

The Influence of Resin Characteristics on the High Speed Melt Spinning of Isotactic Polypropylene. II. On-Line Studies of Diameter, Birefringence, and Temperature Profiles

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Synopsis

An investigation was carried out of the high speed melt spinning of three polypropylene resins with melt flow indices of 12, 35, and 300. On-line measurements were made of diameter, birefringence, and temperature as a function of distance from the spinneret for a range of spinning conditions for each polymer. A plateau (decrease of cooling rate) in the temperature profile was associated with the occurrence of crystallization in the spinline. The position of this plateau correlated with a rapid rise in the birefringence profile and a rapid decrease in the rate of drawdown in the diameter profile. The temperature and birefringence profiles were used to determine the temperature and position on the spinline at which the onset of crystallization occurred. It was found that the position and temperature of crystallization onset varied considerably with changes in take-up velocity, extrusion temperature, and resin melt index (weight average molecular weight). The crystallization onset occurred nearer the spinneret and at higher temperatures with (1) an increase of take-up velocity, (2) a decrease of extrusion temperature, or (3) a decrease of resin melt flow index. An analysis was carried out to estimate the rate of stress development with distance along the spinline; the results were also used to estimate the stress at the onset of crystallization for each spinning condition. It was concluded that the observed behavior could be attributed to the role of spinline stress in producing molecular orientation and consequent increase of crystallization rate.

INTRODUCTION

In this paper, Part II of a series dealing with the effect of resin characteristics on the structure and properties of polypropylene filaments prepared by high speed spinning, we describe the results of on-line measurements of diameter, birefringence and temperature as a function of distance from the spinneret. Measurement of these profiles gives insight, beyond that which can be obtained by examination of the as-spun filaments, into the manner in which changes of spinning and resin variables result in changes in the structure and properties developed in the filaments. Such profiles establish the very great significance of molecular-orientation-induced crystallization in controlling the rate and temperature of structure formation during melt spinning. Ultimately, these effects must be understood if we are ever to be

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able to mathematically model the melt spinning process for crystallizable polymers and if we are to be able to predict *a priori* the effect of changes of spinning conditions and resin characteristics on the structure and properties of the spun filaments. Further, experimental data for diameter, birefringence, and temperature profiles are essential for comparison and testing of any attempts at mathematical modeling.

On-line studies of structure development during melt spinning were first reported by Katayama, Amano, and Nakamura.¹ These authors carried out on-line X-ray diffraction studies and measured temperature, birefringence, and diameter profiles on running spinline of polyethylene, polypropylene, and polybutene-1. They concluded that crystallization kinetics may be greatly enhanced during melt spinning, as compared to quiescent conditions, due to the molecular orientation caused by the stretching of the melt. Later Nadella et al.² among others³⁻⁶ made more quantitative studies of melt spinning of isotactic polypropylene, including measurements of spinline stresses, and showed that the morphology of spun filaments was largely determined by the stress in the spinline at the point of crystallization. These early investigations using on-line techniques were carried out on polypropylenes with relatively low melt flow indices and at low spinning speeds. Other than a few studies⁷⁻¹⁰ dealing with poly(ethylene terephthalate) there have been no previously published investigations using on-line techniques to study spinline dynamics and structure development in the high speed regime. In the present paper we present detailed results of on-line measurements of diameter, birefringence, and temperature profiles for three isotactic polypropylene resins with melt flow indices ranging from 12 to 300 g/10 min and at spinning speeds ranging up to nearly 6000 m/min. These polypropylenes and spinning conditions are the same as those used in our companion paper, Part I, dealing with the structure and properties of the as-spun filaments.¹¹

EXPERIMENTAL

Materials and Melt Spinning Procedures

The isotactic polypropylenes, the melt spinning apparatus and the spinning conditions used in this study were all identical to those used in Part I.¹¹ The reader is referred to that paper for a detailed description of the materials and spinning procedures. In summary, three polypropylenes with melt flow indices of 12, 35, and 300 g/10 min were studied. These are referred to as samples E-012, E-035, and E-300, respectively. The two resins with the higher melt flow indices, E-035 and E-300, had much narrower molecular weight distributions than the E-012 resin.

In the range of spinning speeds of interest, between 1000 and 6000 m/min, the E-012 polymer could only be spun at an extrusion temperature of 230°C or above, while the E-300 sample failed by capillarity at extrusion temperatures of 230°C and above. The latter sample was therefore only studied at an extrusion temperature of 210°C and the former at 230°C. The E-035 sample was studied at extrusion temperatures of 210, 230, and 250°C. The extrusion rate was kept constant at 1.68 g/min through the monofilament spinneret of diameter 0.762 mm.

