Determination of Refractive Index Profile of Partially and Highly Oriented Fibers Using Double Refracting Interference Microscopy

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ABSTRACT: Double refracting polarizing interference microscope designed by Pluta is used with a suggested method to determine the refractive index profile and birefringence profile of partially and highly oriented fibers. The application of this method depends on using Pluta polarizing interference microscope in two positions (crossed position for the duplicated images and subtractive position for the nonduplicated image of the fiber). The mathematical representation of the suggested method is given. The refraction of the light beam inside the fibers is taken into consideration while measuring the fringe shift profile. The refractive index profiles of polypropylene fiber with draw ratio 3.5 are determined using the conventional method. The results are compared with those obtained with the suggested method and found to be in good agreement. The suggested method is applied to determine the refractive index profile of poly(aryl ether ether ketone) partially oriented fiber and poly(ethylene terephthalate) highly oriented fiber. The diffraction of He–Ne laser beam is used to determine the average diameters of these fibers. Microinterferograms are given for illustration. © 2003 Wiley Periodicals, Inc. J Appl Polym Sci 87: 2341–2347, 2003

Key words: refractive index; fibers; poly(ether ketones)

INTRODUCTION

Interference microscopy has long been an invaluable technique for analyzing polymer fibers. It is ideally suited for measuring local refractive indexes, generally good indicators of molecular orientation, and density fluctuations. The double refracting polarizing interference microscope[1,2] is capable of giving either the uniform or fringe interference field with variable amounts and direction of lateral image duplication. The main advantage of this microscope is that it has two double refracting prisms of the Wolaston type. These prisms can be adjusted to give two positions. The first gives directly the nonduplicated image for the birefringence measurements and the other gives the duplicated images for the refractive index measurements for the two components of the vibrating light. These two positions are called subtractive and crossed positions,[3] respectively.

Hanns[4] developed a method to calculate the refractive index profile from the microinterferogram obtained for a piece of fiber examined transversally to its axis without fiber destruction. The fiber cross section is divided into circular zones, each of which is assumed to have a constant refractive index. This is called the ring zonal method. The refractive index of each zone is then measured from the interference fringe shift corresponding to it. Sokkar[5] derived a mathematical formula for the determination of the refractive index profile of a fiber with elliptical cross section by assuming elliptical zones of constant refractive index.

Hamza et al.[6] described a method to determine the refractive index profile of a cylindrical multilayer fiber using a two-beam interference method without the necessity of measuring the refractive index of the immersion liquid. In addition, Hamza et al.[7–9] derived mathematical expressions for homogeneous and heterogeneous (skin–core and multilayers) fibers to measure the refractive index profile, taking the refraction into consideration.

The highly oriented synthetic fibers are termed high-performance fibers and have a high degree of orientation.[10] It gives a large optical path difference in case of using immersion liquid of low refractive index to measure its refractive indexes and birefringence. The measurement of the refractive index profiles of these highly oriented fibers will be strongly affected by the method of calculation. In other words, if we consider that the incident light beam crosses the fiber without refraction, the error due to this approximation will be detectable, while better results can be given if the refraction of the light beam by the fiber is taken into consideration.[9]

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Previous studies[3,5–9,11–13] for measuring the refractive index profile of fibers can be applied only if a suitable refractive index of immersion liquid is used. The connection of the fiber fringe shift, at the edges, with the immersing medium fringe depends on the liquid used. Recently, Sokkar and El-Bakary[14] suggest a method for measuring the refractive index profile of highly oriented fibers that depends mainly on the principle of the variable wavelength interference technique.[15]

In this work, Pluta double refracting polarizing interference microscope is used with a suggested method to determine the refractive index profile and birefringence profile of polypropylene fiber with draw ratio 3.5, poly(aryl ether ether ketone) (PEEK) partially oriented fiber, and poly(ethylene terephthalate) (PET) highly oriented fiber.

EXPERIMENTAL

The double refracting polarizing interference microscope[1,2] designed by Pluta is especially suitable for microinterferometry of birefringent fibers. The setup for the Pluta microscope is shown in Figure 1. It consists of two Wollaston double refracting prisms, W_o and W_t. One of these prisms (W_o) is installed in the microscope objective while the other (W_t) is positioned in the microscope tube. Adjusting these prisms, the microscope can work in two positions, which are the subtractive and the crossed positions. The objective prism W_o is adjusted by rotation at its subtractive or crossed position with respect to the main prism W_t. The subtractive position applies to the measurement of the fiber birefringence while the crossed position applies to the measurement of the fiber refractive indexes.

