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Optical and structural properties of thermally treated iPP fibers: Effect of strain rate

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ABSTRACT

Two-beam polarizing interference (Pluta) microscope was used to study the effect of annealing conditions (temperature and duration) and strain rate on the physical properties of isotactic polypropylene (iPP) fibers. The percentage shrinkage of the fiber length at different annealing conditions was studied. The effect of strain rate on the birefringence and molecular orientation of stretched iPP fibers was carried out before and after annealing. The test samples of iPP fibers were treated at two annealing temperatures: 80 °C and 120 °C and four durations: 0.5, 1.0, 1.5, 2.0 and 4.0 h. Empirical formulae were suggested for correlating the fiber birefringence, the molecular orientation factor, and strain rate at three different stretching speeds; 0.38, 0.57, and 0.77 cm/s. Upon stretching at constant temperature, it is found that the stretching speed has the main effect of controlling the alignment of the polymeric chains in the tested fiber. Birefringence profiles were determined for iPP fibers at different stretching speeds. The average values of maximum (observable) birefringence for iPP fibers were calculated and found to be 0.042, 0.027 and 0.026 for untreated and annealed samples at temperatures of 80 °C and 120 °C, respectively.

1. Introduction

Standard isotactic polypropylene (iPP) fibers are produced by high-speed melt spinning processes extending the fibers from the molten state at take up speeds of typically a few thousand meters per minute. In such processes, only low molecular orientation can be imposed in the fiber since pulling out from the spinneret is directly accompanied with relaxation of the molecules. Nevertheless, standard fibers offer sufficient properties for many industrial applications. Fibers with enhanced stiffness and strength require a higher degree of molecular orientation, which can only be achieved by additional solid-state drawing of the fiber at temperatures being sufficiently low to prevent relaxation process. The induced polymer orientation and consequently, the tensile stiffness and strength of the fiber increase with increasing draw ratio [1].

Drawing of manmade fibers is an important process that could improves the textile characteristics. It is one of the most common methods for changing the physical properties of a polymeric material to strengthen it during processing. Knowledge and prediction of the structural deformation during drawing process is essential to evaluate the product performance. The mechanical properties and morphology of the product are strongly affected not only by its molecular characteristics such as the molecular weight (Mw) and molecular weight distribution (MWD) [2–6] but also by its processing conditions such as the drawing temperature, the drawing ratio, the strain rate, and the cooling rate after drawing [7–9].

One of the most available techniques for changing the polymeric and physical structure of fibers is the annealing process. Annealing is usually used to modify the microstructure and import a permanent set to semi-crystalline polymers and it may lead to changes in both the amorphous and crystalline phases in oriented fibers [10]. Annealing enhances the degree of crystallinity and the crystallite size of iPP fibers [11,12]. The effect of annealing depends on the annealing temperature and also on the time for which sample being held at this temperature [13].

Isotactic polypropylene (iPP) is a semi-crystalline polymer of considerable commercial importance that, when oriented, has a variety of applications. Orientation can be achieved using many techniques, including tensile drawing, roll drawing, blow molding, and solid state extrusion [14–16]. The level of orientation obtained from these techniques is reduced by relaxation, the recovery that occurs when the load is removed. Recovery has been attributed to the movement of the polymer chains in the amorphous regions [17], which reduces their extension. The oriented chains tend to be in the isotropic state which is the most energetically and favorable state. Efforts to maximize

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the orientation during the production would benefit from a more detailed understanding of the relaxation mechanisms.

The refractive index of polymer fiber can indicate the potential of this material for a specific purpose. It is one of the principal optical properties, that is directly related to the other optical, structural, electrical, and magnetic properties. When polymeric material has deformed, the refractive index tensor becomes anisotropic. Anisotropy of the refractive index can be measured using the birefringence, which is mechanically related to the stress via the stress optical rule (SOR) [18]. Birefringence is the opto-structural property that provides information about the overall degree of molecular orientation of a fiber. The characterization of molecular orientation and relaxation behavior under strain is of a particular interest for both industrial applications and fundamental understanding of the molecular mechanisms involved in the polymer deformation [19,20].

The interferometric methods have been widely used to study the opto-thermal and opto-mechanical properties of natural, synthetic and optical fibers. Two- and multiple-beam interferometers are common techniques used to study the optical properties of fibers during mechanical and thermal deformations. Microinterferometry is mainly based on the interaction of light with the object (i.e., fibers). The interferometric technique always gives fringe patterns or microinterferograms from which valuable qualitative and quantitative information of fibers, such as refractive index, birefringence and other related properties, can be obtained [21–27].

