

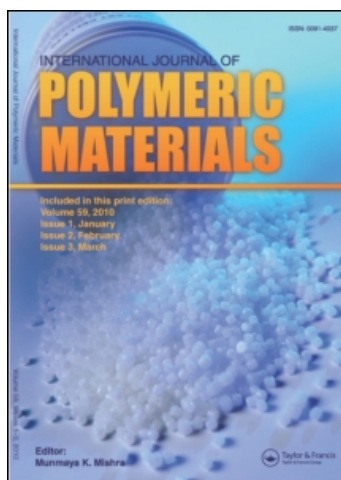
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### Characterization of Highly Oriented Polymeric Fibers Using Modified Optical Fourier Transform Technique

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## Characterization of Highly Oriented Polymeric Fibers Using Modified Optical Fourier Transform Technique

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*This article demonstrates a modified variable wavelength interferometry optical Fourier transform technique applied to highly oriented polymeric fibers. It enables a simultaneous determination of the directional refractive indices and birefringence of fiber, which is a significant progress in comparison to the early discovery of the exit pupil pattern. This technique is based on the analysis of the diffraction pattern in the back focal plane of the polarizing microscope objective. A comparison between this modified technique and another conventional one (variable wavelength interferometry fringe field technique) has been performed.*

**Keywords:** automatic fringe analysis, birefringent polymeric fibers, directional refractive indices, optical Fourier transform, spectral dispersion, variable wavelength interferometry

### ABBREVIATIONS

|          |  |
|----------|--|
| PI:      | Polarizing Interference                                      |
| FFI:     | Fringe Field Interference                                    |
| OFT:     | Optical Fourier Transform                                    |
| VAWIFFI: | Variable Wavelength Interferometry Fringe Field Interference |

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|          |  |
|----------|--|
| VAWIOFT: | Variable Wavelength Interferometry Optical Fourier Transform |
| CCD:     | Couple Charge Device   |
| LCF:     | Liquid Crystal Filter  |

## INTRODUCTION

For textile fibers researchers and technologists, it is important to know the birefringence and directional refractive indices of the fibers. These quantities provide information about the fiber structural homogeneity and molecular orientation. Knowledge of these properties is especially important for evaluating the effect of any chemical or mechanical treatments of polymeric fibers [1].

Fringe field (FF) and optical Fourier transform (OFT) interferometries are a family of nondestructive testing techniques applied in the field of textile fiber research and technology. The Fourier transform is largely used for the description and processing of images, and is especially useful for the representation of microscopic imaging. This representation is complicated; therefore, many scientists described the microscopic imaging mathematically using Fourier transform [2,3]. The optical Fourier transform technique is applied to analyze birefringent properties and possible structural and geometrical defects of textile fibers [4]. The variable wavelength interferometry optical Fourier transform (VAWIOFT) technique based on Biolar polarizing interference (PI) system has been recognized to be a useful tool for the measurement of these optical properties of fibers [4,5].

The diffraction patterns observed in the exit pupil of Biolar polarizing interference (PI) microscope were analyzed and described mathematically by Pluta [5]. He reported that a cylindrical birefringent fiber can produce a specific interference pattern in the back focal plane of the microscope objective outfitted with a subcondenser slit diaphragm. This occurs when the birefringent fiber is oriented diagonally between two crossed polarizers and is trans-illuminated by monochromatic light. These patterns behave as the optical Fourier transforms and consist of circular or annular dark interference fringes. Monochromatic light of continuously variable wavelength was used to change the intensity of the interference pattern center from maximally dark to bright patches, and so on. Then the spectral dispersion of the textile fiber birefringence can be determined. This task was performed manually and faced many problems discussed elsewhere [6].

A mathematical model of the output pattern and new procedure based on variable wavelength interferometry optical Fourier

transform (VAWIOFT) have been applied to measure the spectral dispersion of the textile fiber birefringence [6]. This technique is based on a relation between the radii of the annular dark fringes and the wavelength for each sequence of interference patterns. This technique has been modified and extended to be suitable for both single- and multi-fringe techniques and increase the measurement accuracy of the birefringence of the weakly, moderately and highly oriented polymeric fibers [7–9]. Those tasks have been performed automatically and created potential industrial applications. Also, the VAWIOFT technique is suitable for quick and precise detection of optical inhomogeneities and/or geometrical micro-irregularities of fibers. The only disadvantage of the above techniques is that it is not possible to measure separately the spectral dispersion of directional refractive indices  $n_{\parallel}(\lambda)$  and  $n_{\perp}(\lambda)$ .

