

# Crystalline/Crystalline Polymer Blends: Some Structure-Property Relationships

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## Synopsis

Mechanical properties, morphology, and compatibility of polybutene-1 blended with polypropylene, both crystallizable polymers, are described in the present study. Blends of various compositions were studied using tensile tests, differential scanning calorimetry, wide-angle x-ray diffractometry, and optical microscopy. A discussion on the state of compatibility and structure-property relationships for such blends is presented.

## INTRODUCTION

The importance of polymer blending has recently increased and will continue to do so, as it becomes a prime approach for the preparation of materials with new desirable properties. Such compounds, so-called polyblends, polymeric alloys, multiphase polymeric systems, etc., should provide a relative simple solution to the complex problem of filling the economical and performance gaps that presently exist. There are already such systems of commercial importance. The most interesting among them are the semiengineering materials and stretched polyolefin film fibers. In the latter, for example, blending of polymers allows a reduction in basic costs, enables properties of importance to be maximized, and processability to be improved.<sup>1</sup>

Polyblends are physical mixtures of structurally different polymers which adhere together through the action of secondary bond forces and no covalent bonding between them.<sup>2</sup> The degree of compatibility in such a system, which is one of the major factors in determining the system's final properties, can be described by the size and distribution of segregated phases within the mixture. Major controlling factors include chemical composition, polymer crystal structure and morphology, molecular weights, and processing. While only several important polymer pairs are apparently compatible, most pairs exhibit pronounced incompatibility.<sup>3</sup> A compatible blend exhibits mechanical properties proportional to the ratio of the constituents in the blend, whereas incompatibility leads to a material with very poor mechanical properties.

The morphology of polyblends<sup>4</sup> depends on the arrangement of the phases, whether continuous or discontinuous, and the degree of order in the phases, namely, crystalline or amorphous. In all cases, the importance of the interphase is generally accepted. It should be stressed that the morphology of multiphase polymeric systems has a primary effect on its properties.<sup>5</sup>

One of the definable polyblend classes includes crystallizable alloys, where at least one of the components is a polymer capable to crystallize. Although first it was thought<sup>6</sup> that compatible blends containing crystallinity would be very

rare, several such systems were recently reported, including the following pairs: poly(vinyl chloride)/polycaprolactone (PVC/PCL),<sup>7</sup> poly(vinyl fluoride)/poly(methyl methacrylate) (PVF/PMMA),<sup>8</sup> poly(ethylene terephthalate)/polycarbonate (PET/PC),<sup>9</sup> and various polycaprolactone blends.<sup>10</sup> These systems may be divided into three categories: first, blends which have crystalline interactions between the two polymers; second, blends which have polymer interaction in the amorphous phase only; and third, blends which lack both crystalline and amorphous interactions but still have good mechanical properties. These are said to be mechanically compatible,<sup>11</sup> having some interphase connections but still not compatible to exhibit, for example, a single  $T_g$ .

Polyblends in which both components are crystallizable can form mixed or separate crystals imbedded in a compatible or incompatible amorphous phase. The crystals can be homogeneously dispersed or form various levels of superstructure. The structure of the blend will strongly affect its properties. Some properties of various polyolefin blends have been studied.<sup>1,12</sup> Although such polymers as low- and high-density polyethylene (LPPE, HDPE) and polypropylene are structurally very similar, they do not form a microscopically homogeneous blend. One could think that even if these polymers form a multiphase system, interphase adhesion should suffice to provide good blend properties. However, it has been recently reported<sup>1,12</sup> that at certain composition ranges some properties, such as elongation and impact strength, are very poor; other properties, such as tensile modulus and yield strength, are good and there are even compositions which exhibited positive synergism in their ultimate tensile strength.

To better understand the structure-property relationships in crystalline/crystalline polyblends and to promote the elucidation of the structure of such polyblends, the investigation of blends of two homolog crystalline polyolefins, namely, polypropylene (PP) and polybutene-1 (PB), has been initiated. This pair was chosen for the present study since "PP and PB are highly compatible in all proportions under normal operating conditions."<sup>13</sup> Also, the addition of PP has been found to accelerate the crystalline transformation of PB form I to form II,<sup>14</sup> similar to the effect of propylene as a block comonomer with butylene,<sup>15</sup> though less effectively than propylene as a random copolymer.<sup>16</sup> In addition, partial cocrystallization in PP-PB copolymers has been reported.<sup>15,16</sup>