In this work, the drawing and annealing treatment were used to improve the mechanical properties of iPP fibers. The key of the work is to carry out an interferometric study for investigating the optical and structural changes during the drawing of thermally treated (annealed) iPP fibers. This is in terms of annealing time, annealing temperature and stretching speed (strain rate). Two-beam polarizing interference (Pluta) microscope was used to monitor the online changes in optical-path differences of light passing through the tested fibers upon the different stretching speeds. Birefringence, optical orientation function, and birefringence profile of annealed iPP fibers were determined at different stretching speeds.

2. Theoretical considerations

2.1. Birefringence of fiber

Using two-beam polarizing-interference (Pluta) microscope [28,29], the birefringence of single fiber/filament can be determined via the following equation:

$$\Delta n = \frac{\Delta Z \lambda}{ht} \tag{1}$$

where ΔZ is the displacement of the fringe shift, t is the fiber thickness, h is the interfering spacing and λ is the wavelength of the light used. The technical error of measuring the birefringence by using Pluta microscope ranges from ± 0.001 to ± 0.003 [28,30].

2.2. Birefringence profile

To determine the birefringence profile of fiber using the non-duplicated image, taking into account the refraction of the incident beam by the fiber layers, the following profile equation was used [31]:

$$\Delta n_Q = \left[\frac{1}{R-(Q-1)a}\right]\left[\frac{\lambda \Delta Z_Q}{2b} - a\sum_{j=1}^{j=Q-1}\Delta n_j\right] \tag{2}$$

where a is the layer thickness ($a=R/N$), N is the suggested number of layers, R is the fiber radius, Q is the layer number, j is an integer runs from 1 to $(Q-1)$, and ΔZ_Q is the fringe shift displacement corresponding to the Qth layer. For automatic measurement of the birefringence profile, software program was designed by Sokkar et al. [31]. In this program, the phase distribution was obtained directly from the original microinterferogram by using the Fourier transform technique, then, the birefringence profile was determined using Eq. (2).

2.3. Molecular orientation factor

To examine the effect of stretching on the fiber structure, the orientation factor was suggested and calculated by Hermans [32,33]. This factor/parameter is most probable quantity that used to characterize the molecular orientation of polymers. For a drawn fiber, the molecules are considered to be fairly aligned along the drawing direction (fiber axis) but randomly arranged in the transverse direction. The overall degree of molecular orientation or Hermans orientation factor, $\langle f(\cos\theta)\rangle$, is related to the molecular orientation distribution according to

$$\langle f(\cos\theta)\rangle = \frac{[3\langle \cos^2\theta\rangle - 1]}{2} \tag{3}$$

where θ is the angle between the direction of stretch and the local chain axis of the fiber. The parameter $\langle \cos^2\theta\rangle$ characterizes the orientation distribution. The orientation factor $\langle f(\cos\theta)\rangle$ is related to the birefringence of fibers as follows:

$$\langle f(\cos\theta)\rangle = \frac{\Delta n}{\Delta n_o} \tag{4}$$

where Δn_o is the intrinsic birefringence of the sample or the birefringence of perfectly axially oriented fibers. In case of polypropylene fibers, the value of $\Delta n_o = 0.045$ [34].

2.4. Shrinkage

The percentage shrinkage of thermally treated polymeric fiber can be calculated as follows:

$$\text{Percentage shrinkage} = \frac{L_o - Ls}{L_o} \times 100\% \tag{5}$$

where L_o is the initial length before the thermal treatment and Ls is the length of the treated fiber.

3. Experimental technique

A combination of the stretching device that has been designed and modified by Sokkar et al. [35,36] and Pluta microscope [28,29] was used in this work. It is a programmable system, able to achieve different detections via process control software. Fig. 1

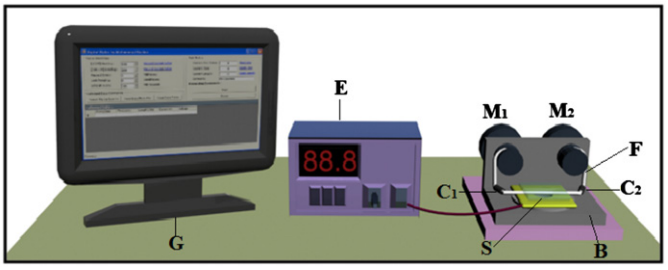

Fig. 1. Mechanical stretching device, where, M_1 and M_2 are two symmetrical cylinders axially fixed to two identical stepper motors, F is the fiber under test, S is a flat glass slid, B is a metallic base, C_1, C_2 are the magnetic clamps, E coupled with electronic temperature controller and G computer.