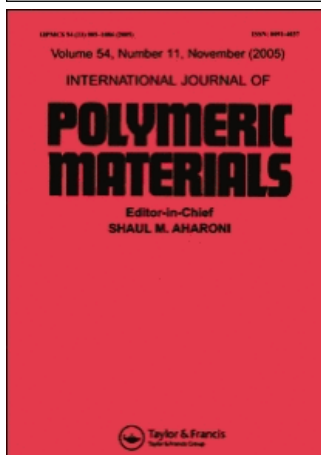


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A. A. Hamza<sup>a</sup>; T. Z. N. Sokkar<sup>b</sup>; M. A. EL-Bakary<sup>b</sup>; A. M. Ali<sup>b</sup>; M. A. El-Morsy<sup>c</sup>

<sup>a</sup> British University, El Sherouk City, Egypt

<sup>b</sup> Physics Department, Faculty of Science, Mansoura University, Mansoura, Egypt

<sup>c</sup> Physics Department, Faculty of Science Demietta, Mansoura University, New Demietta, Egypt

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## Geometrical and Optical Properties of Irregular Fibers as a Function of Draw Ratio

**A. A. Hamza**

British University, El Sherouk City, Egypt

**T. Z. N. Sokkar**

**M. A. EL-Bakary**

**A. M. Ali**

Physics Department, Faculty of Science, Mansoura University,  
Mansoura, Egypt

**M. A. El-Morsy**

Physics Department, Faculty of Science Demietta, Mansoura University,  
New Demietta, Egypt

*The Pluta polarizing interference microscope with a fiber rotator-mechanical device was used to detect the variation in the cross-sectional area, shapes and optical properties of irregular fibers during the cold drawing process. The rotation method was used to overcome the difficulty of measuring the transverse cross-sectional area of fibers with irregular shapes during their drawing process. Acrylic fibers of two denier were used during this investigation. Some optical parameters were measured, such as refractive indices and birefringence. The measurements of the refractive indices show that acrylic fibers have a negative birefringence. The polarizability-per-unit volume was used to clarify the negative birefringence of these fibers. Microinterferograms and the digitized images with their contour lines are given for illustration.*

**Keywords:** fibers, interference microscopy, polyacrylonitrile

## INTRODUCTION

Synthetic polymer fibers play an important role in the textile industry. Most textiles, even those made from natural fibers, are mixed with

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Address correspondence to T. Z. N. Sokkar, Physics Department, Faculty of Science, Mansoura University, Mansoura 35516, Egypt. E-mail: afaf12004@yahoo.com

synthetic yarns drawn from polymer melts. Drawing is an important operation that improves the textile characteristics of man-made fibers. Undrawn synthetic fibers are almost isotropic in their physical properties. When the fibers are mechanically drawn, these fibers become stronger, more birefringent and highly anisotropic [1].

The last three decades have witnessed phenomenal growth in the field of material sciences. Many interferometric methods [2–7] have been used to investigate the optical and geometrical properties of fibers. They usually produce contour maps in terms of fringe pattern. The power of the interferometric method changed greatly with the introduction of the automated method of detection and analysis of the fringe patterns [8–10]. Hamza et al. [11] used the rotator device attached to a Pluta polarizing interference microscope to determine the optical and geometrical properties of fibers having regular and/or irregular transverse sections. They introduced and modified a manipulation device [12,13] to detect the variation of the cross-sectional area, shape and optical properties of regular fibers due to the drawing process. Hamza et al. [14] used the double immersion microscopy method to determine the optical and geometrical properties of fibers with irregular transverse sections.

Acrylonitrile is polymerized by a free radical mechanism and the resulting polymer is mostly atactic [15]. However, some degree of order is present in the polymer due to the large magnitude of the dipole moment of the nitrile groups. This leads to strong intrachain and interchain interactions through secondary bonding. All commercial acrylic fibers are spun from acrylonitrile polymers containing 1–15 wt% comonomer [16]. The majority of textile fibers have a morphology that can be described by the classical two-phase model [17–19]. In this model, discrete crystalline domains of the order of several hundred angstroms (Å) are mixed with amorphous domains of similar or smaller size. A high degree of crystallinity and high orientation of the crystalline molecular segments impart high tensile strength and modulus to the fibers. The amorphous phase gives rise to toughness and dyeability. In contrast to the crystalline fraction, a low degree of orientation of the amorphous phase is preferred, in order to minimize shrinkage caused by stress relaxation upon heating the material above its glass transition temperature. Describing polyacrylonitrile by the classical model is debatable.