On-Line Measurements

Birefringence and diameter of the running spinline were measured as a function of distance from the spinneret with an Olympus polarizing microscope mounted at the desired position and angle with respect to the spinline. The arrangement is shown schematically in Figure 1. A small V-groove, arc-shaped aluminum guide was used to help stabilize the spinline within the microscope viewing field. The diameter of the spinline was first measured using a bifilar eyepiece; five readings were made and averaged at each position. Then the retardation was measured using a four order Berek compensator, and the birefringence was computed in the usual way by dividing the retardation by the filament diameter. It should be noted that there was a small effect on the diameter and birefringence profiles due to the friction

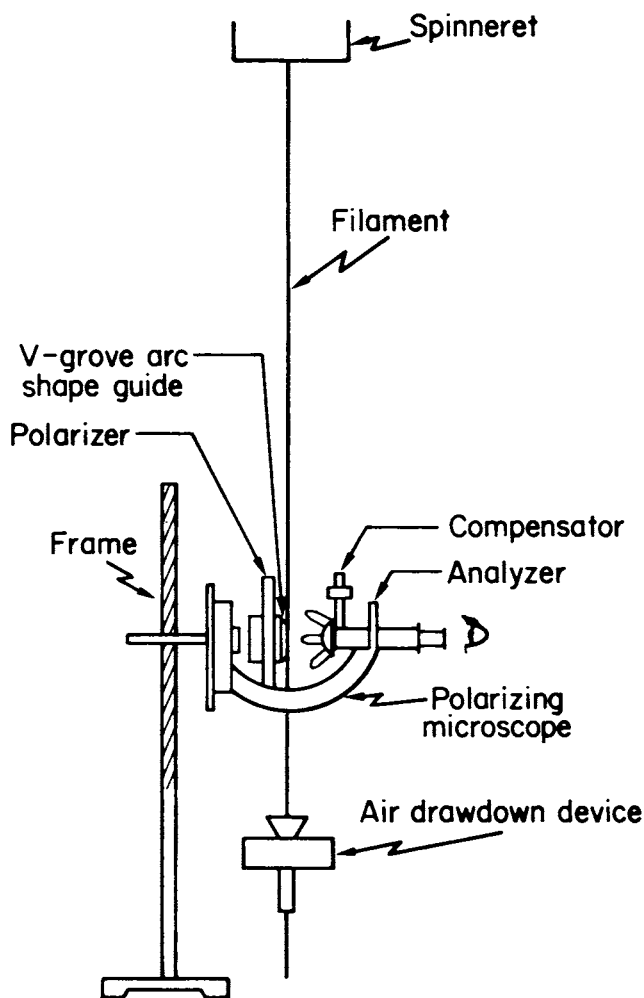


Fig. 1. Schematic of on-line birefringence measurement.

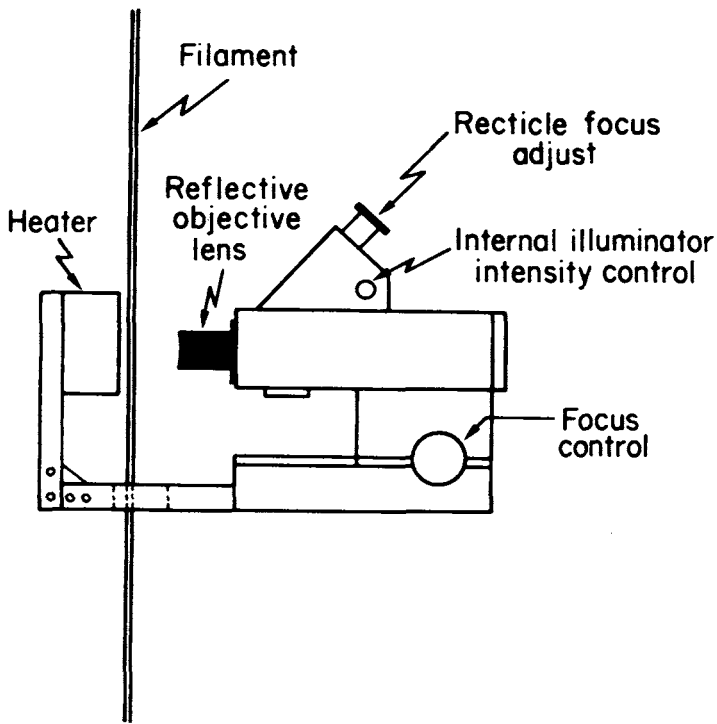


Fig. 2. Schematic of on-line temperature measurement using infrared microscope.

between the guide and the filament. However, by careful adjustment of the position of the guide in relation to the filament this effect could be rendered negligible.

The temperature profiles were obtained using a Barnes infrared microscope. A null-balance technique illustrated in Fig. 2 was used wherein the temperature of the spinline was compared with that of a small heater held at a known temperature. The infrared radiation from the filament was viewed against the background provided by the heater. The point on the spinline at which the radiation from the filament did not perturb that from the heater alone was taken to be at the same temperature as that of the heater.

COMPUTATION OF SPINLINE STRESSES

The stresses in the spinline at the position corresponding to the onset of crystallization were estimated using the following analysis. The rheological force at a distance z from the spinneret, $F(z)$, is given by the force balance:

$$F(z) = F(0) + F_{\text{inert}} + F_{\text{drag}} + F_{\text{surf}} - F_{\text{grav}} \quad (1)$$

where $F(0)$ is the rheological force at the exit to the spinneret. Generally, the surface tension force F_{surf} is negligible. For the case of high speed spinning the gravitational force F_{grav} is often small in comparison to the inertia force F_{inert} and the air drag force F_{drag} ; thus it is frequently neglected.¹² However, in the present case we elected to compute F_{grav} . To a first approximation, we may

