

# Characterization of Fibers Produced from Blends of Polybutylene and Polypropylene

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## ABSTRACT

Blends of polybutylene (PB-1) and polypropylene were used to produce fibers at spinning speeds of 800-2100 m/min. Concentrations ranged from 0% PP to 100% PP. The stress-strain behavior of the resultant fibers was examined, and the fibers were analyzed for crystallinity via DSC (differential scanning calorimetry). Fibers produced from blends of PB-1/PP show mechanical properties that are in between the properties of the pure polymers. The tensile strength of 50% PB-1 fibers is comparable to the tensile strength of pure PP fibers. Fibers produced from blend compositions of 25 and 75% have higher tensile strengths than pure PP fibers, although these blend compositions have lower tensile strengths than pure PB fibers.

## INTRODUCTION

Polymer blending provides an opportunity to obtain new, economically-viable materials with improved properties. However, often polymers are incompatible, and this incompatibility produces a separation of phases that causes poor mechanical properties in the blend. Compatibility of the components in a blend includes many factors such as the chemical composition and the difference in crystallographic parameters of the components. Even processing characteristics and conditions may influence phase separation.

Many researchers have studied different pairs of polymers to try to find the conditions necessary to produce compatibility. Special attention has been given to blends wherein each component is (individually) capable of being crystalline. For example, Stein et al. (1978) studied PBT/PET blends and found a single glass transition temperature in the blends as evidence of miscibility of both polymers. Using WAXD, the authors also found that the crystalline diffraction peak positions did not change at different compositions of the blends. This behavior suggested that there was no co-crystallization present.

Other crystalline polymer pairs have been studied wherein a synergism was found in their mechanical properties. For example, PP/HDPE was studied by Noel and Carley (1975) in a wide range of compositions. They found that very low amounts of HDPE caused a maximum in the tensile modulus and strength of the blends. They concluded that, when the HDPE component is less than 10%, the HDPE acts as a stiffener. In contrast, small amounts of PP were found to make the blend ductile. Stell et al. (1976) studied polymer blends of PS/HDPE in stretched films. These authors found that the mechanical properties of the films (tensile strength and elongation at break) were best when the films were quenched right after hot stretching.

The present study is concerned with crystalline blends of PB-1/PP and characterization of the fibers produced from these blends. Although this type of blend has been studied over the years, especially in the form of thin films and molded samples, there is little research on the characteristics of fibers formed from this particular blend. Foglia (1969) concluded that the PB-1/PP blends are "highly compatible in all proportions under normal operating conditions". In later studies, Siegmann (1979; 1982) examined the interactions of both components and found that the presence of PP in PB depresses the melting temperature of both components in the blend. From thermal analysis he found that PP and PB-1 seemed to crystallize separately with no evidence of co-crystallization. In addition, he reported that the crystallinity ratio of PB-1/PP is not linear with respect to percentage of composition. Siegmann also ran X-ray analyses that supported his conclusion that crystallinity ratio is not linear with respect to composition percentage. In addition, the X-ray results showed that the crystalline size did not change with the ratio of the polymers present in the blend. Siegmann suggested that the morphology of the blends changes from spherulite to branched crystallites as the composition changes.

Gohil and Peterman (1980) conducted studies on PP/PB-1 blends and found indication of co-crystallization in blends that had very low amounts of either one of the components. The authors also found that there can be a segregation of phases during the crystallization process, but this segregation can be suppressed by using their “melt extraction technique” and by making the process relatively fast.

Takahashi et al. (1987) studied crystallization in drawn PB-1/PP blends. They found that the PP/PB-1 interface acts as a nucleating site in the crystallization of the disperse PB-1. They also showed that the lamellae axis of PB-1 grows perpendicular to the drawing direction. They suggested that the crystallization of PB-1 occurs faster and in an oriented manner due to stresses found in this disperse phase.

Hsu and Geil (1987) studied the crystallization of quenched PB-1/PP blends. Their X-ray measurements agreed with the results of Siegmann in that there was no evidence of co-crystallization of the polymers. However, Hsu and Geil used DSC to study the melting temperatures of the blends and found that there was no significant shift of the peaks compared to the individual constituent polymers; these results are in contrast to the results of Siegmann. Hsu and Geil also found that there is a significant shift in the crystallization temperatures. They suggested that the higher  $T_c$  in PP could be caused by the PB-1 molecules acting as impurities.

Hsu and Geil determined that, in the peak ratios of PB-1 versus PP, the total crystallinity of PB-1 is more affected by the presence of PP than vice versa. They also studied the glass transition temperature of the blends. They found two distinct peaks that indicate the existence of two amorphous phases. This contrasts with the complete miscibility of the amorphous phase that was suggested by Foglia (1969). Hsu and Geil concluded that the possibility of miscible blends of PP/PB-1 exists, but this blend is very difficult to obtain from a regular melt mixing process.

Lee and Chen (1987) found that there can be an increase in the crystallinity of PB-1 caused by adding PP, but for this to occur the PP has to crystallize first so that it can act as a nucleating agent for the PB-1. In a subsequent paper, Lee and Chen (1993) stated that in PB-1/PP blends there is a synergism of the mechanical properties that occurs when the blends are processed by injection molding and compression molding. Lee and Chen added that the mechanism for this synergism is unclear. In a recent paper, Ortiz and Shambaugh (2005) spun pure polybutylene (PB-1) fibers at spinning speeds of 250-2500 m/min. A tensile tester was used to analyze the stress-strain behavior of

these fibers. In addition, birefringence and the effect of aging were examined. In the present paper, PB-1/PP blends were used to produce fibers and the mechanical properties of these fibers were analyzed. Because of the unique processing history of fibers (e.g., with respect to high rates of strain) versus other shapes, this work fills a necessary void. Though PB-1 is more expensive than PP, it may be advantageous to blend PP with PB-1 (particularly at low PB-1 concentrations) to gain property advantages versus pure PP.