One of the biggest problems facing anyone dealing with the determination of the optical properties of fibers is the irregularity in their cross-sectional area. To overcome this problem, the rotation method has been used [11]. In this article, the opto-mechanical and the geometrical properties of acrylic fibers having irregular transverse sections will be investigated during the drawing process using the

modified fiber rotator device [13] attached to the Pluta [20] polarizing interference microscope. This study will use image analysis of the obtained microinterferograms. The refractive indices, the birefringence, the transverse sectional area, shape, and the polarizability-per-unit volume of these fibers will be determined at different draw ratios. Moreover the polarizability-per-unit volume will be investigated to clarify the negative birefringence of acrylic fibers.

## REFRACTIVE INDEX AND POLARIZABILITY-PER-UNIT VOLUME

When a homogenous fiber is immersed in a liquid with refractive index  $n_L$  and is examined using two-beam polarizing interference microscope, the refractive index of the fiber  $n$  given by the following formula [6,7]:

$$n = n_L + \frac{F\lambda}{bA} \quad (1)$$

The above equation can be used for light vibrating parallel and perpendicular to the fiber axis.  $F$  is the enclosed area under the fringe shift that represents the optical path difference integrated across the fiber to overcome any irregularity in the fringe shift.  $\lambda$  is the wavelength of the monochromatic light used.  $A$  is the transverse sectional area and  $b$  is the interfringe spacing.

The polarizability-per-unit volume  $\varphi$  of the fiber material is calculated from the refractive index values using the Lorentz-Lorenze expression [21]

$$\varphi = \frac{3 n^2 - 1}{4\pi n^2 + 2} \quad (2)$$

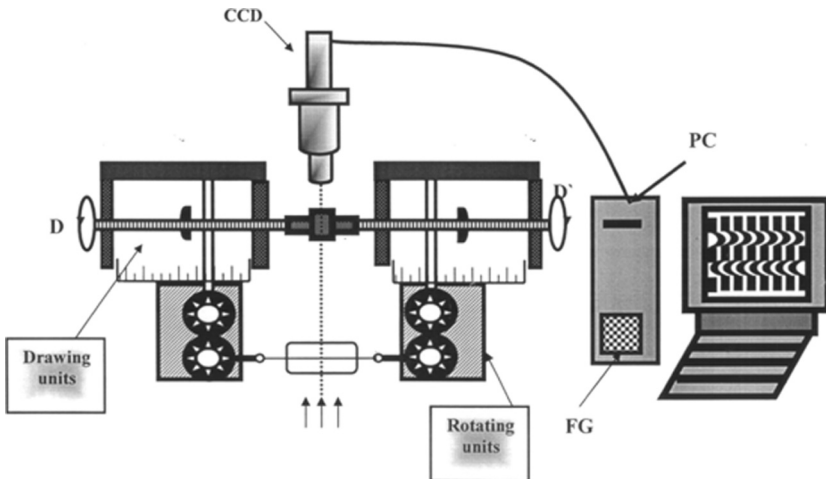
where  $n$  is the appropriate ( $n^{\parallel}$  or  $n^{\perp}$ ) refractive index.

## EXPERIMENTAL SETUP AND TECHNIQUES

The Pluta polarizing interference microscope [20] is a suitable and accurate device in the field of fiber research. The Fourier transform method of fringe analysis was used to enhance the fringe pattern and remove the noise and then extract the contour line of the fringes [22]. The resulting contour line gives qualitative and quantitative information about the optical and structural properties of the fiber under investigation. The Pluta polarizing interference microscope has been used to determine the refractive indices and birefringence of man-made fibers [3,4,7,23].

Previously Hamza et al. [13] modified a rotator device to be used for mechanical drawing of fibers with the rotating process. The modified rotator-mechanical device is attached to Pluta microscope [24] to determine the cross-sectional area and shape of the filament under investigation for different draw ratios, interferometrically, without using other techniques. This device is shown in Figure 1, which consists of the basic drawing-rotating units, Pluta interference system and computerized units.