At this point it is worthwhile to compare some of the properties of PP and PB. As can be seen in Table I,<sup>17</sup> both polymers exhibit two main crystallographic

TABLE I  
Some Properties of PP and PB<sup>17</sup>

| Phase                                     | PP             |                | PB             |                                     |
|---|----------------|----------------|----------------|-------------------------------------|
|   | $\alpha$ Form  | $\beta$ Form   | Type I         | Type II                             |
| $T_g$ , °C                                | 0              |                | -20            |                                     |
| $T_m$ , °C                                | 180            | 150            | 134            | 123                                 |
| Unit cell constants                       |                |                |                |                                     |
| <i>a</i>                                  | 6.65           | 6.36           | 17.7           | 14.85                               |
| <i>b</i>                                  | 20.96          | 6.36           | 17.7           | 14.85                               |
| <i>c</i>                                  | 6.50           | 6.49           | 6.5            | 20.6                                |
| Helix                                     | 3 <sub>1</sub> | 3 <sub>1</sub> | 3 <sub>1</sub> | 11 <sub>3</sub> (40 <sub>11</sub> ) |
| Transverse area per chain, Å <sup>2</sup> | 34             | 35             | 44             | 45                                  |

forms which differ in their unit cell constants and melting temperature, PP being the higher melting material. PB crystallizes from the melt, first into a metastable tetragonal modification (form II), which converts to a rhombohedral stable phase (form I). The rate of conversion depends on such factors as temperature,<sup>18</sup> pressure,<sup>19</sup> orientation,<sup>20</sup> and the presence of certain low and high molecular weight additives.<sup>18</sup> Upon stretching, necking is less pronounced in PB than in PP; and beyond its yield point, PB exhibits an extended strain-hardening region.<sup>21</sup> The creep resistance of PB compared to that of PP is outstanding.<sup>22</sup> Commercially, PB and PP are blended mainly to reduce the brittle temperature and stiffness of PP.

In the present work mechanical properties of PB-PP blends were measured by tensile testing, the structure was studied by x-ray diffraction and thermal analysis, and the morphology by optical microscopy.

## EXPERIMENTAL

The two polymers used in the present study were polypropylene (ICI 1608, GWM-101) and polybutene-1 (Witco Chemical, Witron 0100) in granular form. Blends of various compositions were prepared by mixing predetermined weights of the two polymers in the molten state for 10 min. The mixing took place in a Brabender Plastograph mixing head (model PL 3S), rotating at a speed of 30 rpm, heated to 190°C (ca. 20°C and 55°C above the melting temperature of PP and PB, respectively). The blends were compression molded into 1-mm-thick sheets in a Carver laboratory press at 190°C, followed by air cooling to room temperature. These samples were characterized as follows:

A du Pont 990 thermal analyzer, equipped with a differential scanning calorimeter (DSC) cell, was employed to obtain the melting behavior of the blends. The DSC cell was calibrated at the running conditions using standard materials which melt in the temperature range of interest. The sample weight used in the DSC cell was carefully kept in the 9- to 11-mg range. All samples were heated at a rate of 20°C/min. The relative heat of fusion was calculated from the melting peak area.

Wide-angle x-ray diffraction (WAXD) scans were obtained with a Philips PW 1050 diffractometer including a proportional counter. Crystallinity calculated from diffraction scans obtained in the transmission and reflection modes were comparable.

Tensile properties of the blends were measured at room temperature employing an Instron testing machine. Standard dumbbell-shaped specimens (with a gauge length of 4 in. and a width of 0.5 in.) were cut out from the compression-molded sheets and drawn at a cross-head speed of 0.5 cm/min. Strains were monitored with a strain gauge extensometer. All reported results are the average of five runs.

A Wild M21 optical microscope was used to observe the morphology of molded sheets. A hot stage microfurnace (Mettler FP 52) was installed in the microscope to follow morphological changes upon heating the specimens from room temperature to melting. At present, the attempt to follow the melting of PB and PP phases in the blends, separately, was unsuccessful.