## EXPERIMENTAL DETAILS

Grade 0400 Polybutylene-1 (Basell polyolefins) was used in our study. This PB-1 has a melting temperature of 123.9 – 126.1 °C, a specific gravity of 0.915, and an MFR (melt flow rate) of 20. Fina Dypro® isotactic polypropylene pellets were also used in the experiments. The polypropylene had a MFR of 88, an  $M_w$  of 165,000 g/mol, and a polydispersity of 4. Further details on these polymers are contained in the paper by Ortiz and Shambaugh (2005).

For the production of the PP/PB-1 melt blends, a Reeves® helicone mixer was used. This mixer unit has a capacity of 15 to 150 cm<sup>3</sup> and can operate at pressures up to 345 kPa (50 psig) and temperatures up to 232°C (450°F). For our PP/PB-1 blends we used a mixing time of 1 hour at a temperature of 190°C. The blend was drained by gravity from the mixer and was fed to a mechanical roll and cutter. The resulting pieces were used as feed for the melt spinning equipment described next.

The melt spinning of the blended polymers was carried out with two types of spinning equipment. The first type (see *Fig. 1*) consisted of a Brabender™ single screw extruder, a spin pack equipped with a gear pump, and a spinneret. The extruder barrel had a 19.0 mm (0.75 in.) diameter, a 381 mm (15 in) length, and a compression ratio of 3:1. The spin pack contained a Zenith™ gear pump that metered the molten polymer and pushed the polymer into the spinneret. A single-hole spinneret was used for the production of the fibers. This spinneret orifice had a diameter of 0.407 mm (0.016 in) and a length of 2.97 mm (0.117 in).

The second type of equipment used for the spinning of melt blends was a ram extruder. This extruder operated in batch, rather than continuous, mode and provided smooth control at low polymer flowrates. The ram extruder barrel had a length of 8 inches (203.2 mm) and a 0.375 inch (9.525 mm) diameter. The same spinneret that was used for the continuous system (*Fig 1*) was also used for the ram extruder.

For both types of spinning equipment, the polymer fila-

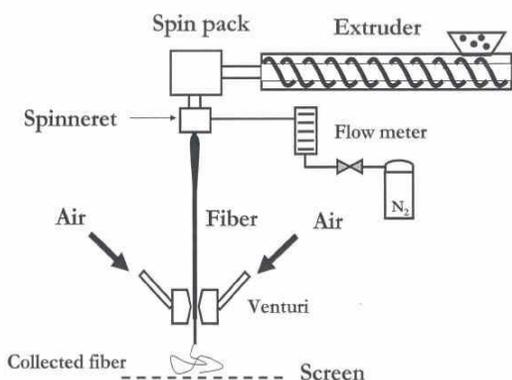


Figure 1. The experimental equipment used to spin fibers.

ment was drawn by a mechanical take up roll located one meter below the spinneret. However, this roll was used only for spinning speeds up to 1500 m/min. For higher spinning speeds (up to 4000 m/min) an air venturi was used. (Since the fiber is solidified at the position where the roll or venturi is used, there is essentially no difference between fibers spun with these different devices.) The Brabender extruder was kept at 225°C, the spin pack was kept at 205°C, and the spinneret was kept at 190°C. For the ram extruder, the extruder barrel was kept at 225°C, and the spinneret was kept at 190°C. The two polymer flow rates that were used for studying the PP/PB-1 blends were 0.23 g/min (run on the ram extruder) and 1.0 g/min (run on the continuous extruder). The fiber collection speeds varied from 800 to 2100 m/min.

Offline diameter measurements on the fibers were taken with a Nikon Labphoto2 POL Optical Microscope equipped with a micrometer eyepiece. Birefringence measurements were also done with this microscope.

A model TT-B-L Instron tensile tester was used to measure the mechanical properties of individual filaments of polybutylene, polypropylene, and blends of PP/PB-1. Each fiber sample was carefully placed between pneumatic grips; a gauge length of 2.3 cm was used. The fiber was then stretched at a constant cross-head speed of 2.54 cm/min (1.0 in/min) until breakage occurred. The resultant stress-strain curve was used to determine yield strength, elongation at break, yield elongation and modulus of elasticity.

A Q1000 TA thermal analyzer was used to do DSC (differential scanning calorimetry) tests to measure the crystallinity of the components of the blends. Fiber samples from 1 mg to 8 mg were cut, weighed, and

then placed in aluminum pans. Silicone oil was used to provide good thermal contact between the fibers and the pan. Scans were taken in a temperature range from 40 to 180 °C at a rate of 5 °C/min.

## RESULTS AND DISCUSSION

Continuous fibers were spun from blends of polybutylene (PB-1) and polypropylene. Concentrations ranged from 0% PP to 100% PP. Spinning speeds were 800-2100 m/min, and polymer flowrates were 0.23 and 1.00 g/min.

### Mechanical Properties

Figure 2 shows the results of tensile runs on fibers produced from various blends of PB-1/PP. The ordinate gives the stress in textile units of g/denier; these stress values were calculated with the following conversion formula (Ahmed, 1982):

$$\text{Tenacity} = \frac{\sigma}{\rho} \cdot 7.82 \cdot 10^{-5} \quad (1)$$

where tenacity is in g/den,  $\rho$  is the fiber density in g/cm<sup>3</sup>, and  $\sigma$  is the fiber stress in psi.

