index, $n^{\|}$, of undrawn polypropylene fibre, before thermal treatment (*) and after annealing at (×) 70, (+) 100 and (★) 130 °C at a constant annealing time of 4 h, as a function of draw ratios.

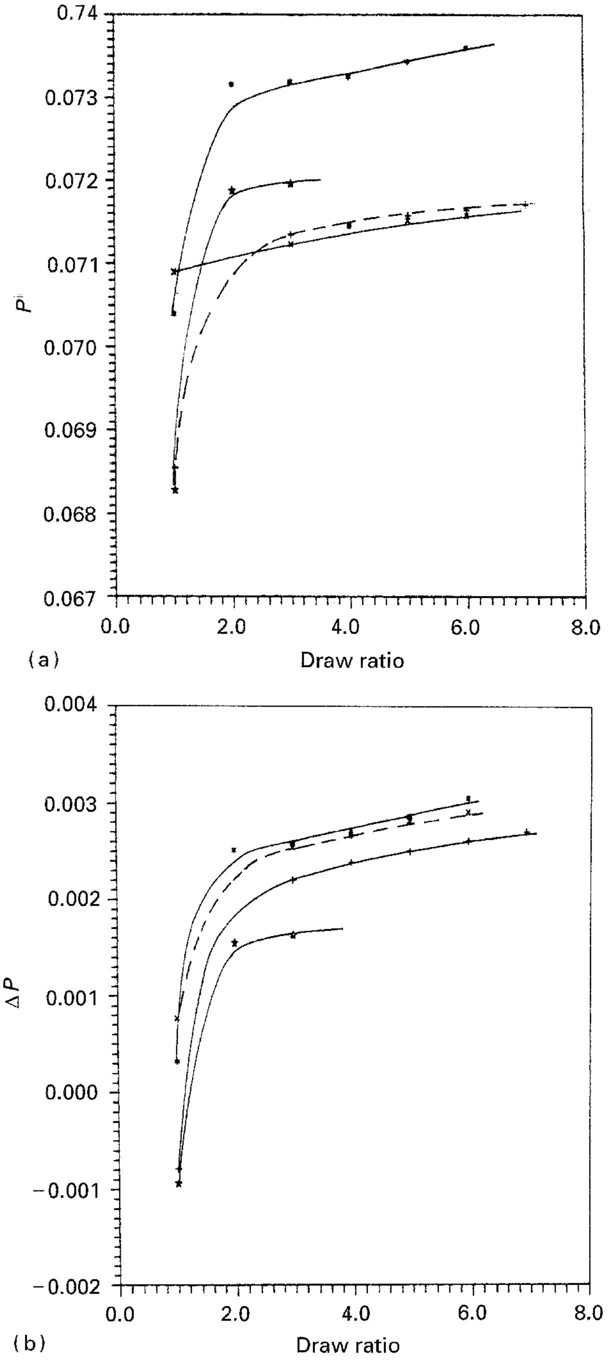

Figure 9 The polarizability of (a) $P^{\|}$, and (b) ΔP, of undrawn polypropylene fibre before (*) and after annealing at (×) 70, (+) 100 and (★) 130 °C at a constant annealing time of 4 h as a function of draw ratios.

Figure 7a–c shows microinterferograms of totally duplicated images of undrawn polypropylene fibres annealed at 100 °C for an annealing time of 4 h, with draw ratios of 4, 5, and 6, respectively. Monochromatic light of 546 nm wavelength was used, and the refractive index of the immersion liquid was 1.4960 at 22 °C.

The mean refractive index, $n^{\parallel}$ and $n^{\perp}$ and birefringence can be determined using Equation 1.

Figure 8a–b shows the general birefringence and refractive index, $n^{\parallel}$, behaviours of undrawn polypropylene fibres, before and after annealing at 70, 100, and 130 °C at a constant annealing time of 4 h, with different draw ratios (monochromatic light of 546 nm wavelength was used). It is clear from this figure that birefringence, Δn, varies with the draw ratio.