This experimental setup is used to determine the optical and geometrical properties of acrylic fibers during the cold-drawing process. Firstly, the fiber ends are fixed between the two clamps using an adhesive material. An immersion liquid with refractive index  $n_L$  is used, and the fiber is free to move with the movement of the two clamps. The fiber rotator-mechanical device is transferred to the stage of the Pluta microscope which is adjusted in the crossed position to produce two separated images of the fiber [4,20]. The obtained fiber image is captured using CCD camera and the computerized unit. This image is digitized directly via the digitizer frame grabber. The digitized image is recorded on the computer storage medium. From the obtained duplicated image, the fiber thickness is measured by recording the average thickness of the two images for light vibrating parallel and perpendicular to the fiber axis.



**FIGURE 1** Systematic diagram of the opto-mechanical system used where (D,D') are the two threaded ends [10], CCD is the camera, PC is a computer unit and FG is a frame grabber.

The images obtained by varying the angle of rotation from 0 to 360° are used to map the cross-sectional shape of the fibers under test. Hence the mean cross-sectional area ( $A$ ) is calculated. Using the two threaded ends ( $D$  and  $D'$ ), the fiber is drawn from the two ends. Rotating the fiber in steps each of 10° and recording the image at each angle from the fiber thickness profile, the transverse sectional area is determined for this draw ratio. By repeating the process of drawing and mapping the thickness variation with the angle of rotation, the variation of cross-sectional area and shape due to the cold-drawing process is detected.

The obtained microinterferograms are used with Equation (1) to calculate the mean area enclosed under the fringe shift ( $F$ ) and the mean refractive index ( $n$ ) of the fiber.

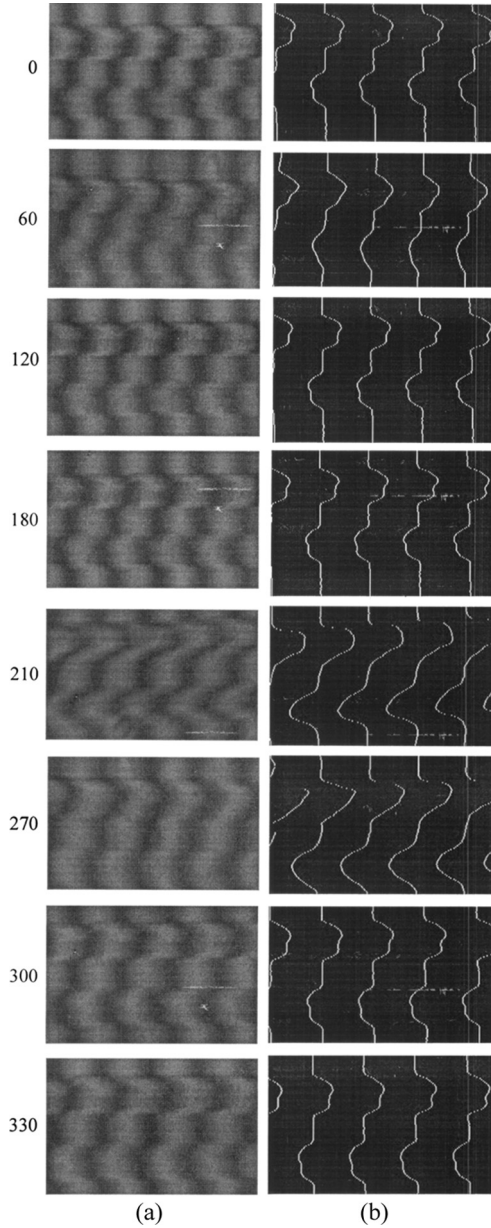
## RESULTS AND DISCUSSION

### Automated Measurement of the Variation of the Transverse Sectional Shape, Area and Refractive Indices due to the Drawing Process using Rotator Mechanical Device