## RESULTS AND DISCUSSION

## Tensile Properties

The common stress-strain behavior of crystalline polyolefins was obtained for PP, PB, and their blends. The major differences between the behavior of the two "pure" polymers are the higher values of modulus and maximum strength for PP and the higher values of elongation, both at yield and break, for PB. The yield point of PB is barely detectable. The stress-strain curves of the blends are all located between those of the two separate polymers, changing gradually with composition. The characteristics of the stress-strain curves are plotted in Figures 1 and 2 as a function of composition. Adding PP to PB increases linearly the system's modulus. Hence, the modulus, which is a low-strain property, of PB/PP blends changes linearly with composition, as if the two components comprising the system were compatible through the whole range of composition. The yield strength values are practically constant for all com-

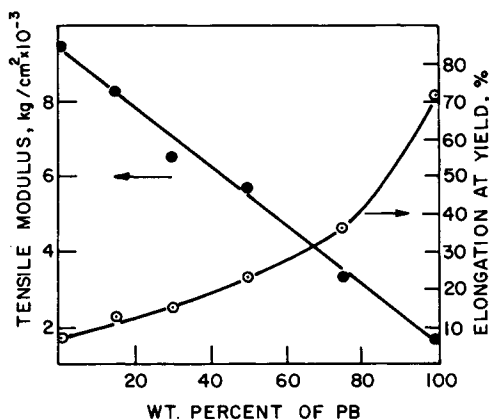


Fig. 1. Composition dependence of tensile modulus and elongation at yield of PB/PP blends.

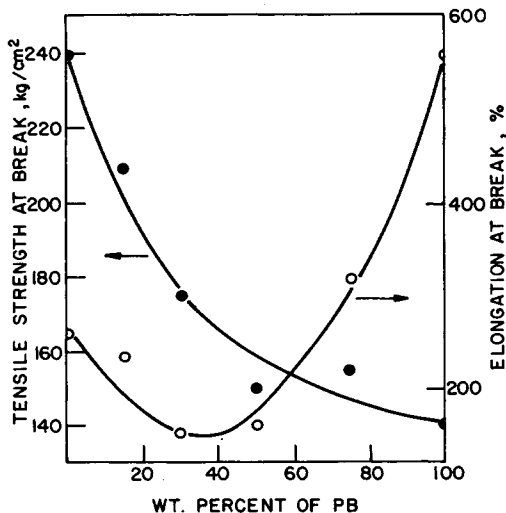


Fig. 2. Composition dependence of tensile strength and elongation at break of PB/PP blends.

positions, whereas the elongation at yield decreases gradually with increasing PP content. At break, the tensile strength, which is for all the studied systems also the ultimate strength, decreases gradually with increasing PB content. However, the elongation at break decreases with increasing the other component content, attaining a minimum at about 40% PB. Technical data for commercial PB/PP blends manufactured by Witco Chemical show gradual changes with composition also in such properties as hardness and brittle temperature.

When two polymers are compatible, the mechanical properties of their blends will be proportional to the ratio of the two in the blended system.<sup>2,3</sup> The tensile properties described above, except elongation at break, do not show any abrupt change with composition; thus, they are evidence for a high degree of PB-PP compatibility throughout the whole range of composition.

No similar data could be found in the literature for comparison. Some mechanical properties of other polyolefin blends have been recently reported. Deanin and Sansone<sup>12</sup> have reported semicompatibility evidences in blends of HDPE/LDPE, HDPE/PP, and LDPE/PP. Modulus and tensile strength were reported as the least sensitive properties, whereas elongation appeared the most sensitive to blend composition and thus to structure. The specific composition dependence of the tensile properties varied among the polymer pairs. Noel and Carley<sup>23</sup> have reported for HPPE/PP blends that most specimens fractured already at elongations less than 15%, while modulus and tensile strength changed gradually with composition. (The sensitivity of polymer elongation to weak points is already well known.) In the present work, it is interesting to note that the elongation is more sensitive to the addition of PP to PB than at the other end of the composition (low PB concentration).

### Thermal Analysis

DSC thermograms of each polymer separately (Fig. 3) show single melting peaks at 172°C and 132°C for PP and PB original pellets, respectively. Compression molding, quenching, and one week of conditioning at room temperature did not affect the melting behavior of PP, whereas PB exhibits two melting peaks,

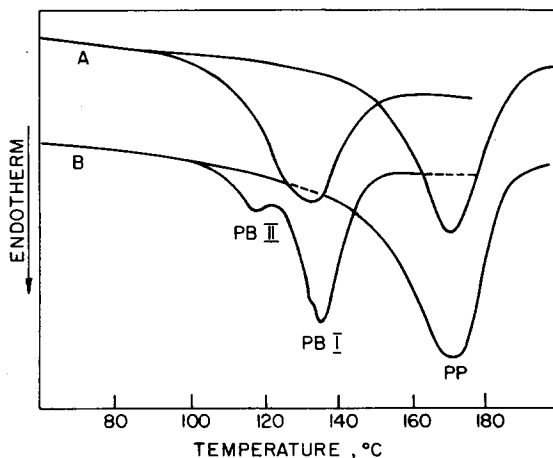


Fig. 3. DSC thermograms of PP and PB graphically superpositioned: (A) original pellets; (B) one week after compression molding and quenching.