write

$$F(z) = F(0) + F_{\text{inert}} + F_{\text{drag}} - F_{\text{grav}} \quad (2)$$

where the values of F_{inert} , F_{drag} , and F_{grav} are given by

$$F_{\text{inert}} = W[V(z) - V(0)] \quad (3)$$

$$F_{\text{drag}} = \int \rho_{\text{air}} C_d V^2 \pi D dz \quad (4)$$

$$F_{\text{grav}} = \pi g \int \rho (D^2/4) dz \quad (5)$$

The local drag coefficient C_d was evaluated from boundary layer theory by Sakiadis.¹³ The density of the melt as a function of temperature was obtained from the literature.¹⁴ The velocity V , diameter D , and mass throughput W are interrelated through the continuity equation

$$W = \rho(\pi D^2/4)V \quad (6)$$

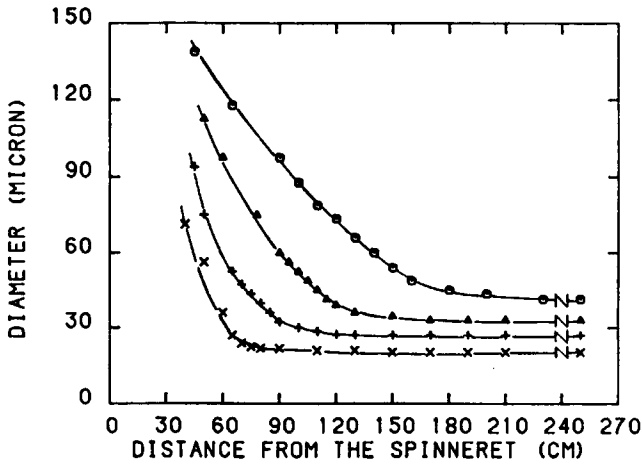
The experimental diameter and temperature profiles were used as input to a computer program to evaluate eqs. (3), (4), and (5) at each point on the spinline. The value of $F(0)$ was estimated from the elongational viscosity of polypropylene as a function of temperature and molecular weight using the data of Minoshima et al.¹⁵ Equation (2) was then used to compute values of $F(z)$, and spinline stress was obtained by dividing $F(z)$ by the filament cross-sectional area.

RESULTS AND DISCUSSION

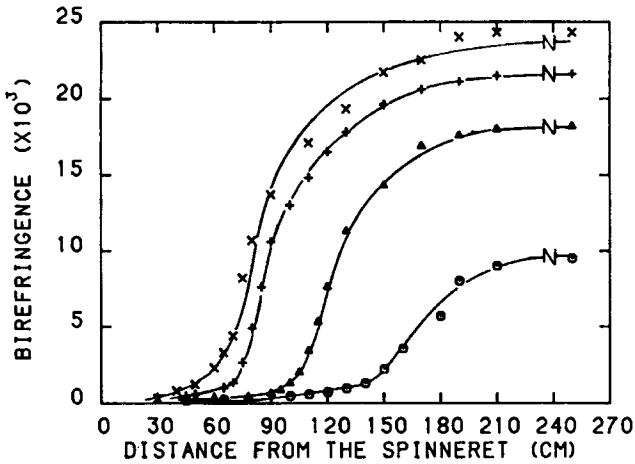
General Observation of On-Line Behavior

Figure 3 presents data obtained by on-line measurement for diameter, birefringence, and temperature as a function of distance from the spinneret for filaments spun from the E-035 resin with an extrusion temperature of 230°C. In general, the diameter [Fig. 3(a)] draws down rapidly in the region a few centimeters below the spinneret, although measurements are difficult in this region due to the size and tackiness of the filaments. Ignoring the die swell region within a centimeter or two of the spinneret, the rate of drawdown slowly decreases at first, but then decreases more rapidly as the filament diameter levels out at its final value. With a constant mass throughput the final diameter decreases with increase of take-up velocity, whereas the rate of the initial drawdown increases with the take-up velocity.

The birefringence, shown in Figure 3(b), rises slowly at first and then exhibits a rapid rise at a position on the spinline corresponding to the point where the diameter is beginning to level out. After this rapid rise, the rate of increase of birefringence decreases again and the birefringence approaches a value very close to the value measured on the as-spun filament. Note that the values for the as-spun filaments are shown as the last points on the curves, following the breaks in the curves, in each figure.



(a)

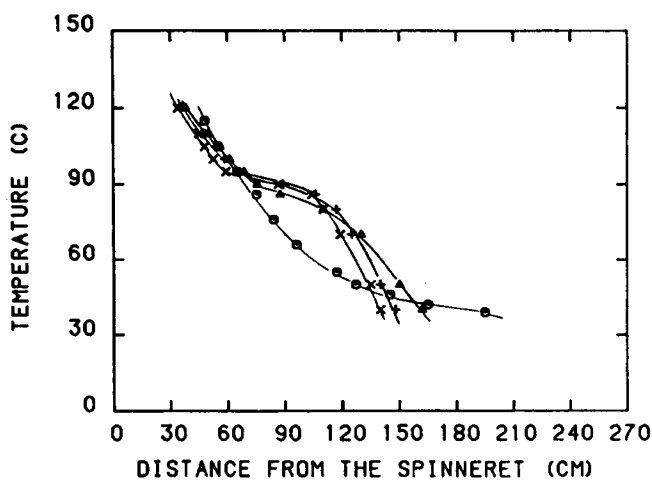


(b)

Fig. 3. On-line measurements for the E-035 resin spun with an extrusion temperature of 230°C: (a) diameter profile; (b) birefringence profile; (c) temperature profile; (○) 1430 m/min; (▲) 2,240 m/min; (+) 3300 m/min; (x) 5700 m/min.