A modified mathematical model based on VAWIOFT has been derived [10]. This modified technique enables simultaneous calculations of three basic fiber parameters (birefringence, refractive indices), which is significant progress in comparison to the previous OFT techniques.

In the present paper, this modified variable wavelength interferometry optical Fourier transform (MVAWIOFT) technique is experimentally applied for determination of the optical parameters of highly oriented polymeric textile fibers. The conventional variable wavelength interferometry fringe field (VAWIFFI) technique is applied to determine these optical parameters for the sake of comparison.

## OPTICAL FOURIER TRANSFORMS OF BIREFRINGENT FIBER

The original variable wavelength optical Fourier transform technique was discovered by Pluta [4,5]. A birefringent polymeric fiber oriented diagonally between crossed polars acts as a specific bifocal cylindrical lens, whose focal lines  $L_{\perp}$  and  $L_{\parallel}$  contain light vibrations perpendicular ( $\perp$ ) and parallel ( $\parallel$ ) to the fiber axis. These focal lines act as two light slits, one of which follows the other. They are mutually coherent across their widths but incoherent along their lengths. Each pair of coherent points produces spherical wavefronts whose radii of curvature are slightly different. These wavefronts can interfere with each other and produce a specific interference pattern with annular fringes observed in the Fourier plane of the microscope objective. The diameter of annular dark fringe depends on the wavelength of light used. This interference pattern is invariable when the fiber under investigation is transversely or vertically translated.

## Measurement of Fiber Birefringence

### **Dark and Bright Patches Technique (Pluta's Method) [5]**

In this conventional technique, the intensity analysis method is applied. The intensity of the annular dark fringe center can be consecutively changed from maximally bright to dark when the wavelength of the continuous monochromatic light varied from short to long region of visible spectrum or vice versa (dark or bright patches depending on the wavelength). The spectral dispersion of the polymeric fiber birefringence  $B_s(\lambda)$  can be determined using the following equation:

$$B_s(\lambda) = \frac{(m_1 + q_s)\lambda_s}{t} \quad (1)$$

where  $m_1$  is the initial interference order ( $m_1 = q_s \frac{\lambda_s}{\lambda_1 - \lambda_s}$ ),  $t$  is the diameter of the polymeric fiber,  $B$  is the birefringence ( $B = n_{\parallel} - n_{\perp}$ ),  $\lambda$  is the wavelength,  $q$  is the interference order difference and  $s = 2, 3, 4$ , and soon.

Sometimes the OFT pattern is noised at its center. Therefore, the measuring accuracy of the initial interference order in the image of the birefringent is decreased. This is the disadvantage of this technique.

### **Single-Fringe Technique [7]**

An extrapolation method based on the single-fringe variable wavelength interferometry optical Fourier transform (single-fringe VAWIOFT) is used to increase the accuracy of measurement of the spectral dispersion of the fiber birefringence. This method has been used for initial interference order measurement when the number of the interference sequences is less than 2 (weak birefringent polymeric fibers) over the range of the visible spectrum. Also, this single-fringe VAWIOFT technique is used to avoid the automatic analysis of the light intensity at the center of the OFT pattern because sometimes these patterns are noised at this center. The spectral dispersion of the polymeric fiber birefringence  $B_s(\lambda)$  can be determined using the following equation:

$$B_s(\lambda) = \frac{(m_1 + i + p_{is})\lambda_s - F_s r_{is}^2}{t} \quad (2)$$

where  $m_1 = (i + P_{is})\lambda_s + (F_1 r_{01}^2 - F_s r_{is}^2) / \lambda_1 - \lambda_s$ ,  $r_{01}$  and  $r_{is}$  are the radii of the annular dark fringe for given wavelengths  $\lambda_1$  and  $\lambda_s$ , respectively,  $i$  is the integer increment or decrement of the interference order

with respect to  $m_1$  at the center,  $(m_1 + i)$  is the current interference order at the center of the optical Fourier Transform (OFT) interference pattern, and  $p_{is}$  is the digital increment or decrement of the current interference order at the edge of the OFT interference pattern with respect to  $(m_1 + i) \cdot F_s = (\Delta f / 2f_{ob}^2)_{\lambda_s}$ , where  $f_{ob}$  is the microscope objective focal length and  $\Delta f = f_{\perp} - f_{\parallel}$ ;  $f_{\perp}$  and  $f_{\parallel}$  are the ordinary and extraordinary focal lengths of the fiber, respectively.