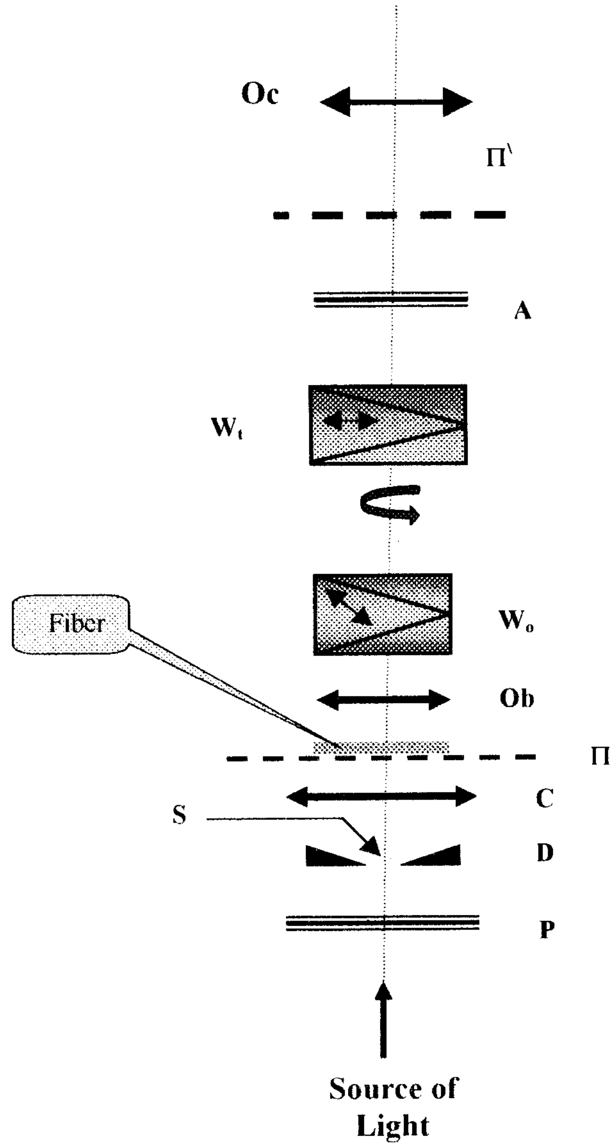


Figure 1 The setup for double refracting interference microscope[1,2] with two birefringent prisms W_o and W_t. P, polarizer; A, analyzer; Π, object plane; Π′, image plane; Ob, objective; C, condenser; D, slit diaphragm; Oc, ocular.

Theoretical considerations

Pluta microscope in crossed position

Using the double refracting polarizing interference microscope in the crossed position, a duplicated images is obtained from which the refractive indexes $n^{\parallel}$ and $n^{\perp}$ can be measured using the following equations[16]:

$$n^{\parallel} = n_L \pm \frac{Z^{\parallel}\lambda}{2ht} \quad (1)$$

$$n^{\perp} = n_L \pm \frac{Z^{\perp}\lambda}{2ht} \quad (2)$$

where $Z^{\parallel}$ and $Z^{\perp}$ are the fringe shifts when using light vibrating parallel and perpendicular to the fiber axis, respectively. λ is the wavelength of the used light, t is the fiber thickness, h is the interfringe spacing, and n_L is the refractive index of the immersion liquid. The negative sign in the above equations is used in case of $n < n_L$ and the positive sign is used in case of $n > n_L$. The double refraction or birefringence is given by[10]

$$B = n^{\parallel} - n^{\perp} \quad (3)$$

By substituting the perpendicular refractive index $n^{\perp}$ from eq. (2) into eq. (3), one gets

$$B = n^{\parallel} \pm \frac{\lambda}{2h}\left(\frac{Z^{\perp}}{t}\right) - n_L \quad (4)$$