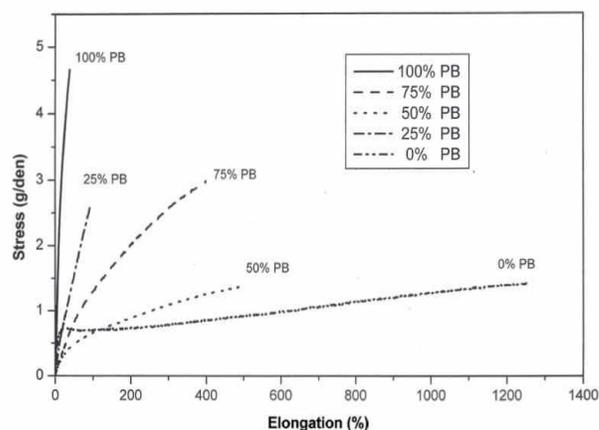


Figure 2. Tensile test runs on fibers produced from PB-1/PP blends.

All fibers were spun using a polymer flowrate of 0.23 g/min and a spinning speed of 800 m/min. The final diameters of the fibers were approximately 22.2 microns. Each curve is the average of 5 separate stress-strain tests.

For the development of Fig. 2, all fibers were spun using a polymer flowrate of 0.23 g/min and a collection speed of 800 m/min. The final diameter of the fibers was approximately 22.2  $\mu\text{m}$ . Fig. 2 shows that the pure PP fibers had high  $E_b$  (elongation at break) of about 1250%. In contrast, 100% PB fibers had much lower  $E_b$ , high modulus (stiffness), and high tenacity. Of the three blends tested, fibers with a composition of 25% PB have highest modulus, but the  $E_b$  is lowest of the

three blends, and the tenacity is about half of the tenacity of pure PB. The fibers with a composition of 50% PB have a tenacity that is less than half of the tenacity for either of the other blends, and the  $E_B$  is about 500%. Fibers with 75% PB show a high  $E_B$  of about 400%, but the tenacity of these fibers is the best of the three blends (about 3 g/den).

Fig. 3 shows additional results for tensile runs on fibers produced from various blends of PB-1/PP. For this figure, all fibers were spun at the same polymer flowrate (0.23 g/min) as for Fig. 2; however, for Fig. 3 a higher collection speed of 1750 m/min was used. The final diameter of the fibers was approximately 14.3  $\mu\text{m}$ , with a standard deviation of 1.4  $\mu\text{m}$ . The higher spinning speed causes a higher stress in the fiber threadline, which in turn lowers the elongation at break ( $E_B$ ) of PB-1/PP fibers to levels comparable to the  $E_B$  for pure PB-1 fibers. On the other hand, the  $E_B$  of pure PP (0 % PB) fibers spun at 1750 m/min is not significantly different than the  $E_B$  for PP spun at a low speed of 800 m/min (as shown in Fig. 2). Fig. 3 shows that fibers with 75% PB have higher tensile strengths than fibers with a composition of 25% or 50% PB; this result parallels the result shown for the lower spinning speed (see Fig. 2).

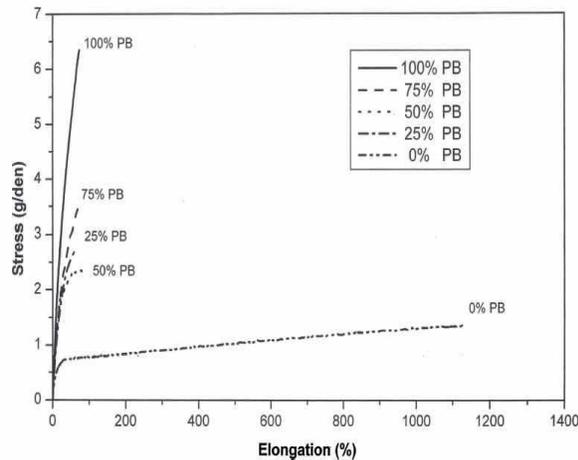


Figure 3. Tensile test runs on fibers spun at a higher speed of 1750m/min. The PB-1/PP blends were spun using a polymer flowrate of 0.23g/min. The diameters of the fibers were approximately 14.3 microns. Each curve is the average of 5 separate stress-strain tests.

Fig. 4 shows the breaking strength (tenacity) of fibers as a function of spinning speed. Data are given for a polymer flowrate of 0.23 g/min and spinning speeds from 800 to 2250 m/min. For each composition, the tenacity versus spinning speed curves are approximately linear; linear best fit curves are shown

on Fig. 4. The slopes of these lines are slightly positive, except for the curve for 100% PB -- for this composition, the slope is much higher, and the increase in tenacity with spinning speed is very apparent. Fibers with different PB weight composition have breaking strengths that lie in between the breaking strength of fibers produced from pure polymers. However, the fibers with 50% PB are only slightly stronger than the 0% PB fibers. In a rich PP blend (25% wt. PB-1); the tenacity of the fibers is lower than PB-1 rich blends (75% wt. PB-1). This result suggests that, for 50% PB, there is very poor synergism between the phases. The other two blends have tenacities that follow simple "mixing rule" behavior – i.e., the tenacity properties are roughly intermediate between the tenacities of the pure components.