at 119°C and 135°C, corresponding to form II and form I, respectively. The endotherms start to deviate from the baseline at rather low temperatures, and consequently the PP and PB melting peaks overlap in a range of about 50°C. Thermograms of PB/PP blends (Fig. 4) show separate melting for the two crystalline species. The peaks also overlap, causing some difficulties in measuring the separate peak areas. Thermograms of blends analyzed immediately after crystallization, Figure 4(b), show melting of PB II and no any PB I, as does the polymer when by itself. Thermograms of blends aged for one week show mainly melting of PB I, but in addition still significant melting of PB II. This behavior is different from that of butene-propylene random copolymers,<sup>15</sup> in which crystallization occurs substantially directly into PB I form. However, in block copolymer, PB crystallizes first as PB II and the transition to PB I is only marginally accelerated compared with the transition rate in butene homopolymer. Boor and Mitchell<sup>14</sup> have reported similar marginal effects when small amounts of PP were added to PB. It should be noted that in the thermograms of 3:1 PP/PB blends, an additional melting peak has appeared at 90–95°C, which probably corresponds to PB III. (Form III has an orthorhombic symmetry,<sup>24</sup> forms usually by precipitation from solution, and has a melting temperature at 100°C.<sup>18</sup>)

A plot of the resulting temperatures (as measured at the tip of the peak) versus composition for PP and PB phases is shown in Figure 5. Some depression of the melting temperature of one polymer associated with the presence of the other component is seen. The main shift is in the PP phase, from 172°C for PP to 164°C in the 1:3 blend. Such melting temperature depression has been reported for other blends, e.g., poly(ethylene terephthalate) (PET) with poly(butylene terephthalate) (PBT),<sup>25</sup> poly(2,6-dimethylphenylene oxide) (PPO) with isotactic polystyrene (iPS),<sup>26</sup> and poly(vinyl chloride) (PVC) with poly-E-caprolactone

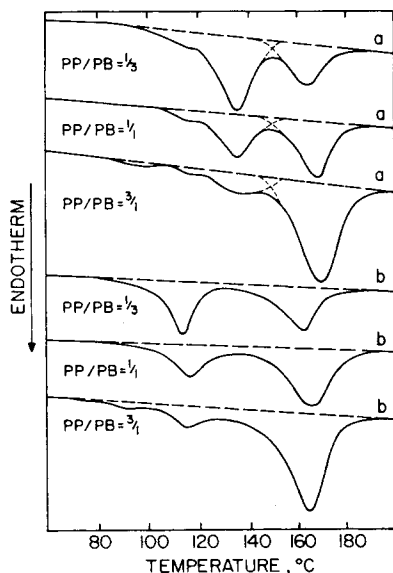


Fig. 4. DSC thermograms of PP/PB blends: (a) one week after compression molding and quenching, (b) immediately after melting and cooling in the DSC cell.

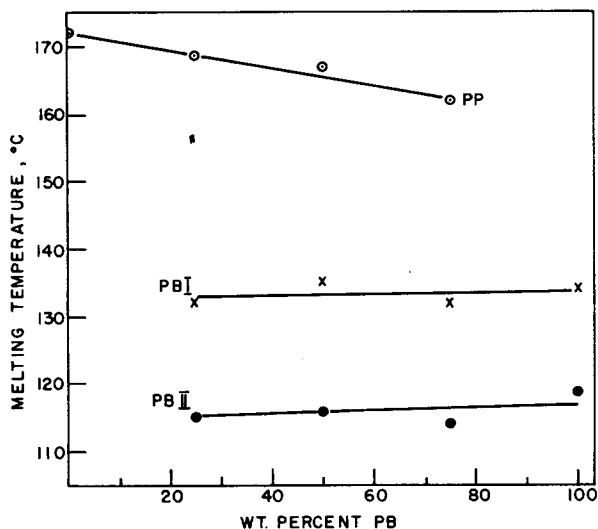


Fig. 5. Variation of PB/PP blends melting temperature, with composition.