The temperature profiles, Figure 3(c), exhibit easily observable plateaus, at least for the higher spinning speeds. The plateaus occur at positions that closely correspond with the rapid rise in the birefringence profiles. The plateaus are caused by the release of the heat of crystallization when the polymer crystallizes; they are a clear indication that the leveling out of the diameter and the rapid increase of birefringence are a result of crystallization occurring in the spinline. Although birefringence itself is a measure of orientation, the growth of crystals from oriented nuclei during stress-induced crystallization causes a rapid increase in orientation and birefringence.

It should be noted that the temperature range corresponding to the plateau increases with increased spinning speed. Further, at the lowest spinning speed



(c)

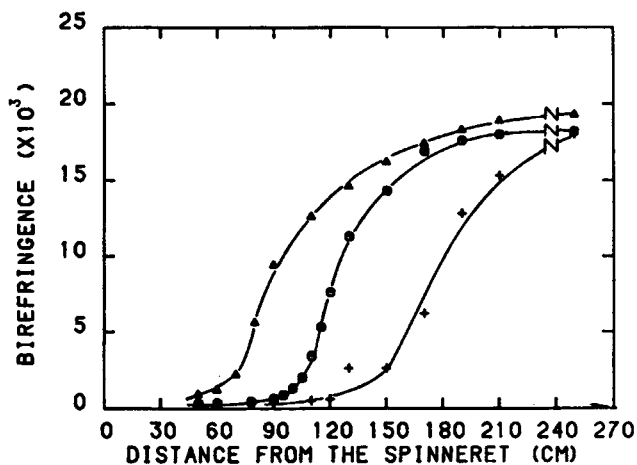
Fig. 3. (Continued from the previous page.)

of 1430 m/min the plateau in the temperature profile is very broad and occurs at a temperature that is very much lower than for the higher spinning speeds. The data of Figure 3 clearly show that increasing the take-up velocity at constant mass throughput causes crystallization to occur nearer the spinneret [Fig. 3(b)] and at higher temperature [Fig. 3(c)]. This occurs, even though the cooling rate of the filament increases due to the increased velocity of the filament through the cooling medium and increased surface to volume ratio due to the reduced diameter. Such a result can only be attributed to an enormous increase of crystallization kinetics resulting from the increased spinline stresses and the accompanying molecular orientation. For the present sample and spinning conditions there is a substantial increase of crystallization start temperature in the take-up velocity range between 1000 and 3000 m/min followed by a leveling off with further increase of take-up velocity. This suggests that the effect of stress (or molecular orientation in the melt) on crystallization rates saturates above a certain stress level.

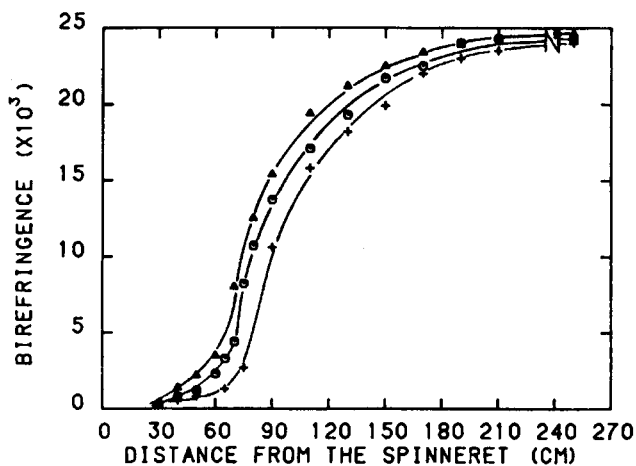
The results shown in Figure 3 should be compared with the wide angle X-ray diffraction (WAXS) patterns for as-spun samples spun under these conditions presented in Figure 6 of our earlier paper, Part I.¹¹ It was shown there that the smectic structure is developed in filaments of the E-035 polymer spun at 1430 m/min; spinning at higher spinning speeds results in filaments with monoclinic structures. Figure 3(c) shows that the smectic structure forms from the melt when the filament is supercooled to a temperature of the order of 50°C and that formation of the monoclinic phase occurs when crystallization takes place at significantly higher temperatures.