### **Multiple-Fringe Technique [9]**

Adaptive interferometric method based on the multiple-fringe variable wavelength interferometry optical Fourier transform (multiple-fringe VAWIOFT) is introduced. This method is applied for measuring the spectral dispersion of the highly oriented fiber birefringence  $B_s(\lambda)$  using the following equation:

$$B_s(\lambda) = \frac{(m_1 + i + \kappa_s)\lambda_s(1 - \sigma_s) - \sigma_s q_{n-1}\lambda_s}{(1 - \sigma_s)} \quad (3)$$

where  $s = 1, 2, 3, \dots$ ,  $\kappa_s = (r_n)_{\lambda_s} - (r_n)_{\lambda_1} / 2(r_n)_{\lambda_s}$ ,  $\sigma_s = (r_1/r_n)_{\lambda_s}^2$ ,  $q_{n-1}$  is the integer increment or decrement of the interference order at  $r_n$  with respect to  $r_1$ , and  $i$  is the integer increment or decrement of the current interference order with respect to  $m_1$ .

Using the above interferometric techniques, the initial interference order ( $m_1$ ) in the image of the object under investigation can be determined. The measuring accuracy of the optical path difference is increased when the single- and multiple-fringe VAWIOFT techniques are applied [7,9]. Having determined the initial interference order the spectral dispersion curve of the object birefringence is calculated. It should be noticed that the above VAWIOFT techniques could not determine separately the directional refractive indices of the birefringent object. This disadvantage is overcome as follows.

### **Measurement of Directional Refractive Indices of Highly Oriented Birefringent Fiber**

It is worth noting that the measurement process using the optical Fourier transform technique is suitable for a fully automatic operation. Progress was made when it became possible to measure the diameters of annular dark fringes applying image processing technique, which extended the measuring range. The most promising situation occurs when at least two annular dark fringes are visible simultaneously and their radii can be measured at the same wavelength

( $\lambda_s$ ). The formulas for the radii  $r_{s1}$  and  $r_{s2}$  take the form:

$$r_{s1}^2 = 2 \frac{f_{Ob}^2}{\Delta f_s} [t \cdot B_s - m_1 \lambda_s]$$

and

$$r_{s2}^2 = 2 \frac{f_{Ob}^2}{\Delta f_s} [t \cdot B_s - (m_1 + q_s) \lambda_s] \quad (4)$$

where,  $\Delta f_s = f_{s\perp} - f_{s\parallel}$  is the difference between the focal lengths of the fiber.

The spectral dispersion of the highly oriented fiber birefringence  $B_s(\lambda)$  can be calculated using the following equation:

$$B_s(\lambda) = \frac{\lambda_s}{t} \left[ m_1 + \frac{R_s}{R_s - 1} \right] \quad (5)$$

where,  $R_s = r_{s12}^2 / r_{s2}^2$ .

After some simple manipulations with the above formulas (4 and 5), it is possible to calculate the directional refractive indices according to the following quadratic formulas [10]:

$$n_{s\perp}^2 + (B_s - 2)n_{s\perp} + 1 - B_s \left( 1 + \frac{t}{4\Delta f_s} \right) = 0 \quad (6)$$

and

$$n_{s\parallel}^2 + (B_s + 2)n_{s\parallel} + 1 + B_s \left( 1 + \frac{t}{4\Delta f_s} \right) = 0 \quad (7)$$

The variable wavelength interferometry fringe field (VAWIFFI) technique was used for the determination of the birefringence and the refractive indices of the fibers. The initial interference order in the fiber image and the fiber birefringence can be calculated using Eq. (1). The fiber refractive indices for polarized light vibrating parallel and perpendicular to the fiber axis are determined using the equation [11]:

$$(n_{sj} - n_{Ls})t = (m_{1j} + q_s)\lambda_s, \quad j \begin{cases} \parallel & \text{for incident light vibrating parallel} \\ & \text{to fiber axis} \\ \perp & \text{for incident light vibrating} \\ & \text{perpendicular to fiber axis} \end{cases} \quad (8)$$