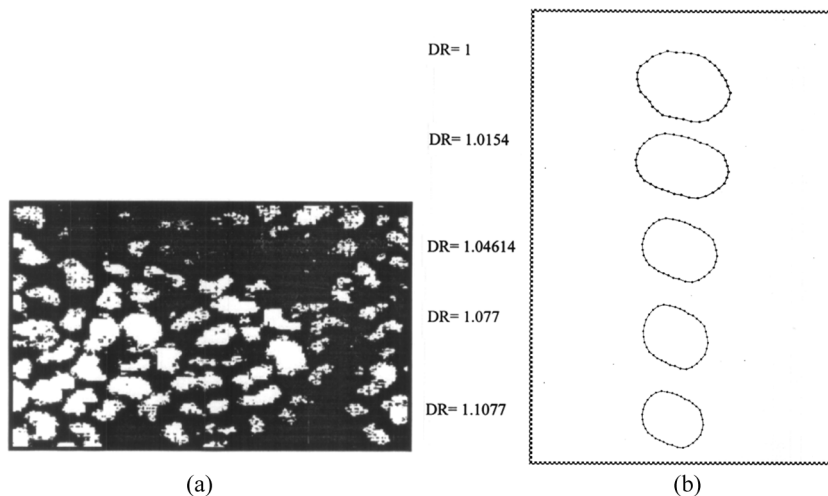
The mechanical-rotator device attached with the Pluta automated polarizing interference microscope allows the determination of the mean refractive indices, the transverse sectional area and shape for the same location during the drawing process.

A filament of an acrylic fiber is fixed between the two clamps of the device. The fiber is immersed in a drop of liquid with refractive index 1.5005 at  $T = 30^\circ\text{C}$ . The rotator mechanical device is transferred to the microscope stage. The wavelength of the monochromatic light used is 546.1 nm. Duplicated images of acrylic fibers are obtained and captured using the CCD camera. The original images are threshold, enhanced and converted into binary image to identify the contour lines, which are analyzed via software program for the refractive index measurements of the fiber [10]. Mapping the variation of the fiber thickness with the angle of rotation, the shape of the transverse section of the fiber is obtained and hence the transverse sectional area of the fiber is calculated.

Figure 2a shows the microinterferograms of the duplicated images of an acrylic fiber at different angles of rotation ranging from 0 to 360° at draw ratio  $DR = 1$ . Figure 2b shows their automatic contour lines. Figure 3a shows the shape of the transverse sections of acrylic fibers using high power optical microscopy. Figure 3b shows the graphical presentation of the transverse sectional shapes of one of these fibers at different draw ratios during the drawing process



**FIGURE 2** (a) Original microinterferograms of an acrylic fiber. (b) Automatic contour line of experimental microinterferograms for duplicated images of this acrylic fiber at different angles of rotation ranged from (0 to 360°) at draw ratio  $DR = 1$ .

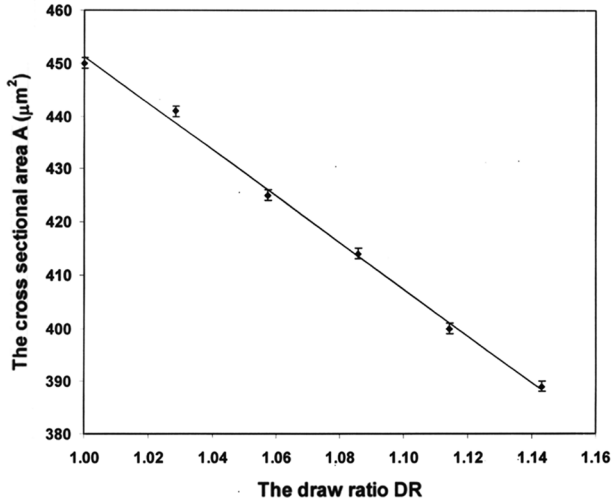


**FIGURE 3** (a) The shape of the cross-sections of acrylic fibers using high power optical microscopy. (b) A graphical presentation of the transverse sectional shapes of one of these fibers at different draw ratios during the cold drawing process.