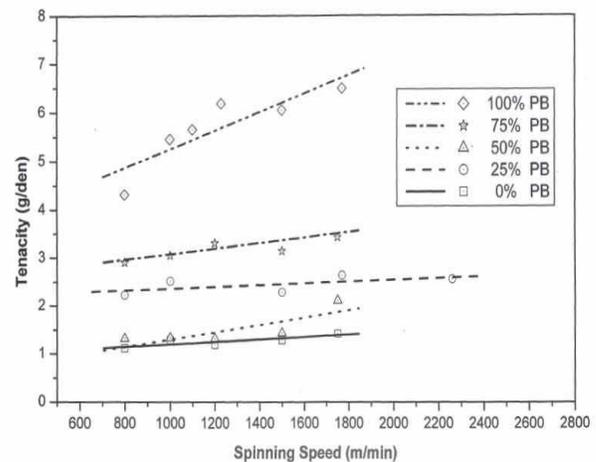


Figure 4. Tenacity as a function of collection speed for fibers produced from blends of PB-1/PP. The fibers were spun using a polymer flowrate of 0.23 g/min; spinning speeds ranged from 800 to 2250 m/min. The final diameters of the fibers ranged from 22.2 microns (at 800 m/min) to 12.6 microns (at 2250 m/min). The lines are linear fits to the data shown on the figure.

Figure 5 shows the  $E_B$  of the fibers. The  $E_B$ , as with tenacity, is sensitive to the interactions of the components in a blend (Robertson, 1973). Fibers with 25, 50 and 75% PB-1 show elongations that lie in between the  $E_B$  values obtained for the pure polymers (PB and PP). The data show that the  $E_B$  of the blends are more comparable to the elongation of pure PB-1, suggesting that, in terms of elongation, even small amounts of PB-1 can significantly reduce the high  $E_B$  behavior of pure PP fibers. As with Fig. 4, the curve for 50% PB does not seem to follow a linear trend based on the concentration of PB. For all three blends, the curves show a steady decrease in  $E_B$  (as spinning speed increases) until a plateau is reached when

spinning speeds exceed about 1800 m/min.

For various blend ratios, *Figure 6* shows the modulus of elasticity of the fibers. In this figure, the modulus is the modulus at zero strain (i.e., the modulus is the slope of the initial segment of the stress-strain curve). In the figure, the data for all concentrations show that the modulus increases linearly with increasing collecting speeds. For the pure components, the modulus curve for the 0% PB (pure PP) is significantly lower than the modulus curve for the 100% PB. Interestingly, the modulus curves for the blends all lie below the modulus curves for either of the pure components.

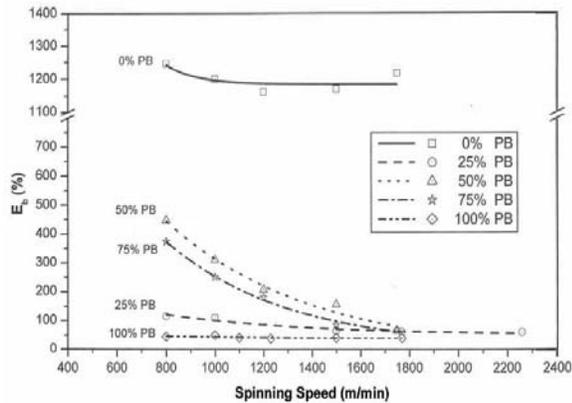


Figure 5. Elongation at break as a function of spinning speed for fibers produced from blends of PB-1/PP. The fibers were spun using a polymer flowrate of 0.23 g/min, and spinning speeds ranged from 800 to 2250 m/min. The diameter of the fibers varied from 22.2 microns (at 800 m/min) to 12.6 microns (at 2250 m/min). The lines are exponential decay fits of the form  $y = a e^{-bx}$ , where  $a$  and  $b$  are fitted constants.

(Keep in mind, however, that zero strain modulus is plotted on *Fig. 6*.) Also, there is little difference between the modulus curves for three different blends of PB.

*Figure 7* shows the birefringence results for fibers spun at a polymer flowrate of 0.23 g/min. Birefringence measurements can help determine the degree of orientation in polymer fibers; high orientation usually corresponds to high strength fibers. Birefringence is defined as the difference between the parallel and the perpendicular refractive indexes of the fiber. Specifically,

$$\Delta n = n_{\text{parallel}} - n_{\text{perpendicular}} \quad (2)$$

where  $\Delta n$  is the birefringence and  $n_{\text{parallel}}$  and  $n_{\text{perpendicular}}$  are, respectively, the birefringence in the parallel and

perpendicular directions to the fiber axis. In practice, fiber birefringence is determined by the equation

$$\Delta n = \frac{\Delta}{d} \quad (3)$$

where  $\Delta$  is the relative retardation in nm, and  $d$  is the fiber diameter in nm. (See Phillips, 1971, for more information on these equations.) The relative retardation was measured using a polarizing microscope and the Sernamont compensator technique.

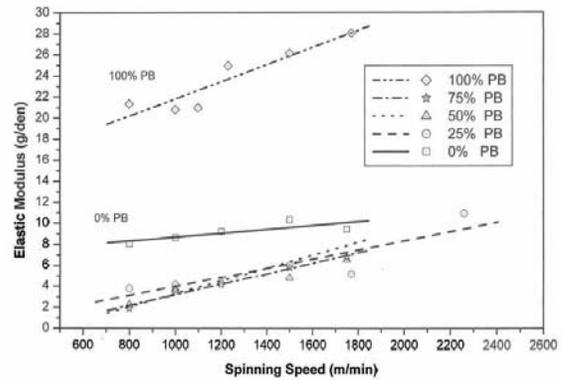


Figure 6. Elastic modulus as a function of spinning speed for fibers produced from blends of PB-1/PP. The fibers were spun using a polymer flowrate of 0.23 g/min, and spinning speeds ranged from 800 to 2250 m/min. The diameter of the fibers varied from 22.2 microns (at 800 m/min) to 12.6 microns (at 2250 m/min). The lines are linear fits of the data shown on the figure.