The radius of the fibers can be divided into layers: each layer of constant refractive index and constant birefringence. For the first layer, the above equation can be rewritten as

$$B_1 = n_1^{\|} \pm \frac{\lambda}{2h}\left(\frac{Z_1^{\perp}}{t}\right) - n_L \tag{5}$$

for the second layer,

$$B_2 = n_2^{\|} \pm \frac{\lambda}{2h}\left(\frac{Z_2^{\perp}}{t}\right) - n_L \tag{6}$$

and in general,

$$B(\mathrm{r}) = n^{\|}(\mathrm{r}) \pm \frac{\lambda}{2h}\left(\frac{Z^{\perp}(\mathrm{r})}{t}\right) - n_L \tag{7}$$

Pluta microscope in subtractive position

In this case, a nonduplicated image is obtained from which the birefringence (B) can be measured directly using the following equation[10]:

$$B = \frac{\Delta Z \lambda}{2ht} = \frac{\lambda}{2h}\left(\frac{\Delta Z}{t}\right) \tag{8}$$

Considering that the fiber has multilayers, with each layer a constant birefringence, eq. (8) can be rewritten in the following form:

$$B(r) = \frac{\lambda}{2h}\left(\frac{\Delta Z(r)}{t}\right) \tag{9}$$

Refractive index profile of partially and highly oriented fibers

Highly oriented fibers have a large optical path difference between the immersion liquid and fiber fringe shift. The interference fringe shift cannot be easily detected from the microinterferograms, especially in case of parallel refractive index, because of the bad connection of the fiber fringes with the surrounding medium fringes. In this case, it is difficult to determine the refractive index profile in case of light vibrating parallel to the fiber axis directly from measuring the distance of the fringe shift. This distance can be determined by measuring the distances $Z^{\perp}$ when using Pluta microscope in the crossed position and the distance ΔZ when using the microscope in the subtractive position. By equating eqs. (7) and (9), one can get

$$n^{\|}(\mathrm{r}) = \frac{\lambda}{2ht}\left[\Delta Z(\mathrm{r}) \pm Z^{\perp}(\mathrm{r})\right] - n_L \tag{10}$$

$$n^{\|}(\mathrm{r}) = \frac{\lambda}{2ht} Z^{\|}(\mathrm{r}) + n_L \tag{11}$$

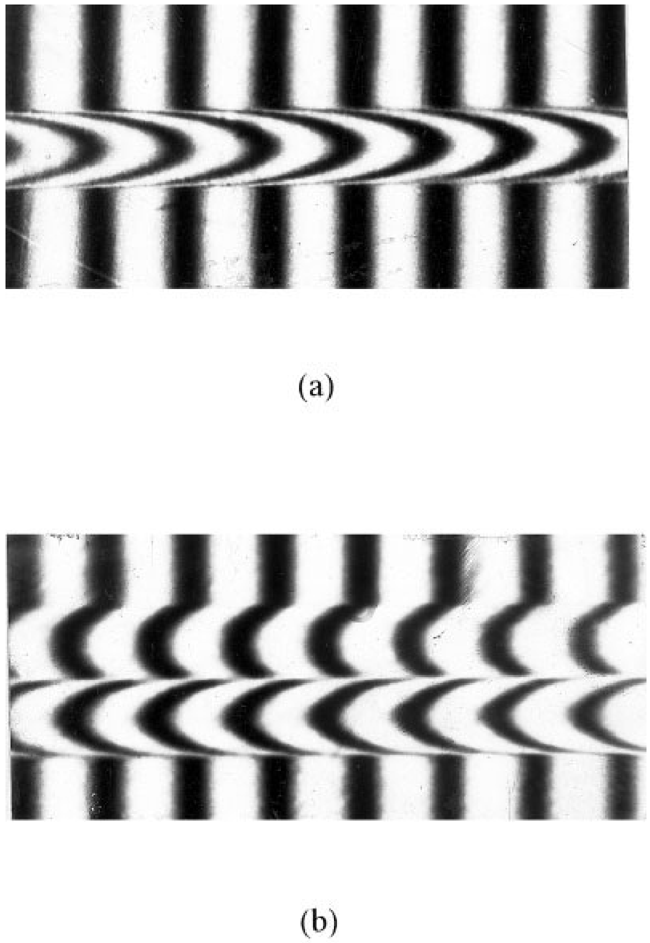


Figure 2 Microinterferograms of nonduplicated and duplicated images of polypropylene fiber with draw ratio 3.5 using monochromatic light of wavelengths 546 nm. The immersion liquid refractive index is 1.497 at 30°C.

where

$$Z^{\|}(\mathrm{r}) = \Delta Z(\mathrm{r}) \pm Z^{\perp}(\mathrm{r}) \tag{12}$$

Equation (11) gives the refractive index profile of highly oriented fibers for light vibrating parallel to the fiber axis in case of neglecting the refraction of the incident light beam by the fiber.