Effect of Extrusion Temperature and Extrusion Rate

Figure 4 compares on-line birefringence profiles for the E-035 resin spun at the same take-up velocities and extrusion rates but different extrusion temperatures. Extrusion temperature has a marked effect on the position in the



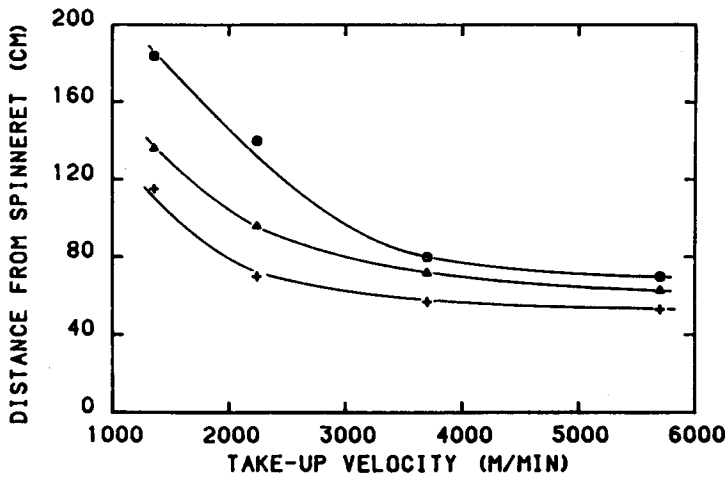
(a)



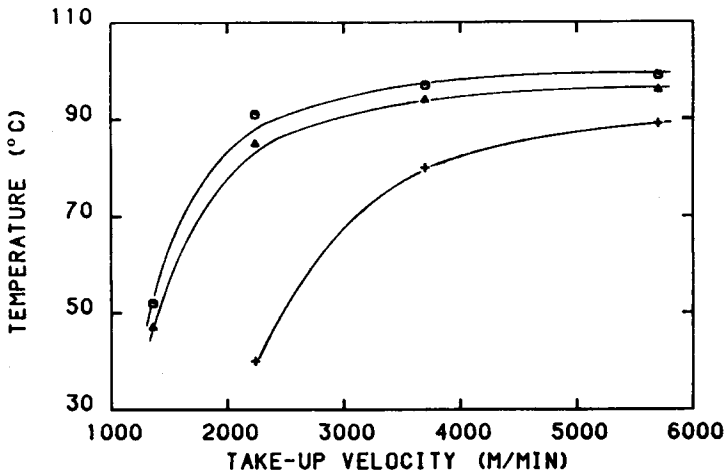
(b)

Fig. 4. Comparison of birefringence profiles for the E-035 resin spun with different extrusion temperature and take-up velocities. Extrusion temperature: (\blacktriangle) 210°C; (\odot) 230°C; ($+$) 250°C; (a) 2240 m/min; (b) 5700 m/min.

threadline at which birefringence develops as is clearly seen in Figure 4(a) where the take-up velocity is 2240 m/min. At higher spinning speed the differences are not as great [Figure 4(b)]. The effect of extrusion temperature on the position in the spinline at which crystallization begins, based on the experimental temperature and birefringence profiles, is plotted vs. take-up velocity in Figure 5(a) for each extrusion temperature studied. At low to moderate take-up velocity the position on the threadline at which crystallization occurs is quite sensitive to extrusion temperature. As the take-up velocity increases, the point on the spinline where crystallization occurs becomes less sensitive to the extrusion temperature.



(a)



(b)

Fig. 5. (a) Position and (b) temperature on the spinline at which crystallization begins for the E-035 resin. Extrusion temperature: (+) 210°C; (▲) 230°C; (○) 250°C.

The temperature at which crystallization begins in the spinline is plotted vs. take-up velocity for each of the three different extrusion temperatures in Figure 5(b). It is clear from Figure 5(b) that the temperature at which crystallization starts is also a function of the extrusion temperature. As extrusion temperature increases, the temperature at which crystallization occurs in the spinline decreases at any given take-up velocity. This can be understood on the basis that higher extrusion temperatures require lower spinline stresses in order to achieve drawdown of the filament. At these lower melt stresses the filament supercools somewhat more before there is sufficient time and driving force to cause crystallization. This effect will be discussed in more detail in a later section in which the calculated spinline stresses at the onset of crystallization are presented.

Effect of Resin Characteristics

The effect of changing the resin characteristics on the development of the birefringence profiles is shown in Figures 6 and 7. See Table I of Part I for molecular weight averages and polydispersities of these resins. Figure 6 presents the birefringence profiles for sample E-012 which has lower melt index, higher weight average molecular weight, and broader molecular weight distribution than sample E-035. Note that the data shown in Figure 6 are for an extrusion temperature of 230°C, the same as that shown for the E-035 samples in Figure 3(b). Several differences are quite apparent between the results shown in Figure 6 for the E-012 polymer and those shown in Figure 3(b) for the E-035 polymer. Both the diameter drawdown and rapid rise of birefringence occur much closer to the spinneret for the E-012 polymer than for the E-035 polymer for any given spinning condition. At the lower take-up velocity of about 1400 m/min, the ultimate on-line birefringence developed is considerably higher for the E-012 resin than for the E-035 resin. However, with increase of take-up velocity, this situation reverses and at the highest take-up speeds the E-035 resin develops the higher birefringence. In the high take-up velocity case, at least, the birefringence rises more slowly for the E-012 resin than for the E-035 resin. Again, the differences of on-line behavior between the two resins is consistent with the final as-spun birefringence values reported in Part I.