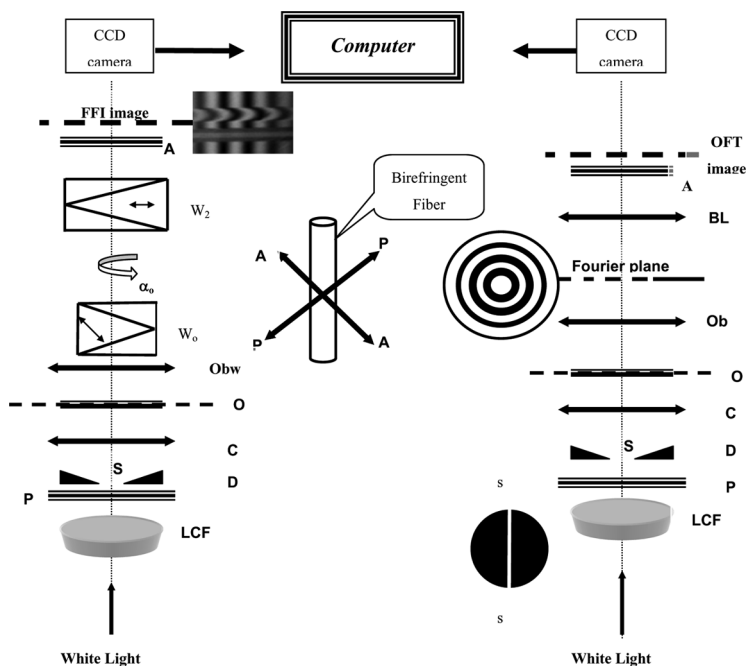
where  $n_{Ls}$  is the refractive index of the surrounding medium of the fiber.

In the present paper, the spectral dispersion curves of birefringence and directional refractive indices of some highly oriented textile fibers have been determined for comparison purposes using the variable wavelength fringe field interference (VAWFFI) and modified VAWIOFT techniques.

## EXPERIMENTAL DETAILS

### Optical Techniques Setup

Experiments described in this paper have been performed with the aid of a commercially available Biolar polarizing interference (PI) microscope equipped with both optical Fourier transform and fringe field interference heads. The setup of the optical system of a typical PI polarized microscope is shown in Figure 1. Two configurations are



**FIGURE 1** Schematic diagram of the automatic computer-aided variable-wavelength systems used for observation and processing of a) the FFI patterns, and b) the optical Fourier transform of the birefringent fibers, where, P-polarizer, D-slit diaphragm, C-condenser, Obw-Wallston objective lens,  $W_0$ ,  $W_2$ -birefringent prisms, A-analyzer; LCF-Lyot tunable birefringent filter, Ob-normal objective lens, O-objective plane, and BL-Bertrand lens.



considered: Figures 1a and b concern orthoscopic (VAWIFFI technique) and conoscopic (VAWIOFT technique) observations. In both cases, the polymeric fiber under study is illuminated according to the Köhler principle.

For the conoscopic observations, the Bertrand lens is used together with the ocular. The polarizer  $P$  and analyzer  $A$  are crossed and their directions of light vibrations make an angle of  $45^\circ$  with the axis of the fiber. For observation of OFT pattern the subcondenser slit  $S$  is oriented parallel to the fiber axis. In case of observation of nonduplicated and totally duplicated FFI pattern, the slit  $S$  is oriented to the fiber axis by an angle  $90^\circ$  and  $45^\circ$ , respectively. The OFT and FFI patterns of the output field of the microscope are captured by a CCD camera for further automatic processing and analysis using a digital image processing technique.

The Bertrand lens is normally used together with the ocular for observation of conoscopic images of birefringent objects. It transfers the exit pupil of the microscope objective into the primary image plane where the field diaphragm of the ocular is placed. The objective prism does not influence the character of the interference observed, but the final image depends on the respective positions of both prisms used. The wavelength is changed using a digital tunable liquid crystal filter (LCF) based on the Lyot principle (produced by CRI).

## Description of Digital Image Processing Technique

Using special software (Radial Vector Tracing Program), the interference pattern is analyzed automatically to measure the radii of the annular dark fringes as a function of the light wavelength. At the initial stage of the measurement, the OFT pattern is adjusted so that the displacement of the fringe center with respect to the frame center is smaller than the radius of the fringe ( $r$ ). The initial radius of the fringe should be as large as possible on the condition that it does not touch the edge of the exit pupil of the microscope objective. These manipulations at the beginning of the measurements speed up the following process of automatic operations.