For the determination of the refractive index profile $n(\mathrm{r})$ of fibers taking the refraction into consideration using two-beam technique, the following equation[14] is used:

$$n^{\|}(r) = \frac{Z^{\|}(r)\lambda}{2ah\left(1 - \dfrac{r^2}{m^{\|2}a^2}\right)^{1/2}} + n_L\left[\left(1 - \frac{r^2}{a^2}\right)^{1/2}\left(1 - \frac{r^2}{m^{\|2}a^2}\right)^{1/2} + \frac{r^2}{m^{\|}a^2}\right] \tag{13}$$

where a is the fiber radius and $m^{\|}$ is the relative refractive index ($m^{\|} = n^{\|}/n_L$; $n^{\|}$ is the mean refractive

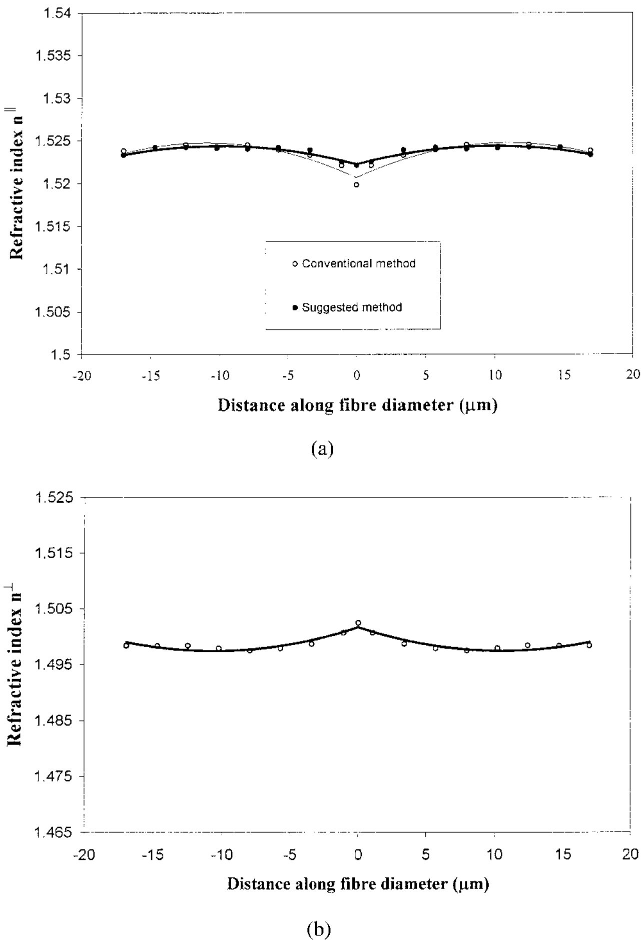


Figure 3 The refractive index profiles of polypropylene fiber with draw ratio 3.5 for light vibrating parallel (a) and perpendicular (b) to the fiber axis.

index for each layer of the fiber). In case of partially and highly oriented fibers, the value of the interference fringe shift taking the refraction of the incident beam into consideration [$Z^{\|}(r)$] is given by eq. (12). An analogous equation for the perpendicular refractive index, $n^{\perp}(r)$, can be used.

RESULTS AND DISCUSSION

Determination of fiber diameter using diffraction technique

Diffraction patterns of polypropylene with draw ratio 3.5 and PEEK and PET fibers are produced on a screen using a He–Ne laser beam.[17,18] By observing the distance (p) of the first minimum from the center of the pattern, the diameter (d) of the fiber can be determined using the following equation:

$$d = \frac{\lambda L}{p} \tag{14}$$

where L is the distance between the fiber and the screen on which the diffraction pattern observed and λ is the wavelength of the light used. The diameters are measured over different directions in the cross section and it is found that the diameter of polypropylene fiber with draw ratio 3.5 is 41.4 μm. Also, the diameter of PEEK fiber is 22.4 μm; of PET fiber, 21.8 μm. The uncertainty in calculating the fiber diameters is measured. It is found to be $\pm$0.15 μm for polypropylene fiber with draw ratio 3.5, and $\pm$0.12 μm for both PEEK and PET fibers.