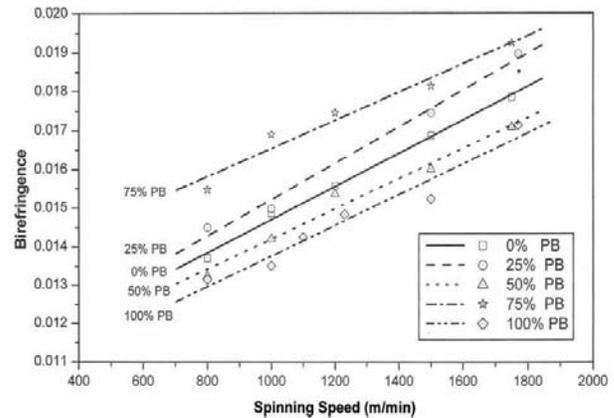


Figure 7. Birefringence as a function of spinning speed for fibers produced from blends of PB-1/PP. The fibers were spun using a polymer flowrate of 0.23 g/min, and spinning speeds ranged from 800 to 2250 m/min. The diameter of the fibers varied from 22.2 microns (at 800 m/min) to 12.6 microns (at 2250 m/min). The lines are linear fits of the data shown on the figure.

The birefringence values for fibers made of pure PB-1 are lower than for the pure PP fibers because in PB-1 the fully oriented fibers have an intrinsic birefringence of 0.02 (Tanaka, 1975), while in fully oriented PP fibers the intrinsic birefringence has a value of 0.03 (Dumbleton, 1968; Samuels, 1972). At a blend composition of 50% PB-1, the fiber birefringence values are in between the values of the two pure polymers. Fibers with compositions of 25% and 75% PB-1 showed birefringence results that are higher than the birefringence of pure PP fibers. Hence, simple linear mixing rules cannot be applied to the prediction of birefringence for blends of PB and PP.

Figures 8 to 11 show the mechanical properties and birefringence as a function of weight percent of PB-1. The trends shown in these figures are not monotonic or simple, which suggests that the blends do not follow a simple mixing rule. Our results agree with Siegmann (1979; 1982) who found the same type of behavior for the mechanical properties of PP/PB blends in thin films; our results contrast with the synergistic results found by Lee (1993) for the blend samples that he prepared by injection molding.

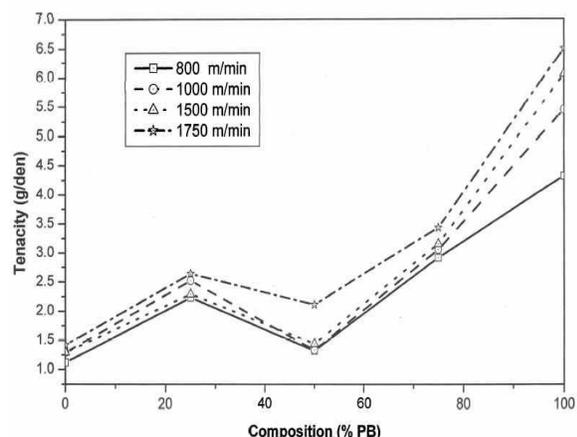


Figure 8. Tenacity of fibers as a function of percent PB-1 in the blend. The fibers were spun using a polymer flowrate of 0.23 g/min. Spinning speeds were 800, 1000, 1500, and 1750 m/min.

### DSC Results

DSC scans were taken to measure the crystallinity of the fibers as a function of blend composition and spinning conditions. The DSC scans also determined the effect of composition on the melting peak temperatures of the fiber samples. Replicates of each sample were run in the DSC within a temperature range from 40 to 180°C at a heating rate of 5°C/min. The fibers used in the DSC measurements were spun at two different polymer flowrates (0.23 and 1.00 g/min) and at a spinning speed of 1000 m/min.

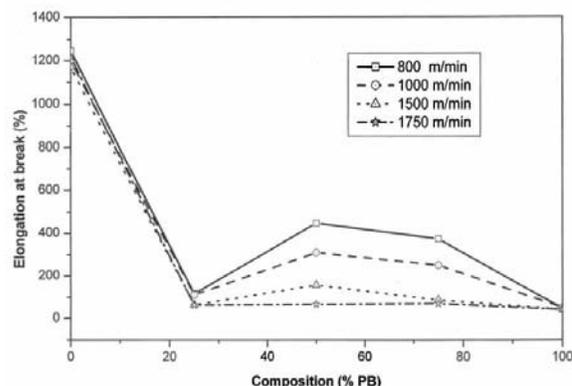


Figure 9. Elongation at break of fibers as a function percent PB-1 in the blend. The fibers were spun using a polymer flowrate of 0.23 g/min. Spinning speeds were 800, 1000, 1500, and 1750 m/min.

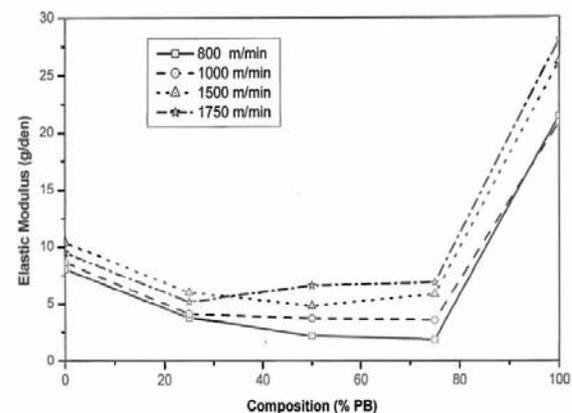


Figure 10. Elastic modulus of fibers as a function of percent PB-1 in the blend. The fibers were spun using a polymer flowrate of 0.23 g/min. Spinning speeds were 800, 1000, 1500, and 1750 m/min.