(PCL).<sup>10</sup> Melting temperature depression may be the result of several phenomena such as cocrystallization compatibility in the amorphous phase,<sup>27,28</sup> smaller crystallite size, and decreasing crystalline order.<sup>29</sup> The latter appears to be the case for PPO/iPS blends.

In the present system cocrystallization seems not to occur. Both WAXD (discussed later) and DSC show the presence of both polymers crystalline phase, in the compositions studied. In general, cocrystallization is not likely to take place in polyblends because of the very tight requirements that need to be fulfilled simultaneously. The compatibility in the amorphous phase cannot be excluded. Although PP and PB seem to be compatible in the molten state (their melt is transparent), the compatibility in the amorphous solid should still be proved by measuring, for example, their  $T_g$ , a difficult task to be undertaken because of their high degree of crystallinity. The third reason for melting temperature depression, namely, small crystallite size and crystalline disorder, is very possible. However, WAXD data, shown elsewhere, do not clearly support it.

By measuring the areas under the melting peaks, it should be possible to calculate the degree of crystallinity of each component in the blend (not possible by techniques such as WAXD and density measurements). The PB and PP peaks were first graphically separated. The ratios of PP to PB crystallinity in three blends having various thermal histories are summarized in Table II. In blends quenched from melt and conditioned up to one week, the presence of PB is increasingly disturbing the crystallization of PP. The behavior of blends analyzed immediately after cooling in the DSC cell is the other way around, which is probably due to the slow cooling from the melt. It should be pointed out here that PB crystallizes at rather high degrees of supercooling, hence, at relative low rates.<sup>13</sup> The blends degree of crystallinity is discussed in the WAXD section.

TABLE II  
PP Crystallinity/PB Crystallinity in PP/PB Blends (Calculated from DSC Thermograms)

| PP/PB<br>wt ratio | Quenched <sup>a</sup>              |  | 1 Week <sup>b</sup>   |                             | 2nd Run <sup>c</sup>  |                             |
|-------------------|------------------------------------|--|-----------------------|-----------------------------|-----------------------|-----------------------------|
|                   | Peak<br>area<br>ratio <sup>d</sup> | Crystal-<br>linity<br>ratio <sup>e</sup> | Peak<br>area<br>ratio | Crystal-<br>linity<br>ratio | Peak<br>area<br>ratio | Crystal-<br>linity<br>ratio |
| 3:1               | 6:1                                | 1.05:1                                   | 6:1                   | 1.05:1                      | 4:1                   | 0.56:1                      |
| 1:1               | 2:1                                | 1.05:1                                   | 0.83:1                | 0.43:1                      | 17:1                  | 0.71:1                      |
| 1:3               | 0.5:1                              | 0.78:1                                   | 0.43:1                | 0.69:1                      | 1:1                   | 1.26:1                      |

<sup>a</sup> Blend quenched from the melt and analyzed on the same day.

<sup>b</sup> As in (a) and analyzed after one week.

<sup>c</sup> Melted and cooled in the DCS cell followed by analysis.

<sup>d</sup> Measured melting peak areas normalized for samples weight.

<sup>e</sup> As in (d), taking into account composition and diving by heats of fusion,<sup>14</sup>  $\Delta H_f(\text{PP}) = 2.25$  kcal/mole;  $\Delta H_f(\text{PBI}) = 1.58$  kcal/mole;  $\Delta H_f(\text{PBII}) = 1.27$  kcal/mole.

### Wide-Angle X-Ray Diffraction

Wide-angle x-ray diffraction (WAXD) was used to explore the structure of the binary blend, which consists of two crystallizable polymers. Do they form a mixed crystal and, if not, how and to what extent do they mutually affect the crystal structure and the ability to crystallize at each compound?

Figure 6 shows a representative x-ray diffraction scan of a 1:1 PB/PP blend. As for the other two studied compositions, the scan is practically a superposition of PP and PB scans. Figure 7 summarizes the main peaks location and relative intensities. The  $2\theta$  values for PP phases stay constant through the whole range of composition, whereas, for the PB phase, upon the incorporation of 25% PP, all peaks shift to lower  $2\theta$  values and stay constant up to 75% PP (maximum PP concentration studied). Thus, the  $d$ -spacings of PP do not change upon crystallization in the presence of PB, probably still in the melt, while the lattice dimensions of PB are modified upon crystallization in the presence of PP, probably already in a crystalline state. As expected, it can be seen in Figure 7 that the relative intensities change with composition, decreasing with increasing the