Measurements of refractive index profiles and birefringence profile of polypropylene fiber with draw ratio 3.5

The double refracting polarizing interference microscope[1,2] is especially suitable for microinterferometry of birefringent fibers. This microscope is adjusted at its subtractive and crossed positions[3] to measure the birefringence profile and the refractive index profiles $n^{\|}$ and $n^{\perp}$ of polypropylene fiber with draw ratio 3.5. The sample is investigated using the suggested and conventional two-beam methods to confirm the results of the suggested method. The fiber is fixed on a glass slide with an adhesive from the two ends and a drop of a suitable immersion liquid is used with a thin glass cover placed on it. The glass slide is placed on the microscope stage. The intensity of the interference field and the contrast of the fringes are optimized by adjusting the width of the microscope condenser slit.

The microscope is adjusted first in its subtractive position so that a nonduplicated image is obtained. Figure 2(a) gives the microinterferogram of nonduplicated images of polypropylene fiber with draw ratio 3.5 using monochromatic light of wavelengths 546 nm. The immersion liquid refractive index is 1.497 at 30°C. Also, the microscope is adjusted in its crossed position to obtain the duplicated image of the fiber under

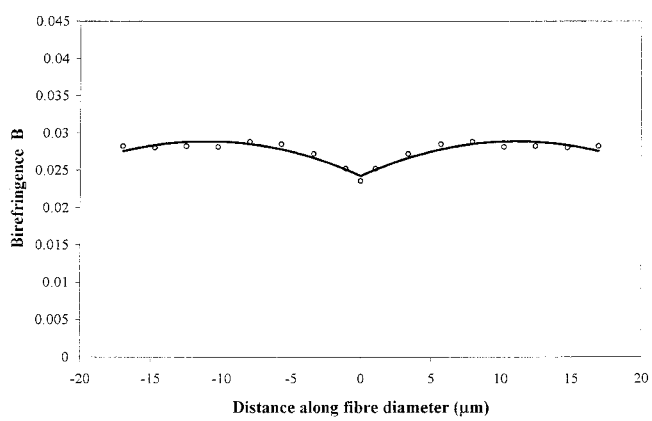
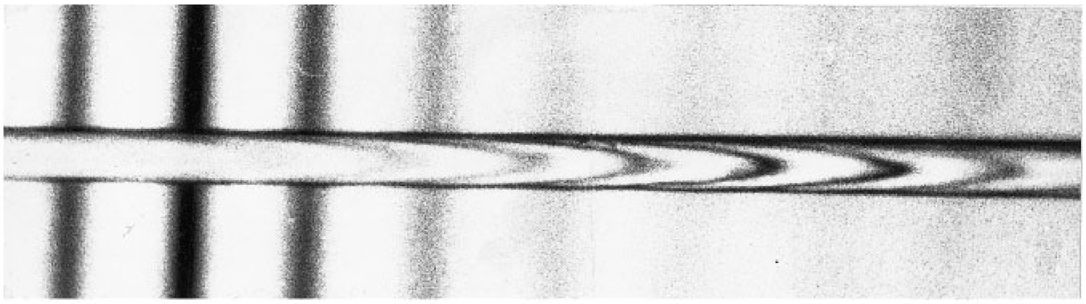
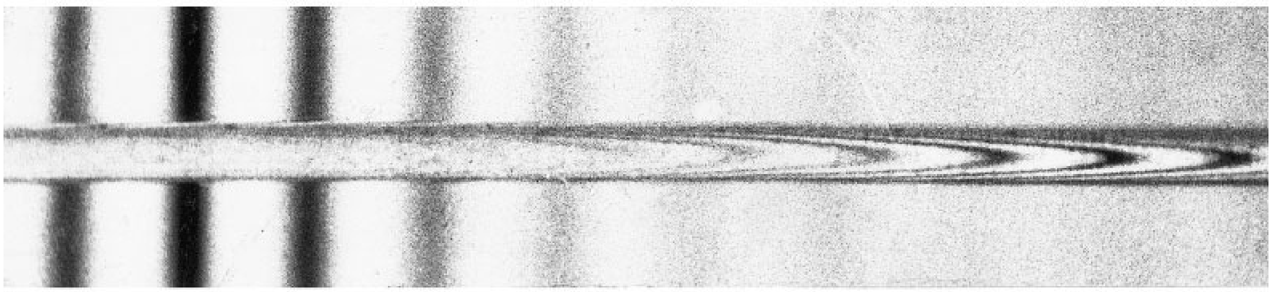


Figure 4 The birefringence profile of polypropylene fiber with draw ratio 3.5 using two-beam interference microscope.