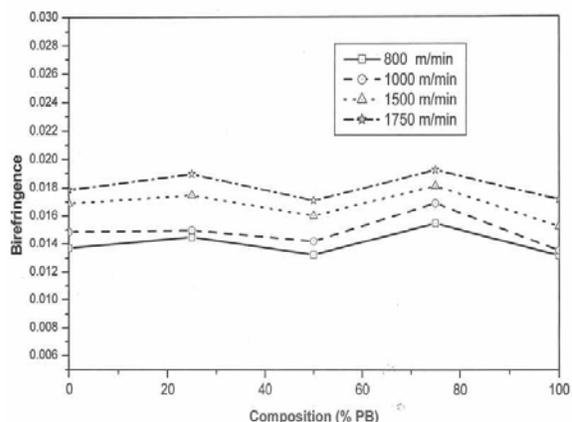


Figure 11. Birefringence of fibers as a function of percent PB-1 in the blend. The fibers were spun using a polymer flowrate of 0.23 g/min. Spinning speeds were 800, 1000, 1500, 1750 m/min. Pure PB-1 fibers spun at flowrates of 0.23, 0.30, 0.50,

and 1.00 g/min were also studied on the DSC for the effect of spinning stress on the melting behavior. The study showed that, by increasing the polymer flowrate used for spinning the fibers, the peak melting temperature of PB-1 decreases. A lower polymer flowrate causes higher stress on the spin line as the fiber is forming and therefore causes a higher melting peak temperature. DSC scans for the highest and lowest polymer flowrates (the extremes) are shown in *Figure 12*. The melting peak that corresponds to the hexagonal form of PB-1 (Form I; see Ortiz and Shambaugh, 2005) shifted from a temperature of 121.9°C for a flowrate of 0.23 g/min to a temperature of 114.1°C for a flowrate of 1.00 g/min.

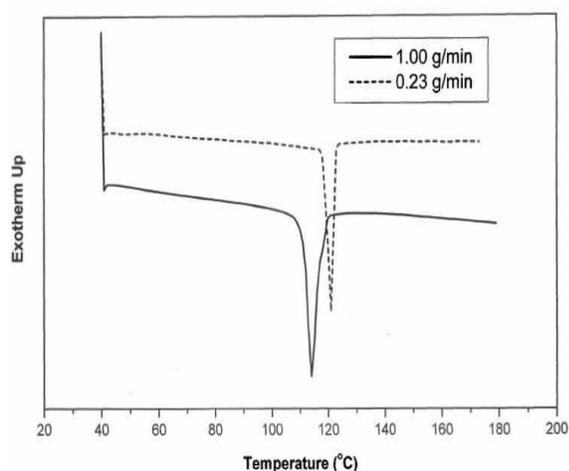


Figure 12. DSC scans of pure PB-1 fibers spun at a spinning speed of 1000 m/min and at polymer flowrates of 0.23 and 1.00 g/min. The DSC heating rate was 5°C/min, and the DSC scan ranged from 40 to 180°C. These DSC scans were first scans.

*Figure 13* shows the crystallinity (left axis) and melting peak temperature (right axis) of PB fibers spun using polymer flowrates of 0.23, 0.30, 0.50, and 1.00 g/min. The peak temperature decreases as polymer flowrate increases; the percent crystallinity remains fairly constant as flowrate increases. Percent crystallinity of the fibers was calculated as the ratio of the heat of fusion of the sample divided by the heat of fusion for 100% crystalline polymer.

The formula is

$$\% \text{ Crystallinity} = \frac{\Delta H_{\text{polymer}}}{\Delta H_{\text{crystalline}}} \cdot 100 \quad (4)$$

where  $\Delta H_{\text{polymer}}$  is the heat of fusion of the sample (as measured by DSC) and  $\Delta H_{\text{crystalline}}$  is the theoretical heat of fusion for a 100% crystalline sample. *Table I* shows

the values of heat of fusion used to calculate the crystallinity of PP and PB in the fiber samples.

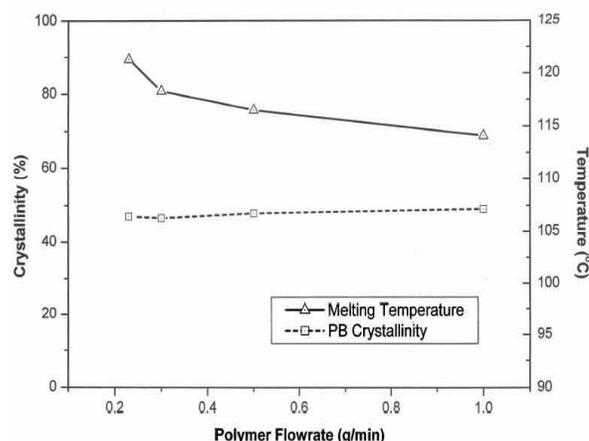


Figure 13. Mid-width peak melting temperature and percent crystallinity of pure PB-1 fibers. The melting temperature were obtained from the DSC scans, and the crystallinities were calculated from the area under the peaks (with equation 4). The DSC heating rate was 5°C/min, and the DSC scan ranged from 40 to 180°C. All DSC scans were first scans.