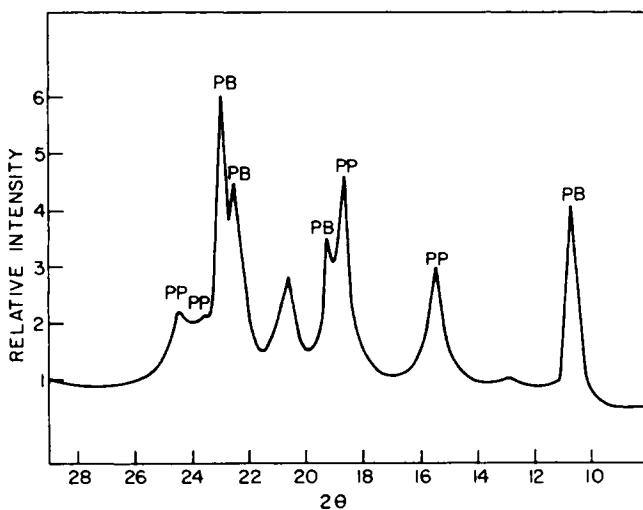


Fig. 6. Wide-angle x-ray diffraction scan of a 1:1 PP/PB blend ( $\text{CoK}\alpha$ ).



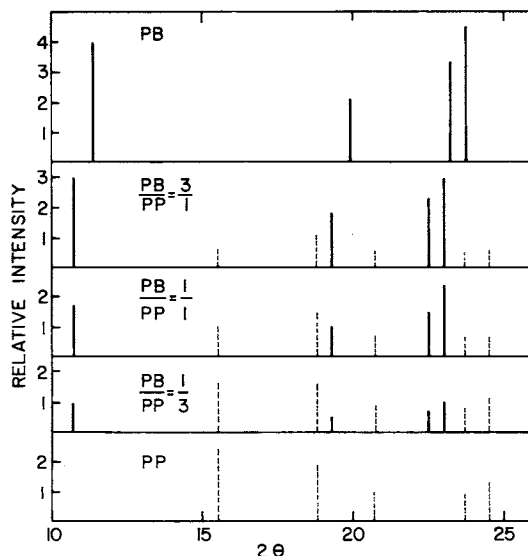


Fig. 7. Relative intensity and location of main WAXD peaks for PB/PP blends.

concentration of the other phase. Plotting the relative intensities of the main peaks as a function of blend composition (not shown) results in a family of straight lines, all of which extrapolate to zero intensity at zero concentration. Thus, the relative intensities change linearly with composition, as if each phase crystallizes completely separate. It should also be mentioned that the peak widths practically do not change with composition, hence it can be interpreted as no change in crystalline size.

The blends' total degree of crystallinity was determined by dividing the area under the peaks into amorphous and crystalline areas, with no further corrections. The ratio of the crystalline area to the total area was taken as the degree of crystallinity. The data so obtained are not absolute but the use of more absolute methods was ruled out by variations among the blend compositions, phases, etc. The total degree of crystallinity (including both phases) dropped gradually from 78% for PB to 64% for blend containing 75% PP, followed by an increase to 67% for PP (see Fig. 8). The total crystallinity attained is lower than the linearly added contributions of the compounds. Thus, the presence of two components in the system hinders to some extent the crystallization processes of both phases. The mutual hindrance is not symmetrical, namely, the molten PB affects the degree of order attained by PP stronger than the effect of already crystalline PP on the later crystallization of PB.

### Morphology Studies

To observe the morphology of PB/PP blends in a transmission optical microscope, 1-mil-thick films were prepared by compression molding of blends (mixed in the melt), quenching to room temperature, remelting with a free surface on a hot plate, followed by slow cooling back to ambient temperature. The specimens were observed in the microscope after one week of conditioning. As can be seen in Figures 9(a) and 9(g), PB and PP crystallize from the melt in the

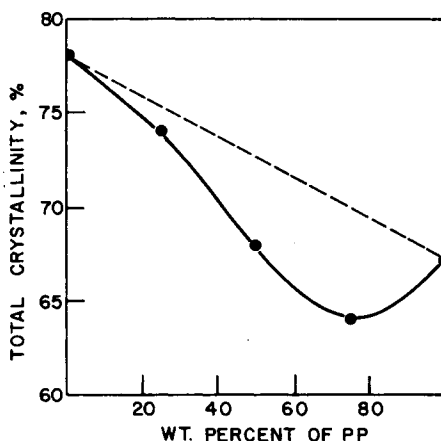


Fig. 8. X-ray total crystallinity of PB/PP blends.