3.2. Mean polarizability per unit volume

The experimental values of the refractive indices, $n^{\parallel}$ and $n^{\perp}$, of undrawn polypropylene fibres, before thermal treatment and after annealing the fibres at 70, 100

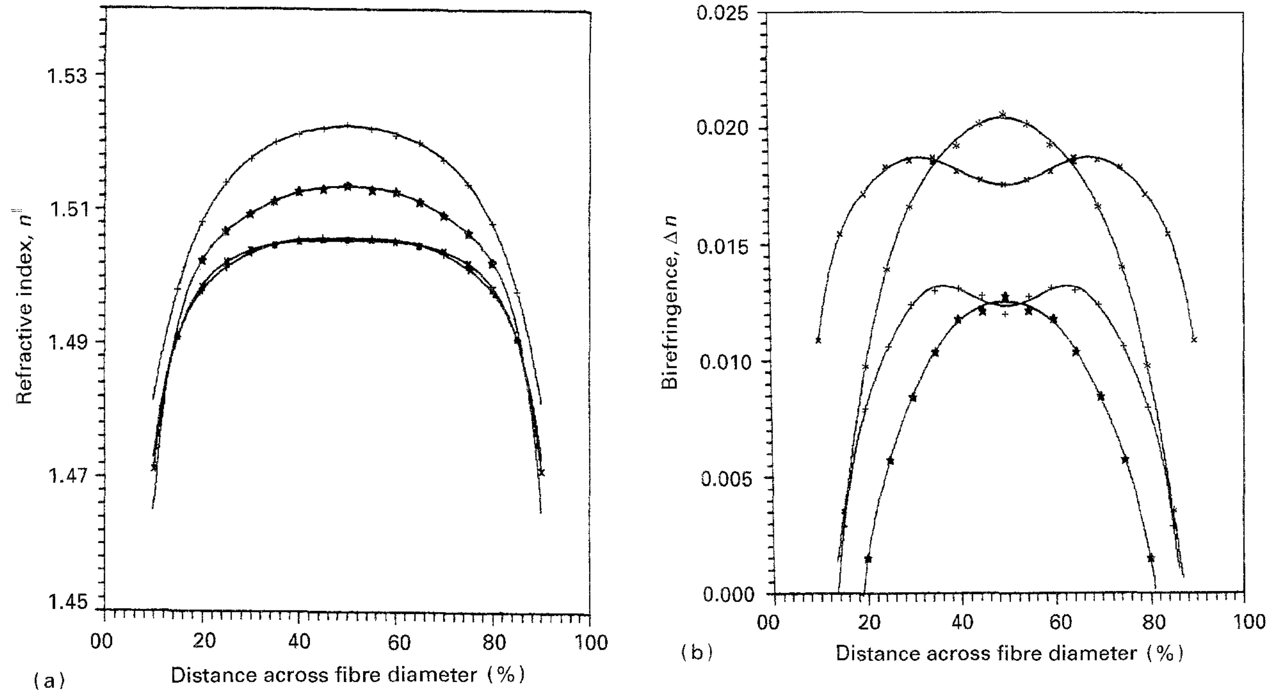

Figure 10 The variation of (a) refractive index profile, $n^{\parallel}$, and (b) birefringence profile, Δn, of undrawn polypropylene fibre before (*) thermal treatment and after annealing at (×) 70, (+) 100 and (★) 130 °C at an annealing time of 4 h and drawing to a draw ratio of 3.