TABLE I. Parameters used for calculating crystalline percentages in blends of PB-1/PP

Crystal Form	$T_m$ (°C)	$\Delta H_{\text{crystalline}}$ (J/g)	Reference
PB (form I)	125-136	125.5	Choi (1966)
PB (form II)	118-126	71.1	Choi (1966)
PB (form III)	90-193	117.2	Choi (1966)
PP	187	183	Jezl (1988)

*Figure 14* is the DSC scan for fibers made of 25% PB-1. The fibers were spun using a polymer flowrate of 0.23 g/min and a spinning speed of 1000 m/min. The solid line represents the DSC scan as the fiber sample was heated from 40°C to a temperature of 180°C. The dashed line represents the second scan on the same sample (which, because it was melted, is no longer in fiber form) after the pan was cooled down to a temperature of 40°C. On the first scan, only one peak occurred in the range of temperature studied. This one peak, which has a melting temperature of 158.2°C, corresponds to the melting temperature of polypropylene. No other peak is apparent, which suggests a suppression of the crystallinity of PB-1 in the fibers due to interactions with polypropylene. On the second scan, a second peak occurred at a temperature of 94.6°C. This melting temperature

corresponds to either Form I' or Form III; both of these forms have melting temperatures in the range of 90-103 °C (Choi,1966). Form I' and Form III are usually obtained directly from solution; however, other authors have presented DSC results showing that these polymorphs can occur from the melt (Siegmann, 1979; Hsu, 1987). The very similar melting behavior of the two polymorphs makes them hard to individually identify by means of thermal analysis.

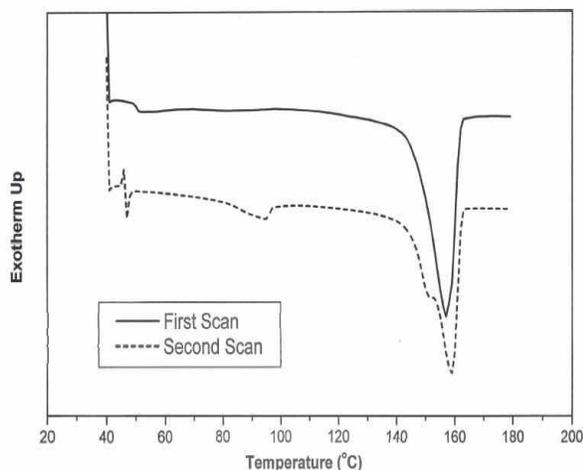


Figure 14. DSC scans of 25% PB-1 fibers spun at a spinning speed of 1000 m/min and a polymer flowrate of 0.23 g/min. The solid line represents the first heating scan from a temperature of 40 to 180°C. After completing this first scan, the pan was quickly cooled from 180 to 40°C (this cooling process took about 8 min). Then, the second scan was initiated after 1 additional min; the dashed line represents the second heating scan on the same sample. The heating rate used was 5°C/min for both scans.

Figure 15 is the thermal scan of 50% PB-1 fibers that were spun using the same conditions as for Fig. 14. In the first scan, the largest peak occurred at a temperature of 157.9°C; this peak corresponds to the melting temperature of polypropylene. A very small peak was detected at a temperature of 92.4°C; this peak corresponds to polybutylene. The second scan in the DSC is shown by the dashed line. As before, this scan was done after cooling the sample to a temperature of 40°C. In this second scan, there were two peaks that occurred in the vicinity of 100°C. The first of these peaks occurred at 95°C, a temperature corresponding to the presence of Form I' or III. The second peak occurred at 103°C, a temperature indicating the presence of Form II. The area under these two peaks (the peaks near 100°C) shows that PB crystallinity was increased by the heating, cooling, and reheating process.

Figure 16 shows the DSC scan for fibers containing

75% weight of PB-1. Again, the biggest peak (at 158.2°C) on the first scan corresponds to polypropylene. A second peak at a temperature of 96.4 °C also occurs; this peak corresponds to Form I' or III of PB-1. A third peak occurs at a temperature of 124.9°C; this peak corresponds to Form I, the higher melting type of PB-1. In spite of the fact the fiber is 75% PB-1, the small areas under the PB-1 peaks show that the PB-1 in this fiber is not highly crystalline. The second DSC scan on the polymer shows a peak at a temperature of 103.6°C, a temperature corresponding to Form II of PB-1. This second scan peak (at 103.6°C) is much larger than the peaks for PB in the first scan. Thus, PB has much higher crystallinity in this second scan of the polymer. This is an indication that polymer interaction is suppressing crystallinity in the fiber form.

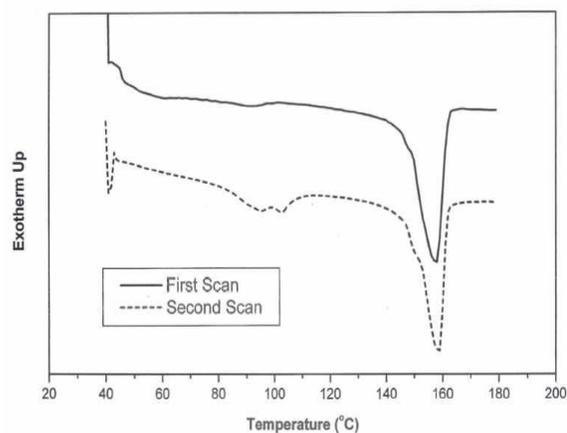


Figure 15. DSC scans of 50% PB-1 fibers spun at a spinning speed of 1000 m/min and a polymer flowrate of 0.23 g/min. The solid line represents the first heating scan from a temperature of 40 to 180°C, while the dashed line represents the second heating scan on the same sample. The heating rate used was 5°C/min for both scans.