Sometimes the annular dark fringe is deformed and takes an elliptical shape due to the fact that both the condenser slit and the object axis are not parallel, or the birefringent object suffers from local optical inhomogeneities and geometrical irregularities [6]. In this case, the ellipse fitting is applied for the pattern and then the ellipse parameters are calculated. The above procedures, except the initial stage, must be applied each time when the wavelength is changed using a filter or the fiber under study is translated along its axis.

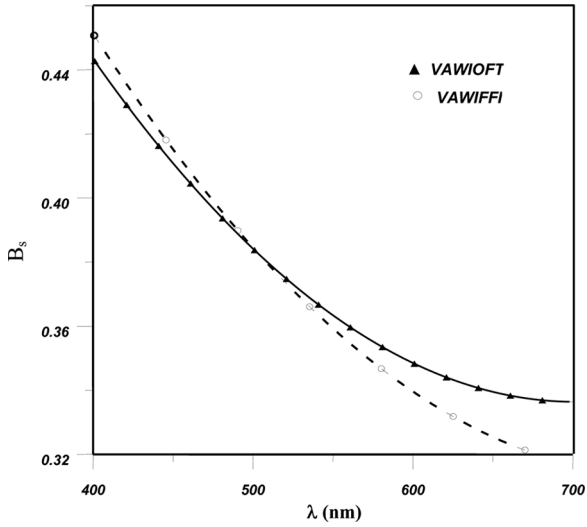
A special program is prepared to determine the initial and current interference orders in the image of the object under study. Finally, the spectral dispersion curves of the optical parameters of the highly oriented objects are calculated.

## RESULTS AND DISCUSSION

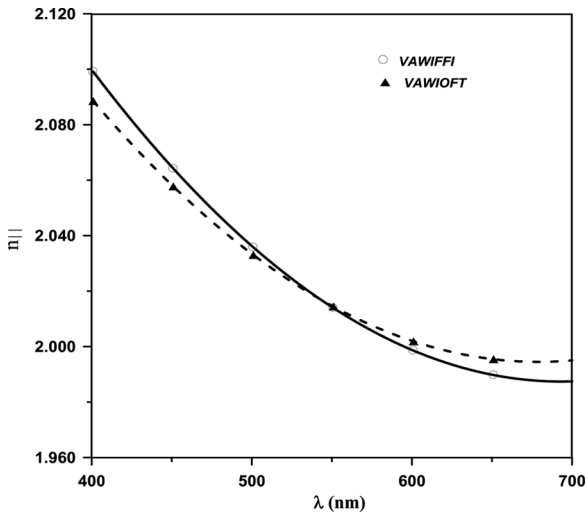
The polarizing microscope is configured for observing the optical Fourier transform patterns. These patterns of the output field of the microscope are captured by CCD camera for automatic processing analysis by the computer-aided system. This diffraction pattern consists of two annular dark fringes and is simultaneously visible in the exit pupil of the objective (objective magnification/numerical aperture:  $40\times/0.65$ ). Starting from the long wavelength region of the Lyot filter and passing towards the short wavelength region, a sequence of the OFT pattern and its annular dark fringes of consecutive orders can be observed. The radius of the annular dark fringe increases with decreasing wavelength and finally the annular fringe reaches the edge of the objective exit pupil. Subsequent decreasing of the wavelength repeats the above sequence of OFT patterns. The number of these sequences (i) depends on the polymeric textile Matrix-2 fiber birefringence.

The radii of two neighboring annular dark fringes of each OFT pattern have been precisely measured for given wavelengths and then the initial ( $m_1$ ) and current interference orders ( $m_s = m_1 + q_s$ ) may be calculated. To increase the accuracy of the measurement of the radius of the annular dark fringe, the process of tracing is repeated 16 times. Having current interference orders and using the modified VAWIOFT technique (Eqs. (5–7)) the spectral dispersion curves of both birefringence  $B_s(\lambda)$  and directional refractive indices ( $n_{||}(\lambda)$  and  $n_{\perp}(\lambda)$ ) of the polymeric textile Matrix-2 fiber are determined as shown in Figures 2–4. The advantage of the modified technique is that the evaluation is simple and does not involve any immersion liquids, as is usually necessary in the conventional microinterferometry of highly birefringent textile fibers. Also, this technique visualizes any irregularities in the fiber structure in terms of distortion of the annular fringes.