(a)



(b)

Figure 5 Microinterferograms of the nonduplicated images of PEEK (a) and PET (b) fibers. The wavelength of the monochromatic light used is 550 nm.

study. Figure 2(b) gives the microinterferogram of duplicated image of polypropylene fiber with draw ratio 3.5 using the same wavelength and the same immersion liquid.

From the microinterferogram [Fig. 2(b)], the fiber fringe shifts are divided into layers and the distance under the fringe shift is measured due to each layer in case of light vibrating parallel [$Z^{\parallel}(r)$] and perpendicular [$Z^{\perp}(r)$] to the fiber axis. Using eq. (13) with the measured distances from the microinterferograms, the refractive index profiles $n^{\parallel}(r)$ and $n^{\perp}(r)$ are calculated using conventional technique. Also, using the microinterferograms [Fig. 2(a)], the fiber fringe shifts are divided into layers and the distances under the fringe shift in case of the nonduplicated images to determine $\Delta Z(r)$. These distances are added to the distances under the fringe shifts $Z^{\perp}(r)$ in case of light vibrating perpendicular to the fiber axis, so that the distances under the fringe shift in case of light vibrating parallel to the fiber axis is obtained according to eq. (12). In this case, eq. (12) is used with its negative sign. The results of these measurements are used with eq. (13) to calculate the refractive index profile, taking the refraction of the incident light beam into consideration, for the polypropylene fiber with draw ratio 3.5. The birefringence profile is obtained from the difference between the refractive index profiles $n^{\parallel}(r)$ and $n^{\perp}(r)$ according to eq. (3).

Figure 3(a) shows the refractive index profiles, $n^{\parallel}(r)$, for light vibrating parallel to the fibers axis for polypropylene fiber with draw ratio 3.5 using the conventional and the suggested methods. Figure 3(b) shows the refractive index profile, $n^{\perp}(r)$, for light vibrating perpendicular to the fiber axis using the conventional technique. Figure 4 shows the birefringence profile of polypropylene sample with draw ratio 3.5 using monochromatic light of wavelength 546 nm.

It is clear that the results of the suggested and conventional methods, shown in Figures 3 and 4, for the refractive indexes and birefringence profiles of

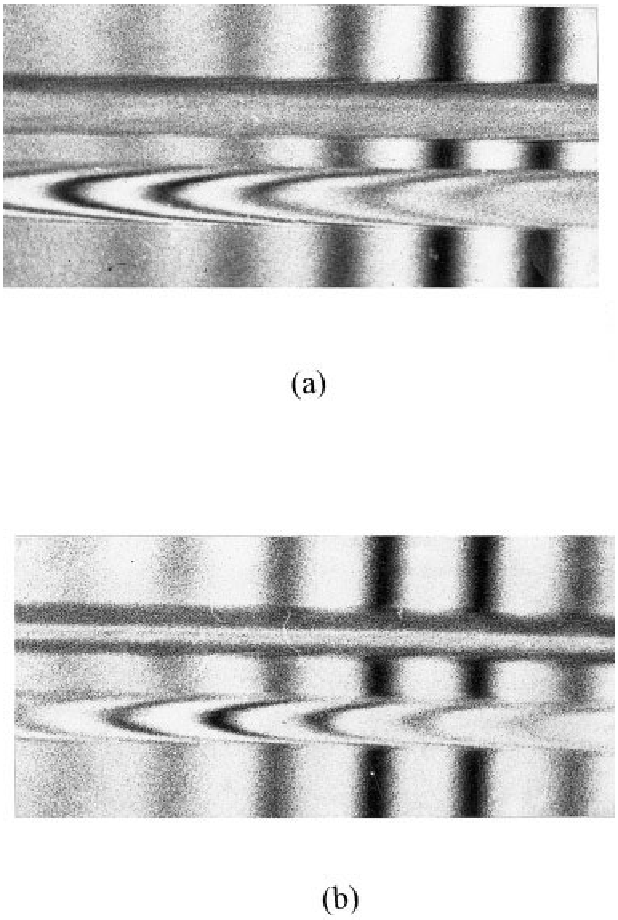


Figure 6 Microinterferograms of the duplicated images of PEEK (a) and PET (b) fibers. The upper image for parallel direction and the lower for the perpendicular direction. The wavelength of the monochromatic light used is 550 nm.