The development of the birefringence for the E-035 and E-300 resins is compared in Figure 7. The extrusion temperature for the data of both Figures 7(a) and 7(b) was 210°C. As pointed out previously, sample E-300 could not be spun at 230°C and above due to its low viscosity and resulting spinline failure due to capillarity. Resins E-300 and E-035 have very similar polydispersities but E-300 has a lower weight average molecular weight and hence a higher melt index than E-035. The results of Figure 7 exhibit the expected differences; at any given take-up velocity the birefringence starts to rise at a position further from the spinneret and achieves a lower ultimate value for

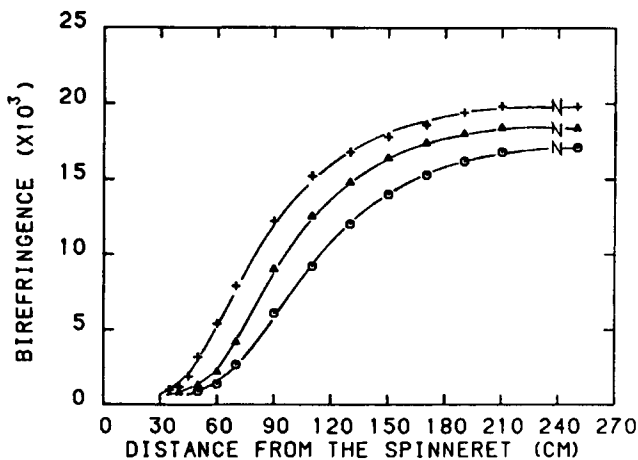
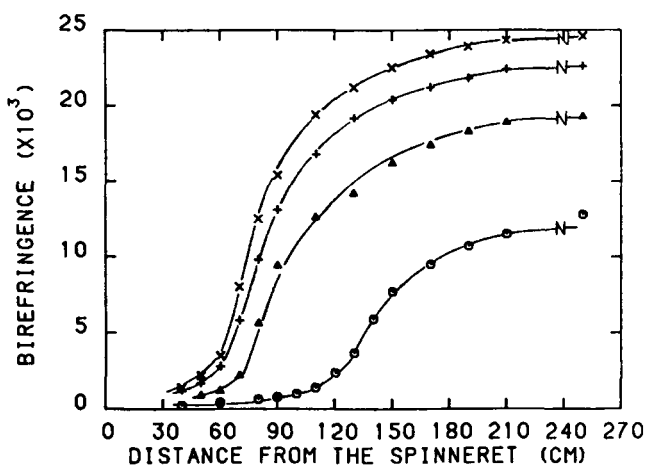
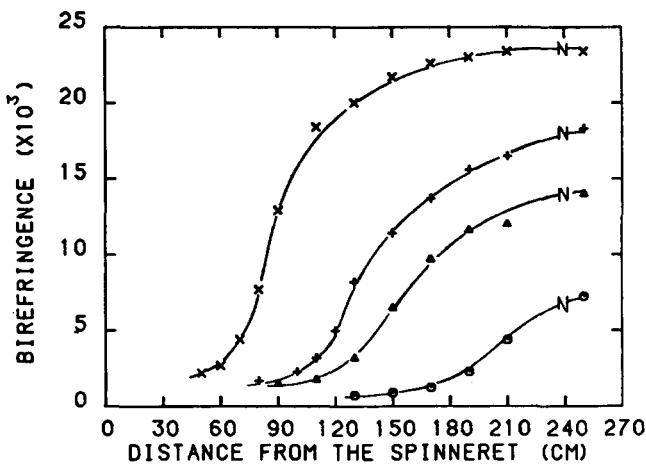


Fig. 6. Birefringence profiles for the E-012 resin. Extrusion temperature = 230°C. (○) 1360 m/min; (▲) 2500 m/min; (+) 3500 m/min.



(a)



(b)

Fig. 7. Comparison of birefringence profiles for the E-035 and E-300 resins for an extrusion temperature of 210°C. (a) E-035 resin: (○) 1360 m/min; (▲) 2240 m/min; (+) 3500 m/min; (x) 5700 m/min. (b) E-300 resin: (○) 1430 m/min; (▲) 2140 m/min; (+) 3300 m/min; (x) 5600 m/min.

the high melt index E-300 resin than for the E-035 resin. This is consistent with slower spinline stress development and lower spinline stresses for the E-300 resin. The general shape of the birefringence profiles are very similar for the two resins in keeping with the similarity of their polydispersities.

The temperature at which crystallization begins is compared for each of the three resins in Figure 8. Note that the curve for sample E-012 is for an extrusion temperature of 230°C while the other two curves are for extrusion temperatures of 210°C. These curves show that increasing the weight average molecular weight (i.e., reducing the melt flow index) causes a substantial increase in the crystallization start temperature at a given take-up velocity.

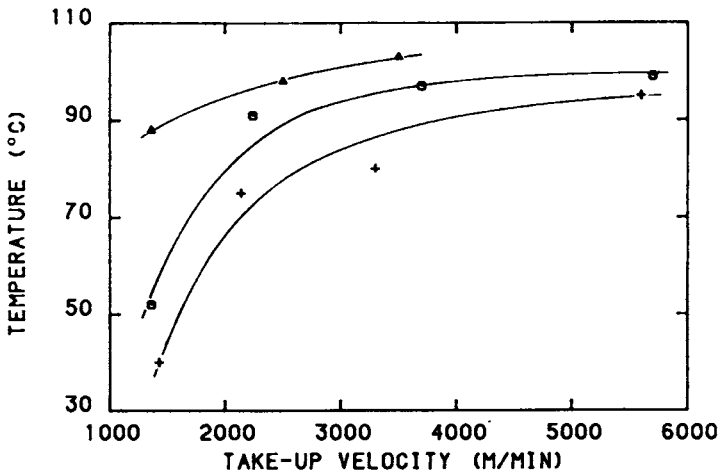


Fig. 8. Temperature at which crystallization begins for each resin: (▲) E-012; extrusion temperature 230°C; (○) E-035; extrusion temperature 210°C; (+) E-300; extrusion temperature 210°C.