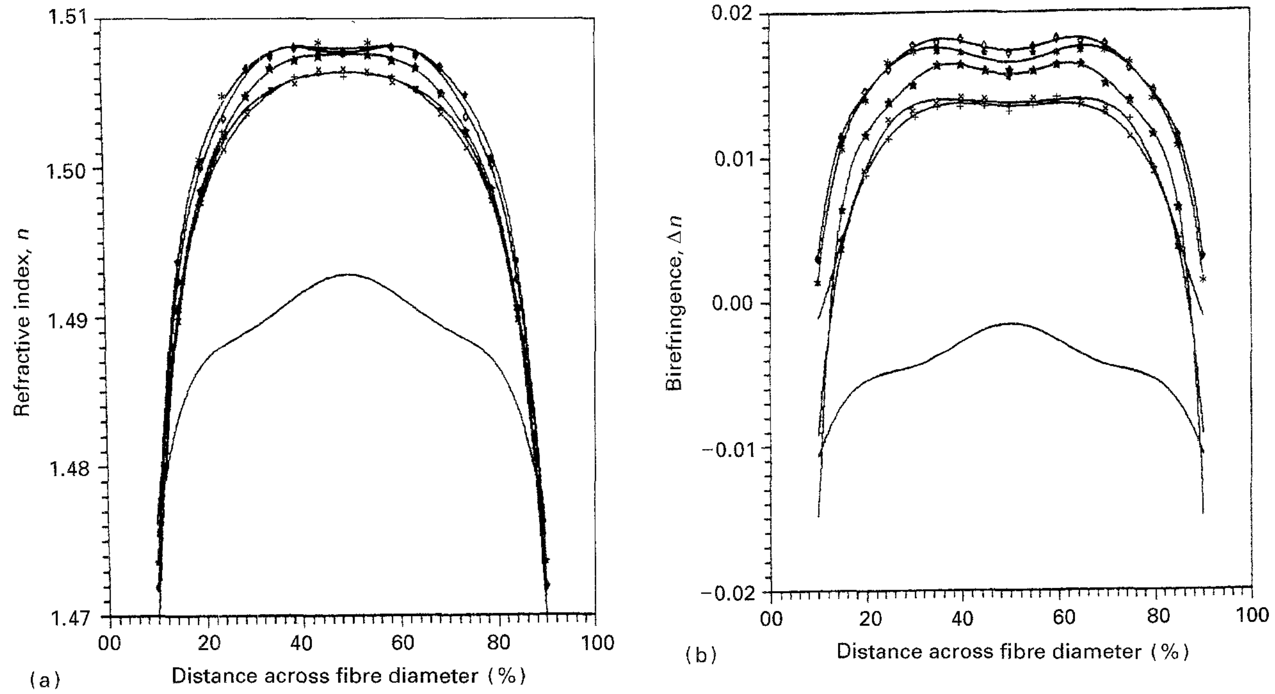

Figure 11 The variation of (a) refractive index profile, $n^{\parallel}$, of annealed undrawn polypropylene fibre, and (b) birefringence profile, Δn, of annealed drawn polypropylene fibre at 100 °C and an annealing time of 4 h with different draw ratios of (—) 1, (×) 3, (+) 4, (☆) 5, (◇) 6 and (★) 7, respectively.

and 130 °C at a constant annealing time of 4 h are determined using Equation 1 at different draw ratios. These values are used also in calculating the mean polarizability, $P^{\parallel}$, per unit volume according to Lorentz–Lorenz equation [13]

$$P^{\parallel} = 3(n_{\parallel}^{2} - 1)/4\pi(n_{\parallel}^{2} + 2)$$

and with an analogous formula for the perpendicular direction.

Figure 9a, b gives the relationship between the polarizability per unit volume of polypropylene fibre before and after annealing at 70, 100 and 130 °C at a constant annealing time of 4 h with different draw ratios.

3.3. Determination of refractive index profile in annealed polypropylene fibres by the two beam interference method taking into account refraction of the light beam by the fibre

The variation of these values across the diameter of the fibre was determined, taking into account the actual refracted beam inside the fibre [14].

Figure 10a, b shows the refractive index, $n^{\parallel}$, and birefringence, Δn, profiles of undrawn polypropylene fibres, before and after annealing at 70, 100 and 130 °C at an annealing time of 4 h with a constant draw ratio of 3.

Figure 11a, b shows the refractive index, $n^{\parallel}$, and birefringence Δn, profiles of annealed drawn polypropylene fibres at 100 °C. The annealing time is 4 h. The draw ratios are 1, 3, 4, 5, 6 and 7, respectively.

4. Conclusions

The change in optical properties during stretching undrawn polypropylene fibres was determined before and after annealing at 70, 100 and 130 °C, at a constant annealing time of 4 h. The following conclusions may be drawn

1. As the draw ratio increases, birefringence, Δn, increases at a constant rate and then levels off, reflecting an increasing crystallized volume in the fibres.
2. Stretching causes orientated crystallites to appear, leading to increases in refractive index, $n^{\parallel}$, and polarizability, $p^{\parallel}$, with different draw ratios. But for fibres annealed at 70 and 100 °C, there are no observable variations. For fibres annealed at 130 °C increases are observed in refractive index compared to those fibres annealed at 70 and 100 °C.
3. As crystallinity and orientation increase, so the ability of annealed polypropylene fibres to dye is expected to decrease [15].
4. The effect of the annealing process depends on the temperature and time of annealing. Also, the annealing process affects other physical properties (mechanical, colour, thermal, electrical, ... , etc.) of polypropylene fibres, as well as their optical properties.
5. The density, ρ, of the samples was expected to vary due to the drawing and annealing processes [6, 16].
6. On straining, the undrawn polypropylene fibre becomes thinner and this change in radius can be related to the change in length, which is a function of Poisson's ratio and strain optical coefficient changes [12].
7. The Poisson's ratio is reduced by strain effects.
8. An increase in temperature causes a decrease in the slope of the birefringence extension curve.
9. The results of the refractive index profile of undrawn and drawn fibres before and after annealing throw light on the structural variation of optical properties across the diameter of these fibres.

It is concluded from the above results that the practical importance of these measurements provides an acceptable view for the structural features of undrawn and drawn polypropylene fibres. The mechanism of optical variations associated with the annealing and drawing processes is also clarified. These optical changes throw light on the function of the amount of strain on the fibre after annealing processes.

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