polypropylene fiber with draw ratio 3.5 are in good agreement. Also, the results show that the polypropylene fiber with draw ratio 3.5 has a homogeneous structure.

Measurements of refractive index profiles of PEEK partially and PET highly oriented fibers

The suggested method is used to determine the refractive index profiles of PEEK partially oriented fiber and PET highly oriented fiber. Figure 5 gives the microinterferograms of the nonduplicated images of PEEK and PET fibers, respectively. The refractive index of the immersion liquid in case of PEEK fibers is 1.592 at 25°C and in case of PET fibers is 1.476 at 25°C. The wavelength of white light used is considered to be 550 nm.

Figure 6(a,b) gives the microinterferograms of the duplicated images of PEEK and PET fibers, respectively. The immersion liquids of the same refractive indexes 1.592 and 1.476, respectively, are used. The temperature of the experiment was 25°C and the wavelength of the light used was considered to be 550 nm for white light. The direction of the fringe shift inside the fiber depends on the relative values of the mean refractive index of the fiber and the refractive index of the immersion liquid.

From these microinterferograms (Figs. 5 and 6), the interference fringe shift in case of the nonduplicated images are added to the fringe shifts in case of the lower image, in which light vibrating perpendicular to the fiber axis, of the duplicated images according to eq. (12) with its positive sign. Thus, the distances under the fringe shift in case of light vibrating parallel to the fiber axis is obtained. The results of these measurements are used with eq. (13) to calculate the refractive index profile, taking the refraction of the incident light beam into consideration, of PEEK partially oriented fiber and PET highly oriented fiber for light vibrating parallel and perpendicular to the fiber axis. The birefringence profile is obtained from the difference between the refractive index profiles $n^{\parallel}$(r) and $n^{\perp}$(r) according to eq. (3).

Figure 7 shows the refractive index profiles, $n^{\parallel}$(r) and $n^{\perp}$(r), for light vibrating parallel and perpendic-

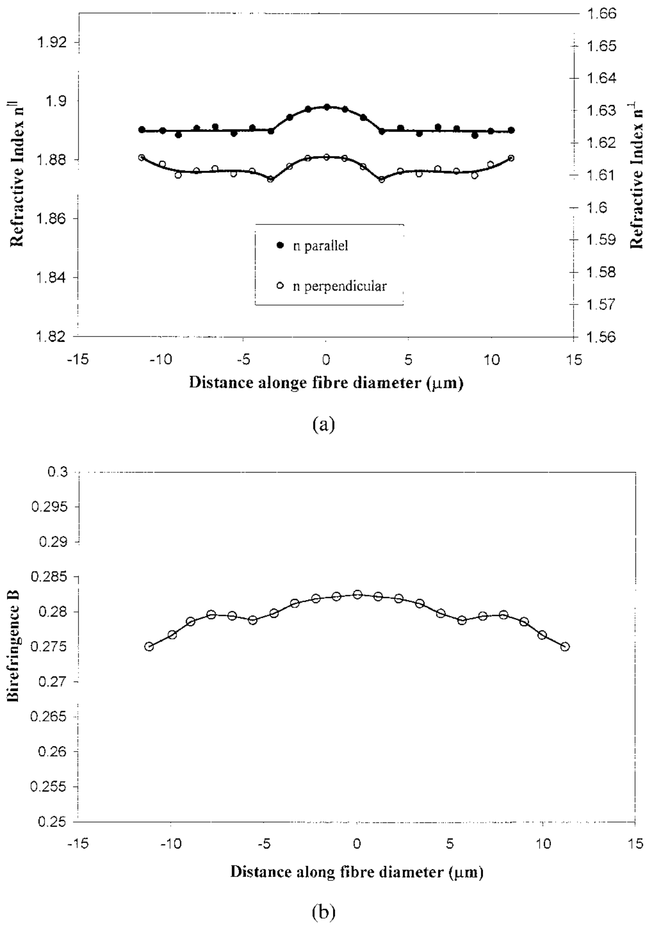


Figure 7 The refractive index profiles of PEEK fibers using the suggested method (a) and the birefringence profile of PEEK fibers (b).