common spherulitic structure; generally PB forms larger spherulites. Adding 25% PP to PB (PB/PP = 3:1) results in a solid comprised of well-developed spherulites resembling the PP morphology, although less perfect, having coarser fine structure. In the 1:1 blend, no spherulitic morphology could be observed. Instead, the solid blend consists of a mixture of small crystalline aggregates of the two components. Figure 9(d) shows part of the specimen in which the structure is highly branched, and Figure 9(e) shows another part of the specimen which consists of a dispersion of a rod-like structure. When cross-polarized light was employed, Figure 9(c), the characteristic dispersion of small crystalline species in the 1:1 blend was clearly observed. When a larger amount of PP is added to PB, specifically 75% PP (PB/PP = 1:3), a very irregular or fragmented spherulitic structure is observed, Figure 9(f). Hence, similar to the degree of crystallinity data, it was also observed here that the presence of molten PB affects the crystallization of PP stronger than the presence of already crystalline PP in the PB case, at the same second component content; compare Figures 9(b) and 9(f). In the intermediate composition range there is a strong mutual disturbance preventing spherulitic morphology to develop.

The observations are generally in agreement with the work of Stein et al.<sup>25</sup> in which PET/PBT blends were studied. They have observed spherulitic morphology appearing to become less perfect and more open in structure with the increase of second component concentration, similar in appearance to those observed for iPS/aPS and iPP/aPP blends.<sup>30</sup> With larger amounts of the second component, they have also observed a nonspherulitic morphology. (They are using the term "second" or "other" compound without differentiating which is the "first," assuming that both polymers are capable of crystallization at low degree of supercooling; hence, they can crystallize simultaneously.) They<sup>25</sup> have also examined the crystallized blends using small-angle light scattering (SALS) and have observed that the addition of a second component in increasing amounts results in SALS patterns characteristic of a greater degree of spherulitic disorder and/or the presence of randomly oriented material within the spherulites. At still larger PET content, they have observed the loss of azimuthal dependence, which may arise from uniform dispersion of anisotropic crystals of the two components. Similar observations have been recently reported for PVC/PCL and CAB/PCL blends.<sup>10</sup>