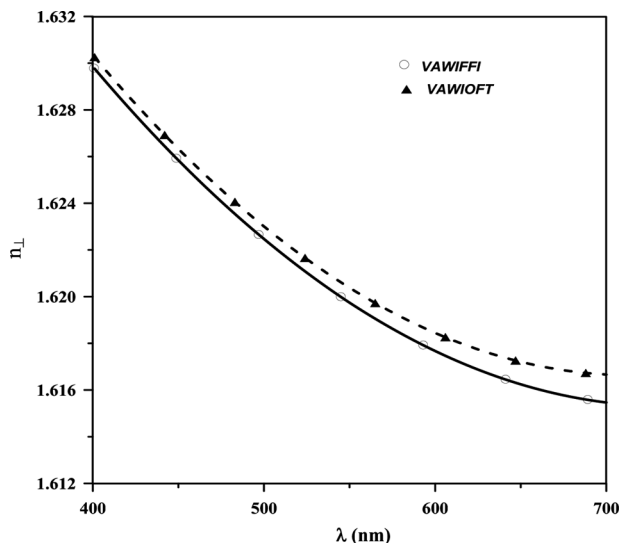
In order to confirm the illustration of the current modified VAWIOFT technique, the measurements have been performed using the well-known conventional variable wavelength interferometry fringe field (VAWIFFI) technique. The polarizing microscope was reconfigured for observing the fringe field interference pattern of the Matrix-2 fiber. The fiber was immersed in a suitable liquid of refractive index ( $n_L = 1.60427$ ) ( $n_L < n_{\perp} < n_{||}$ ) at temperature  $28.5^{\circ}\text{C}$  and



**FIGURE 2** The spectral dispersion of Matrix-2 fiber birefringence by use of the modified VAWIOFT (solid line) and the conventional VAWIFFI (dashed line) techniques.



**FIGURE 3** The spectral dispersion of the refractive index of Matrix-2 fiber ( $n_{||}$ ) when the incident light vibrating parallel to the fiber axis by use of the modified VAWIOFT (dashed line) and the conventional VAWIFFI (solid line) techniques.

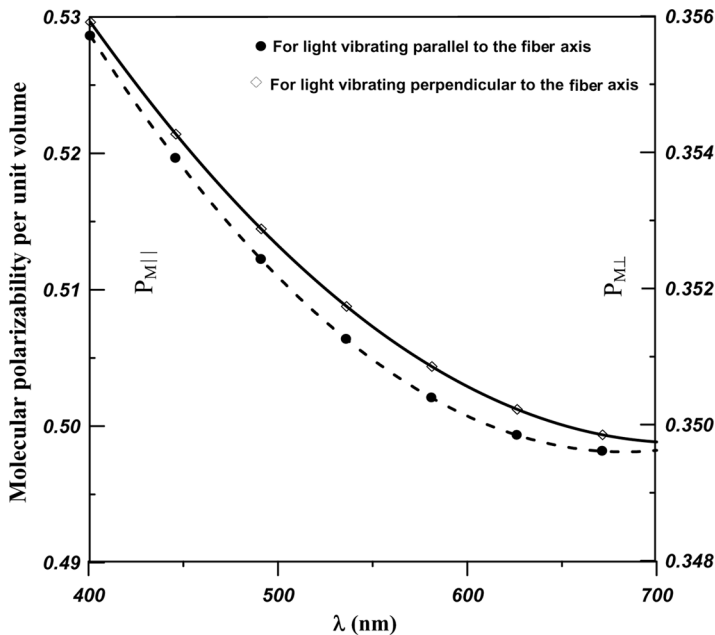


**FIGURE 4** The spectral dispersion of the refractive index of Matrix-2 fiber ( $n_{\perp}$ ) when the incident light vibrating perpendicular to the fiber axis by use of the modified VAWIOFT (dashed line) and the conventional VAWIFFI (solid line) techniques.

was illuminated by the light of wavelength 550 nm. The application of the VAWIFFI technique for characterizing of a highly oriented textile fiber enables the spectral dispersion of directional refractive indices to be determined if the optical path difference produced by the fiber is small. To achieve a satisfactory result, the highly oriented textile fiber must be immersed in a liquid whose refractive index  $n_L$  is close to that of the fiber ( $n_f$ ) over the visible spectrum. In such situation, the light intensity of the fiber image does not differ from the background intensity. Such an immersion liquid must be individually prepared because the spectral dispersion of the commercially available immersion liquids  $n_L(\lambda)$  for microscopy differ from that of the fiber  $n_f(\lambda)$  and dispersion staining appear in white light. Moreover, the surface of the fiber under study must be free from any contamination which can modify the distribution of the light intensity across the fiber image.