Estimated Spinline Stresses

Although there have been many studies that have inferred the importance of stress-induced crystallization in polymer processing operations, few have actually attempted to calculate the stress at the point in the process where crystallization begins or as a function of position in the process. Based on the analysis described previously, we have estimated the stress as a function of distance from the spinneret for each profile. Since we also know the position at which the onset of crystallization occurs, we can obtain the stress at which crystallization starts for each of the spinning conditions and resins.

The results of these calculations show that, for constant mass throughput per spinneret hole, the stress increases more rapidly with distance from the spinneret as take-up velocity increases, as extrusion temperature decreases, or as weight average molecular weight increases (melt flow index decreases). Typical examples are shown in Figure 9. Note that arrows indicate the point corresponding to the onset of crystallization.

We may assume that crystallization kinetics are primarily a function of molecular orientation and temperature.¹⁶⁻¹⁸ The driving force for crystal nucleation increases with increasing molecular orientation or increased supercooling below the equilibrium melting temperature. Since, according to the rheoptical law, increased melt stress should correspond to increased molecular orientation,¹⁹⁻²¹ an increase of stress at a given temperature should represent increased driving force for crystal nucleation. In order for crystallization to begin on the spinline, a critical level of driving force for nucleation must be developed to allow nucleation within the time scale that a volume element of polymer in the filament remains within the temperature range where molecular mobility is sufficient for crystal growth.¹⁷ This time scale depends on the rate of cooling through the critical temperature range. These considerations readily explain the observations that crystallization takes place nearer the spinneret [refer to Figs. 5(a), 6 and 7] and at higher temperatures

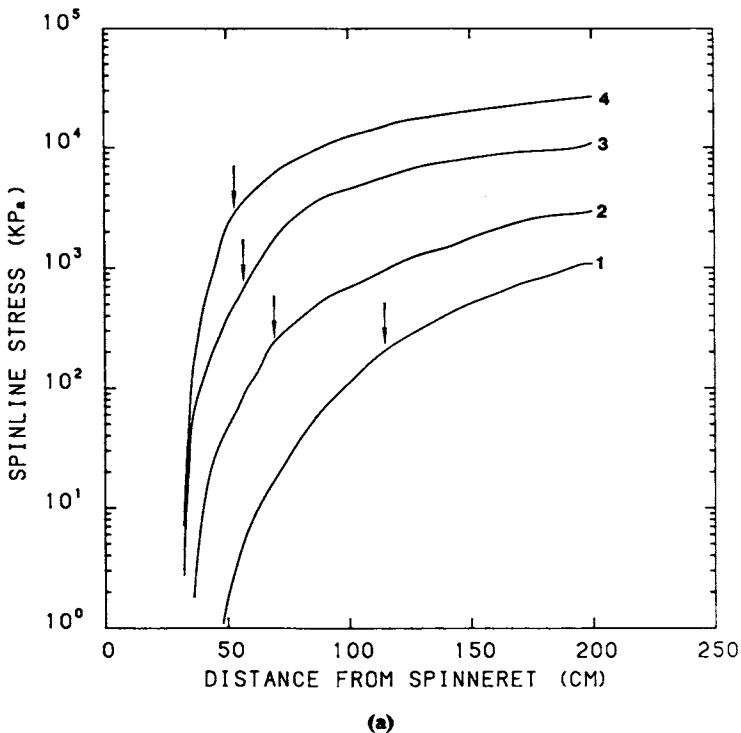
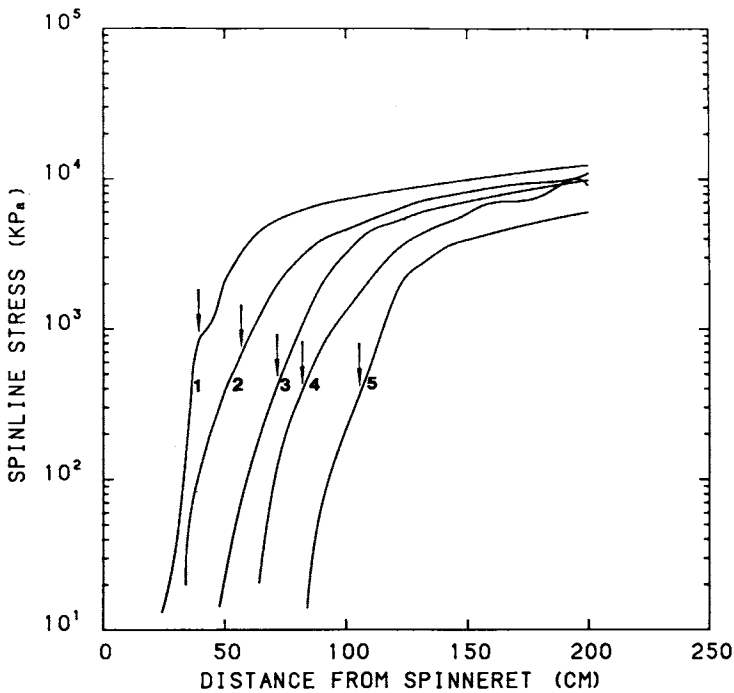