For the range of compositions, Figure 17 shows the crystallinity of the PB and PP components in the fibers. It is clear from this figure that the interactions of the components adversely affect the crystallinity of the PB in the fibers. The crystallinity of the PB component decreases substantially as PP concentration increases. The PB crystallinity reaches almost negligible values at a composition of 50% PB. The crystallinity of the polypropylene, on the other hand, remains fairly constant, which indicates that the presence of PB-1 in the blend does not significantly influence the crystallinity of PP. The results are summarized in Table II.

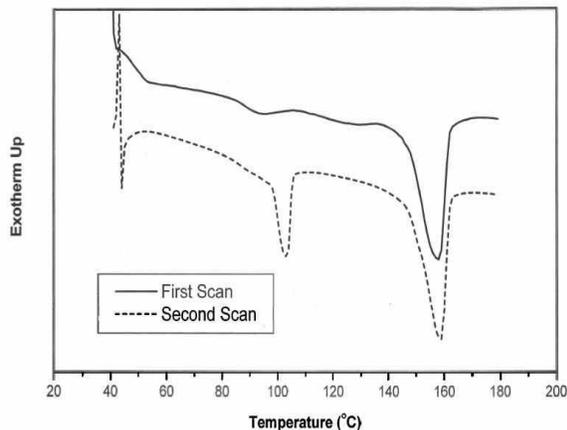


Figure 16. DSC scans of 75% PB-1 fibers spun at a spinning speed of 1000 m/min and a polymer flowrate of 0.23 g/min. The solid line represents the first heating scan from a temperature of 40 to 180°C, while the dashed line represents the second heating scan on the same. The heating rate was 5°C/min for both scans.

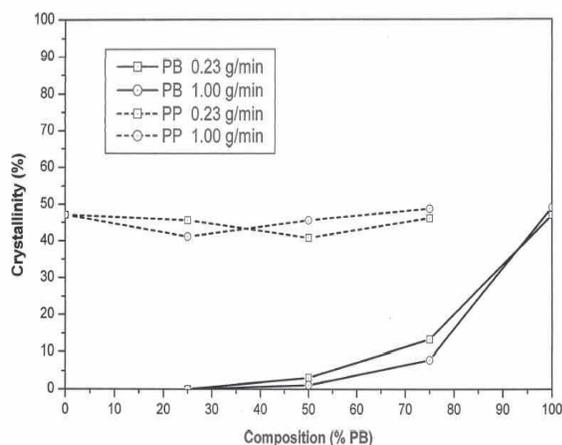


Figure 17. Individual crystallinity of PP and PB-1 in fibers produced from blends of PB-1/PP. The fibers were spun using polymer flowrates of 0.23 and 1.00 g/min; The spinning speed was 1000 m/min. The crystallinity was determined from the DSC scans (all scans were first scans).

TABLE II. Crystallinity of PB-1 and PP obtained by DSC scans of fiber samples. The fibers were spun with a polymer flowrate of either 0.23 or 1.00 g/min, and the spinning speed was 1000 m/min. The DSC scans were all first scans.

Composition (weight % PB)	Percent Crystallinity			
	0.23 g/min		1.00 g/min	
	PB-1	PP	PB-1	PP
0	--	47.1	--	47.0
25	0.0	45.6	0.0	41.1
50	3.1	40.8	1.1	45.6
75	13.3	46.2	7.9	48.8
100	47.0	--	49.1	--

The separate melting peaks (shown in *Figures 14-16*) in the DSC scans of the blends indicate that there is no co-crystallization occurring when the fibers are produced. However, a slight shift in melting temperatures is shown in these scans. For example, the melting peak for polypropylene shifted from a temperature of 164.0°C for pure PP fibers to a temperature of 158.2°C for fibers with 75% composition of PB. This shift could be caused by factors such as a decrease in crystallite size or a change in crystalline order. These findings are in contrast to the results from Hsu (1987); he found no depression of melting temperatures when analyzing ultra-quenched and air-quenched compression molded samples. Our results do agree with Siegmund (1979) who found depression of the  $T_m$  in thin films.

## CONCLUSIONS

Fibers produced from blends of PB-1/PP show mechanical properties that are in between the properties of the pure polymers. The tensile strength of 50% PB-1 fibers is comparable to the tensile strength of pure PP fibers. Fibers produced from blend compositions of 25 and 75% have higher tensile strengths than pure PP fibers, although these blend compositions have lower tensile strengths than pure PB fibers.

In elongation, even small amounts of PB in the blend are sufficient to produce a decrease to values comparable to elongations for pure PB fibers. There is approximately 100% elongation for 25% PB fibers compared to 1200% elongation for pure PP fibers.

The DSC results show no evidence of co-crystallization of PP and PB in the blends. The melting temperatures of both polymers shifted to lower values in the blends, a result possibly caused by a change in the crystallite size.

Fibers at a composition of 25% PB show no PB crystallinity. At a composition of 50% the crystallinity of PB is still quite low. Fibers with 75% PB showed two PB melting peaks corresponding to the stable Form I and possibly Form I' or Form III. The second scans on the DSC suggest that the presence of PP in the blends is depressing the crystallinity of PB as the fiber is forming.

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## Nomenclature

$n_{\text{parallel}}$  = refractive index parallel to the fiber axis  
 $n_{\text{perpendicular}}$  = refractive index perpendicular to the fiber

axis

T= fiber tenacity, g/den

T<sub>c</sub>= crystallization temperature, °C

T<sub>m</sub>= melting peak temperature, °C

### Greek Symbols

Δ= relative retardation, nm

Δn= fiber birefringence

ΔH<sub>sample</sub>= heat of fusion of sample, J/g

ΔH<sub>crystalline</sub>= heat of fusion of 100% crystalline polymer, J/g

ρ= fiber density, g/cm<sup>3</sup>

σ= fiber stress, psi

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