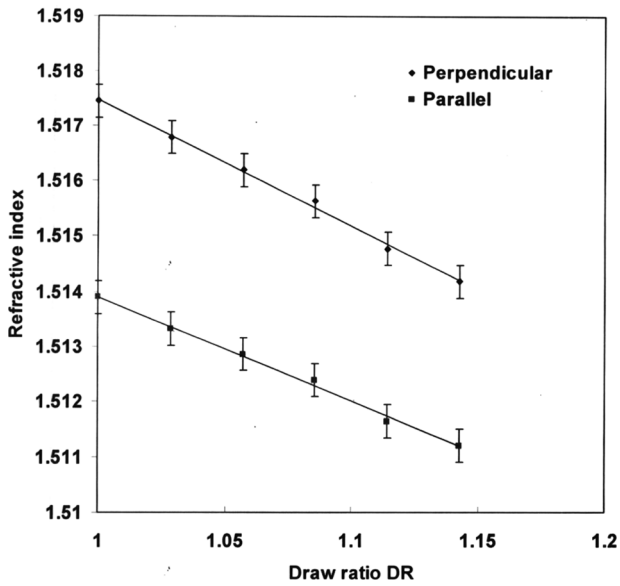
using rotation method. It is clear that the cross-sectional area decreases gradually with increasing draw ratio without significant variation in the cross-section shape. Figure 4 shows the relationship between the cross-sectional area of the fibers and their draw ratios. Using the microinterferograms illustrated in Figure 2 and the calculated cross-section area from Equation (1), the mean refractive indices  $n^{\parallel}$  and  $n^{\perp}$  were calculated for each draw ratio. Figures 5 and 6 show the variation of the mean refractive indices  $n^{\parallel}$  and  $n^{\perp}$  and the birefringence of these fibers with their draw ratios, respectively.

It is observed that the refractive indices decrease with increasing draw ratio as shown in Figure 5 and the birefringence increases with increasing draw ratio as in Figure 6. For each draw ratio we noticed that the ratio of decreases in the refractive index  $n^{\parallel}$  for light vibrating parallel to the fiber axis is greater than the refractive index  $n^{\perp}$  for light vibrating perpendicular to the fiber axis. This indicates that the molecules constituting the fiber are oriented in the perpendicular direction more than the parallel direction during the cold drawing process of these fibers. Thus the birefringence ( $n^{\parallel} - n^{\perp}$ ) has always a negative sign and increases with increasing the draw ratio.

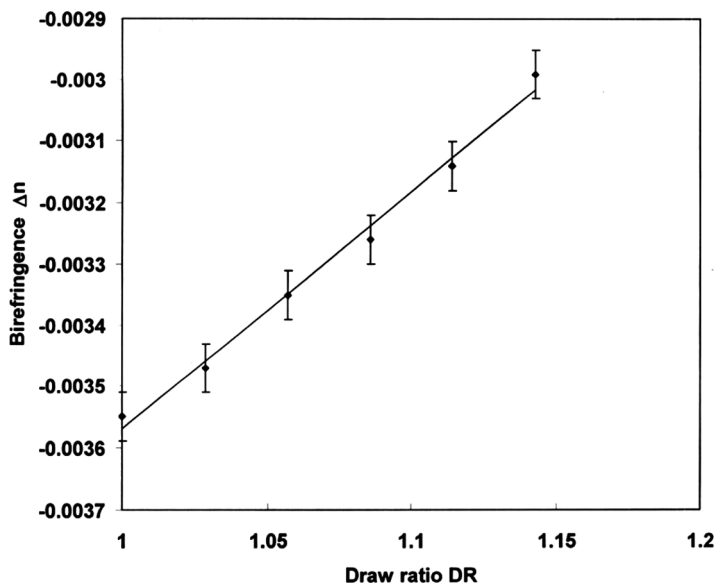




**FIGURE 4** The variation of the cross-sectional area with the draw ratio during the cold drawing process of acrylic fibers.



**FIGURE 5** The variation of the mean refractive indices  $n^{\parallel}$  and  $n^{\perp}$  with the draw ratio of acrylic fibers.