The automatic VAWIFFI technique was applied using a prepared software program for measurement of the spectral dispersion curves of the optical parameters of the Matrix-2 fiber. This can be done by selecting certain wavelengths, at which the coincident and anti-coincident configuration between the displaced fringes in the fiber and undisplaced fringes of the same order of the empty interference

field occurs. The spectral dispersion curves of the birefringence and directional refractive indices were determined by using the VAWIFFI technique (Eqs. (1) and (8)) as shown in Figures 2 to 4. It is seen that the spectral dispersion of optical parameters of the Matrix-2 fiber using the modified VAWIOFT (fiber surrounded by air) and fringe field (fiber surrounded by liquid) techniques slightly differ from each other over the small range of the visible spectrum (long-wavelength). Because the VAWIFFI technique faced an undesirable effect, sometimes it is necessary to change the initial interference order  $m_1$  by  $\pm 3$  (in our experiments  $m_1$  has been changed by  $-2$ ) to obtain the correct dispersion curve. In the VAWIFFI technique, the zero order fringe in the image of the object under investigation should be a chromatic fringe. In particular, the Wollaston prism in the VAWIFFI technique suffers from a spectral dispersion of birefringence that causes the zero-order fringes to be colored when they are displaced by the object at a distance larger than several interference spacing. Additionally, the VAWIFFI technique cannot detect the irregularities of the



**FIGURE 5** The spectral dispersion of molecular polarizability per unit volume of Matrix-2 fiber when the incident light vibrating parallel ( $P_{M||}$ ) (dashed line) and perpendicular ( $P_{M\perp}$ ) (solid line) to the fiber axis by use of the modified VAWIOFT technique.

geometrical shape of the fibers at the same time. However, the VAWIOFT technique overcomes these problems.

The VAWIFFI technique is affected by a small defocusing on the fiber under study, i.e., the FFI pattern changes when the fiber is transversely or vertically translated, whereas the OFT technique is not affected by small defocusing on the fiber image under study, because the OFT pattern does not change when the fiber is transversely or vertically translated. So, the accuracy of the optical path difference measurement of highly oriented polymeric fiber was estimated and found to be  $\pm 0.007\lambda$  and  $\pm 0.002\lambda$  when the VAWIFFI and modified VAWIOFT techniques are used, respectively.

By using the modified VAWIOFT technique, the structural and optical parameters of the Matrix-2 fiber could be determined. For example, the molecular polarizability  $P_M$  is related with the directional refractive indices ( $n_{||}$  and  $n_{\perp}$ ). Thus, the spectral dispersion curves of molecular polarizability per unit volume ( $P_M$ ) ( $P_{Mj} = \frac{3}{4\pi} \left( \frac{n_j^2 - 1}{n_j^2 + 2} \right)$ ) of the Matrix-2 fiber, when the light vibrating parallel ( $P_{M||}$ ) (dashed line) and perpendicular ( $P_{M\perp}$ ) (solid line) to the fiber axis, can be calculated as shown in Figure 5.

## CONCLUSIONS

- In this paper, the spectral dispersions of the directional refractive indices and birefringence of polymeric fibers have been measured using modified variable wavelength interferometry optical Fourier transform (VAWIOFT) and conventional fringe field interference (VAWIFFI) techniques.
- The modified technique depends on the relationship between the radius of the dark fringe and the wavelength. The digital image processing technique is used to measure the fringe radius when the wavelength is varied within the visible spectrum.
- Using this modified technique, the initial interference order is more precisely calculated when the OFT interference patterns contain two annular dark fringes compared with the conventional VAWIFFI.
- It is worth noting that by using the VAWIFFI technique, there is confusion with identification of interference orders in the image of the highly oriented fibers. This is due to the zero order fringe which is colored. So, the advantage of the modified VAWIOFT technique is that it avoids this confusion. Also, the VAWIOFT technique need not take a certain reference, such as the zero order fringe, and it is insensitive to small defocusing of the microscope.

- The modified variable wavelength interferometry optical Fourier transform technique is more sensitive to any optical and geometrical microdefects.
- Finally, compared with the conventional VAWIFFI technique, the mathematical model and the procedures of the modified VAWIOFT technique are relatively simple and give good results. Therefore, this modified technique is considered to have potential industrial application.

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