Fig. 9. Calculated spinline stress as a function of distance from the spinneret. (a) Comparison showing the effect of changing the take-up velocity for the E-035 resin, extrusion temperature = 210°C: (1) 1360 m/min; (2) 2240 m/min; (3) 3700 m/min; (4) 5700 m/min. (b) Comparison showing the effect of extrusion temperature and resin melt flow index: (1) E-012, 230°C, 3500 m/min; (2) E-035, 210°C, 3700 m/min; (3) E-035, 230°C, 3700 m/min; (4) E-035, 250°C, 3700 m/min; (5) E-300, 210°C, 300 m/min.

[Figs. 5(b) and 8] with (1) increased take-up velocity, (2) decreased extrusion temperature, or (3) increased weight average molecular weight. In each case the increased rate of stress build-up with distance along the spinline produces an increase in the driving force that allows crystal nucleation to occur at a higher temperature and within a shorter time period (and hence closer to the spinneret).

Although not studied here, the spinline stress should also be a strong function of mass throughput per spinneret hole. In general, increasing the extrusion rate at a given take-up velocity increases the filament diameter and reduces drawdown ratio (the ratio of take-up velocity to extrusion velocity). This reduces cooling rates but lowers spinline stresses. Under conditions for which stress is largely controlling the crystallization rates, we would expect this to move the position at which crystallization occurs to a point further from the spinneret and lower the temperature at which crystallization occurs.

Thus, whether an increase in the rate of stress buildup along the spinline is caused by an increase of take-up velocity, by a decrease of extrusion temperature or extrusion rate, or by an increase of resin molecular weight, it leads to an increase of the temperature and a decrease in the distance from the spinneret at which the onset of crystallization is observed.



(b)

Fig. 9. (Continued from the previous page.)

The values of the computed stresses at the onset of crystallization are presented in Table I. The trends are consistent with expectation and show that the stress at the onset of crystallization increases (1) with increase of spinning speed, (2) with decrease of extrusion temperature, and (3) with increase of weight average molecular weight. In general, the temperature at the start of crystallization on the spinline increases as spinline stress increases. An increase of resin melt index, for example, must be compensated by an increase of take-up velocity or a decrease of extrusion temperature (or both) if the onset of crystallization is to occur at the same temperature. However, based on the discussion of the last few paragraphs, we would expect a precise correlation with stress level only if cooling rate remained constant as such changes were made. It is also clear from the results that, at cooling rates met with in high speed spinning, stress levels of the order of 300 kPa are required to cause crystallization to the monoclinic phase for isotactic polypropylene. Further, since the effect of stress on crystallization rates is very great and the range of cooling rates met with in melt spinning processes is limited, we expect that the resulting orientation, morphology, and properties of as spun filaments should correlate approximately with spinline stress at the onset of crystallization as shown by previous investigators.^{2,5} In the case of high speed spinning, factors not accounted for by this argument, such as structural variations across the filament radius,^{22,23} may become important. In addition this argument does not allow for morphological variations resulting from

TABLE I
Stress at the Onset of Crystallization as a Function of Spinning Conditions and Resin

Sample	Extrusion temperature (°C)	Take-up velocity (m/min)	Distance at cryst. onset (cm)	Stress at cryst. onset (kPa)
E-300	210	1430	170	80.5
		2140	114	241
		3300	105	384
		5600	65	959
E-035	210	1360	115	208
		2240	70	253
		3700	57	702
		5700	53	2860
E-035	230	1360	136	142
		2240	96	190
		3700	72	437
		5700	62	2340
E-035	250	1360	184	118
		2240	140	162
		3700	80	330
		5700	70	2150
E-012	230	1360	60	331
		2500	50	548
		3500	40	885

changes in molecular weight distribution, as described in our earlier paper, Part I.¹¹

CONCLUSIONS

The results of the present on-line studies provide clear insight into the importance of stress-induced crystallization in the melt spinning of isotactic polypropylene filaments. The temperature and position on the spinline at which crystallization starts was found to vary substantially with take-up velocity, extrusion temperature, and resin melt flow index. For a given polymer and extrusion temperature, increasing the take-up velocity raised the temperature at which crystallization began in the spinline. Increasing the polymer viscosity in the upper part of the spinline by increasing weight average molecular weight or decreasing the extrusion temperature also resulted in an increase of crystallization onset temperature. It was concluded that the observed behavior can be attributed to the influence of stress in generating molecular orientation and consequent increase of crystallization kinetics.

For the E-035 resin the crystallization onset temperature was very sensitive to the take-up velocity in the range of approximately 1000–3000 m/min with the effect tending to saturate with further increase in take-up velocity. This corresponded to estimated stresses in the range of 100–1000 kPa. A similar, though not identical, level of stress was needed in order to achieve the same crystallization onset temperatures for other resins and spinning conditions.

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