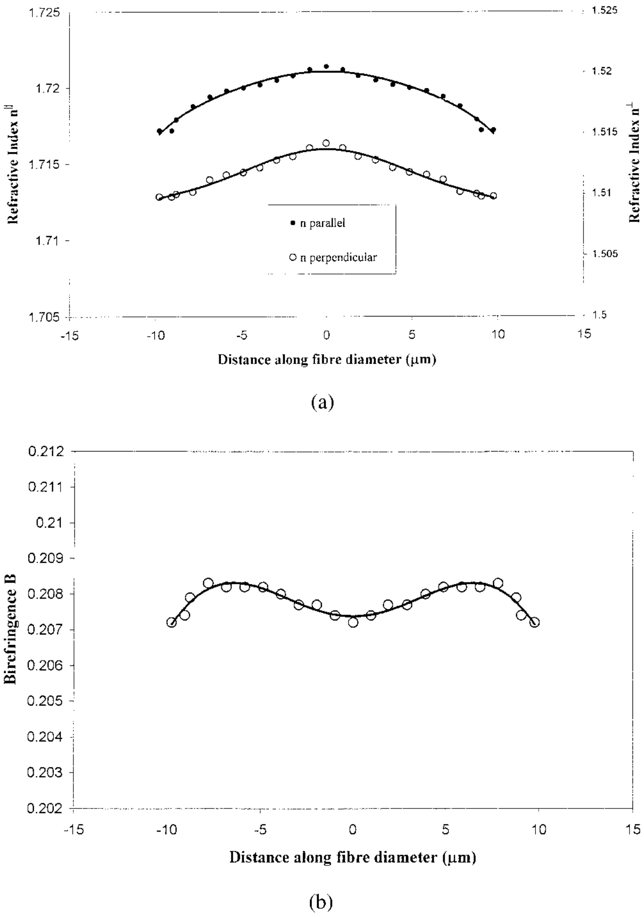


Figure 8 The refractive index profiles of PET fibers using the suggested method (a) and the birefringence profile of PET fibers (b).

ular to the fibers axis for PEEK partially oriented fiber in Figure 7(a), while the birefringence profile of PEEK fiber is given in Figure 7(b). It is clear form the refractive indexes and birefringence profiles of PEEK fiber that the fiber has a skin–core structure. The skin layer is of homogeneous structure; it has nearly a constant refractive index. But the core layer has a refractive index increased gradually until its maximum value at the fiber center. Figure 8(a) shows the refractive index profiles, $n^{\parallel}(r)$ and $n^{\perp}(r)$ for the PET highly oriented fiber for light vibrating parallel and perpendicular to the fibers axis. Figure 8(b) gives the birefringence profile of PET fiber.

The results of the measurements of the optical path length difference (the interference fringe shift for the fiber) have an accuracy of about 0.05 λ, where λ is the wavelength of the light used, and therefore the error in the determination of the refractive index and birefringence cannot be better than 0.003–0.001 (cf. Pluta[2]).

CONCLUSIONS

A method depending on using the double refracting polarizing interference microscope in two positions is suggested for measuring the refractive index profiles of partially and highly oriented fibers. For such fibers, the fringe shifts for light vibrating parallel to the axis are very difficult to be measured. This method is applied to measure the refractive indexes and birefringence profiles of PEEK partially oriented fiber and PET highly oriented fiber taking the refraction of incident light beam into consideration.

The refractive index profiles and birefringence profile of polypropylene fiber with draw ratio 3.5 are determined using the suggested and the conventional methods. The results of the two methods are in good agreement.

Pluta microscope is a very suitable technique for measuring the refractive index profile of such partially and highly oriented fibers. A limitation, however, must be taken into account when using this method, since the fibers have suitable optical path difference. In other words, their fringe shift in the subtractive position must be detected in the field of view of the microscope. Also, one must choose carefully the immersion liquid to minimize the fringe shift as much as possible.

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