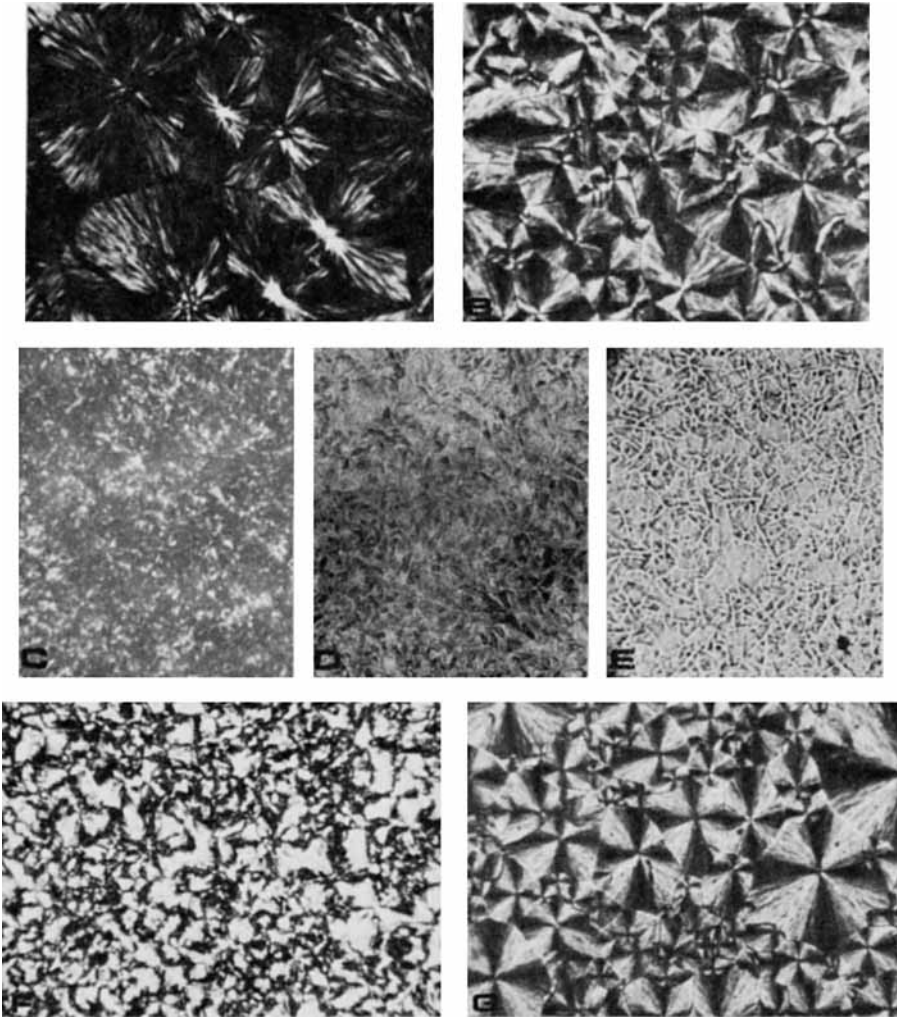


Fig. 9. Optical micrographs of PB/PP blends of various compositions containing: (a) 100% PB; (b) 25% PP; (c, d, e) 50% PP; (f) 75% PP; (g) 100% PP; (a), (b), (c), (f), and (g) cross-polarized light;  $\times 240$ .

Stein et al.<sup>25</sup> have suggested four basic morphologies for blends where both components can crystallize, namely, crystals of both components imbedded in an amorphous matrix; spherulites of one component and crystals of the other; volume-filling spherulites of both components; and volume-filling spherulites of one component within which crystals of the other component are dispersed. The type of expected morphology should depend on such factors as melting temperatures, diffusion rates, crystallization rates, etc.

In the presently studied system, there is quite a large difference in the melting temperature of the two polymers (ca. 30–40°C). Thus, it is highly probable that PP should be the first to crystallize out of the molten blend, except when it exists at very low concentration. At moderate and high PP concentration the Keith and Padden model<sup>30</sup> should be applicable, namely, during PP crystallization PB molecules are being segregated, followed by crystallization within and/or next

to PP spherulites, depending on such factors and PB concentration, crystallization, and diffusion rates. The rejected material should have a retarding effect on both the kinetics of bulk crystallization<sup>31</sup> and spherulitic growth.<sup>30</sup>

### SUMMARY

The mutual influence of two highly crystallizable polymers, namely, PB and PP, crystallized from a molten blend has been systematically studied. In general, PP and PB blends form a solid, the properties of which are evidence of various degrees of mutual influence between the two components.

Tensile mechanical properties show in general quite a high degree of PB-PP compatibility, which is probably the justification for the common quote, especially in technical literature, that "PB and PP are practically compatible throughout the whole range of composition." However, microstructure analyses prove the existence of at least two phase structure representing the two components comprising the blends. The adhesion between the two phases, probably through a highly compatible amorphous phase, is strong enough to result in a blend having good mechanical properties.

The presence of one component exerts a considerable influence on the formation process and final structure of the other one, and consequently the following behavior results: the melting temperature of PP is significantly depressed; the total degree of crystallinity, as measured by WAXD, changes more than expected by plain additivity; the PB/PP crystallinity ratio, as obtained from DSC, does not change linearly with composition; the crystallographic parameters of PB are modified; and the morphology changes from spherulitic, through coarser and fragmented spherulitic, to a homogeneous dispersion of branched crystallites.

Furthermore, in light of the results, it appears that PP is the component which starts to crystallize out of the molten blend first, and consequently its crystallization process and structure are quite strongly affected by the presence of still molten PB molecules, whereas the latter crystallizes from a melt comprised of mainly PB, however, in the presence of PP crystals. This sequence of events causes the following: melting temperature of PP and only slightly of PB are depressed; crystallographic parameters of PP are not modified, whereas the *d*-spacings of PB are all shifted to larger values; the total degree of crystallinity and overall morphology are more affected by the other component at high PP content than in the other composition end. Hence, the "disturbance" introduced by small amounts (probably up to 25%) of molten PB to a crystallizing PP is stronger than that introduced by small amounts of PP crystals to a crystallizing PB. At the middle composition range the "disturbance" is such that the crystallization processes of both polymers occur probably simultaneously or close to it.

As already mentioned, the results presented above show various degrees of mutual influence in PP/PB blends. Two extremes should be pointed out. On the one hand, tensile modulus changes linearly with composition as expected from a compatible blend. On the other hand, the WAXD relative intensities of the main peaks of both polymers change linearly with composition, extrapolating to zero intensity at zero concentration, as expected from a system in which the two components were first crystallized and only then blended.

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