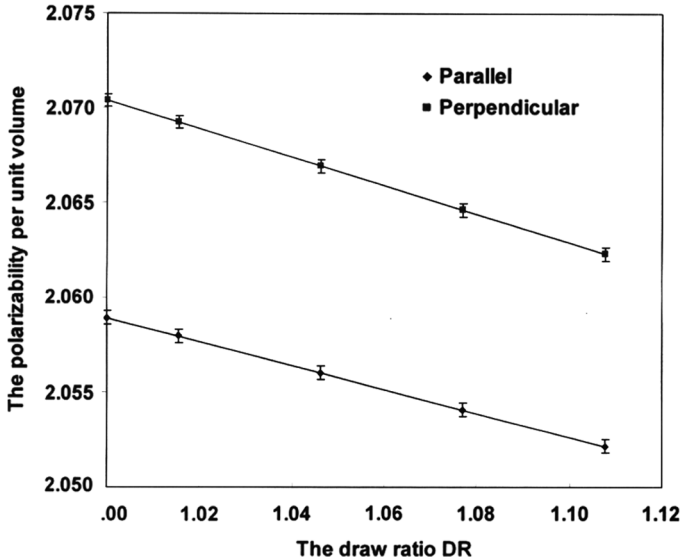


**FIGURE 6** The variation of the birefringence with the draw ratio of acrylic fibers.

### Measurement of Polarizability-per-Unit Volume

The polarizability-per-unit volume for light vibrating parallel and perpendicular to the fiber axis of acrylic fibers were calculated using Equation (2). Figure 7 shows the variation of the polarizability-per-unit volume with the draw ratio for light vibrating parallel and perpendicular to the fiber axis. It shows that the polarizability-per-unit volume decreases with increasing draw ratio. The decrease in the polarizability-per-unit volume for light vibrating parallel to the axis is greater than the decrease in the polarizability-per-unit volume for light vibrating perpendicular to the fiber axis, i.e.  $\varphi^{\perp} > \varphi^{\parallel}$ . This indicates that the molecules constituting each fiber are oriented (with respect to the fiber axis) in the perpendicular direction more than in the parallel direction during the drawing process. Thus the birefringence always has a negative sign and decreases with increasing draw ratio.

Most synthetic fibers exhibit positive birefringent properties ( $n^{\parallel} > n^{\perp}$ ). However, acrylic fibers have a negative birefringence due to the presence of cyanide groups ( $-\text{C}\equiv\text{N}$ ). This could be argued on the basis of bond polarizability in polyacrylonitrile fibers or the polarizability-per-unit volume [25]. Using the values given by Denbigh [26]



**FIGURE 7** The variation of the polarizability-per-unit volume of the fiber material with the draw ratio for light vibrating parallel and perpendicular to the fiber axis, respectively.

for undrawn acrylic fiber,  $\alpha^{\parallel}$  and  $\alpha^{\perp}$  are found to be  $65.8 \times 10^{-31} \text{ m}^3$  and  $67.9 \times 10^{-31} \text{ m}^3$ , respectively where  $\alpha^{\parallel}$  and  $\alpha^{\perp}$  are the polarizability along and across the chain axis, respectively. This leads to  $\mathbf{n}^{\perp} > \mathbf{n}^{\parallel}$  so the birefringence has negative value. Due to the drawing process of acrylic fibers, a relatively small value of  $\Delta n$  is to be expected, because the highly polarizable CN groups are oriented nearly perpendicular to the backbone direction, so that the side groups and the backbone nearly compensate each another [27,28].

The accuracy of the measurements depends on the accuracy of the Pluta polarizing interference microscope [20]. The accuracy of the Pluta microscope in the thickness and the length of the fringe shift measurements is  $0.05 \lambda$ , where  $\lambda$  is the wavelength of the monochromatic light used. Therefore, the error in the refractive indices and birefringence measurements is  $\pm 0.003$  [20].

## CONCLUSION

The rotator-mechanical device attached to the Pluta polarizing interference microscope is a very promising technique for studying

the opto-mechanical and geometrical properties, for the same location, of irregular fibers. The rotation method is used to overcome the difficulty of measuring the transverse sectional shape and area of fibers with irregular cross-sections. It permits determination of the variation of the cross-sectional area and shape for each draw ratio.

From the measurements carried out, it follows that:

1. The cross-sectional shape of the tested filament doesn't change with the drawing process but the cross-sectional area decreases due to the drawing process.
2. The polarizability-per-unit volume clarifies that the birefringence of acrylic fibers always has a negative sign with the drawing process.

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