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Diblock copolymer compatibilizers for blends of isotactic polystyrene and isotactic polypropylene

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The compatibilizing effect of 50/50 diblock isotactic polystyrene–isotactic polypropylene (iPS-*b*-iPP), prepared from a sequential block copolymerization of styrene and propylene by using a $NdCl_3$-modified Ziegler–Natta catalyst, was examined in isotactic polystyrene (iPS)–isotactic polypropylene (iPP) blends. As investigated by differential scanning calorimetry (d.s.c.), dynamic mechanical thermal analysis (d.m.t.a.) and scanning electron microscopy (SEM), the thermal properties, dynamic mechanical properties and observed morphologies of the iPS/iPP blend pairs unambiguously support the interfacial activity of the iPS-*b*-iPP diblock copolymer as both that of a dispersant and an 'anchoring' compatibilizer. The addition of less than 10% of this diblock copolymer to an iPS/iPP blend has a large compatibilizing effect on its morphology and properties, showing the importance of the adhesion between the components of a non-compatible blend. Not only is the phase size dramatically reduced but also the phase dispersion and the interphase interactions are significantly increased when the compatibilizer is added. Improvements in the mechanical properties of iPS/iPP blends containing compatibilizers have been noted. The addition of the diblock copolymer significantly promotes an enhancement of the tensile strength, elongation at break and Izod impact strength of the iPS/iPP blends. The mechanical properties can be explained by the compatibilizing and anchoring effects of individual diblock copolymer segments with the blend components, which retards mechanically induced slippage in the immiscible iPS/iPP binary blends.

(Keywords: styrene–propylene copolymer; compatibilization; blend)

INTRODUCTION

The field of polymer blends is developing rapidly, both in terms of scientific understanding and in terms of commercial utility. Blending of polymers has been employed to produce materials with special properties for various applications at lower cost than that of new polymer developments, to improve the performance of polymeric materials, and to enable the reuse of recycled industrial and/or municipal plastics waste[1,2]. However, most polyblends are generally immiscible due to the very low entropy of mixing[3], thus exhibiting poor mechanical properties for both processing and end use. The physicomechanical properties of blends are not only a function of the blend composition but also depend crucially on the degree of dispersion, phase size and phase interaction between the components of the blend. Control of morphology is therefore the control of polymer blend properties[4]. Among the parameters controlling the morphology of immiscible blends are the interfacial tension[5,6], the nature of interactions or interfacial adhesion[5,7], viscoelastic properties of each component of the blend, the thermal history, blending procedure, and the characteristics (composition and molecular weight) of the polymers[4,8-10]. Making interpenetrating networks, adding block copolymers, enhancing *in situ* coupling reactions and/or grafting of one of the components, crosslinking, and mixing polymers containing similar monomer units[11-17] could all be expected to be possible solutions for ways of reducing the interfacial tension and improving the phase adhesion between two immiscible phases.

The use of block copolymers in polymer blending technology is diverse and of increasing importance. The block copolymers are capable of acting as interfacial agents in polymer blends, thus modifying the homopolymer mechanical properties[18,19] (e.g. increase the toughness) and compatibilizing homopolymer blends by enhancing interface adhesion and stabilizing phase dispersion against coalescence[20-22]. In the past a considerable amount of theoretical and experimental work has been carried out to elucidate the phase behaviour and properties of block copolymer/homopolymer blends[23-25], and to understand the importance of various molecular parameters which determine their action in binary or ternary blends[26,27]. As previously reported in the literature, some of these block copolymers act as emulsifiers[5,27-29], while others 'anchor' together blend partners by the athermal mixing/dissolution of homopolymer in the chemically identical block[5,22,30,31], or by mixing through an enthalpic/specific interaction with a 'complimentary dissimilar' block[20,31-33]. Recently, there exists increasing interest in the effect of block copolymer architecture on blend miscibility or compatibilization [5,22,28,34], Riess *et al.*[35] first reported that well defined diblock copolymers

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were more effective than triblock copolymers in dispersing two homopolymers. This behaviour is also shown by the experimental work of Sjoerdsma *et al.*[36]. The findings of Heikens and coworkers[10,24,36] indicated that a tapered diblock and/or pure diblock copolymer is more efficient than a graft copolymer in compatibilizing low-density polyethylene (LDPE)/PS blends, as both the tapered and the pure diblock copolymers enhance the phase dispersion and the interphase interactions in the blends. Furthermore, Teyssie and coworkers[34] suggested that a tapered diblock copolymer was the best compatibilizer for LDPE/PS blends when compared with a pure diblock copolymer. In addition, Jiang *et al.*[37,38] supported this conclusion by experimental work which indicated that the miscibility of polyisoprene (PI) in PI–PS block copolymers decreased in the following order: tapered diblock > pure diblock > triblock > four-arm star.

Recently, strong interest has been generated regarding polyolefin engineering materials with enhanced thermal and mechanical properties. Isotactic polypropylene (iPP) has had an important position among synthetic polymers. Blends of iPP and isotactic polystyrene (iPS) with excellent thermal properties and rigidity could be regarded as a new form of polyolefin engineering material[39]. Up to now, few studies of compatibilized blends of the immiscible semicrystalline isotactic polystyrene (iPS) and isotactic polypropylene (iPP) have been reported[40], perhaps due to the difficulty in synthesizing a semicrystalline/semicrystalline block copolymer (iPS-*b*-iPP) as a compatibilizer by using conventional Ziegler–Natta catalysts. We have synthesized in our laboratory iPS-*b*-iPP diblock copolymers with different iPS contents, controlled by the experimental conditions, by using a $NdCl_3$-modified Ziegler–Natta catalyst [41,42]. In previous communications[39,43] the effect of a iPS-*b*-iPP(40/60) block copolymer on the mechanical and morphological behaviour of iPS/iPP (40/60) binary blends was discussed. In this present paper we have further investigated the compatibilizing effect of a well defined iPS-*b*-iPP(50/50) diblock copolymer with a crystalline isotactic structure in each block on iPS-*b*-iPP/iPS/iPP ternary blends.

EXPERIMENTAL

Materials

The styrene/propylene block copolymer was synthesized by sequential copolymerization of styrene and propene using a $NdCl_3$-modified Ziegler–Natta catalyst which had been developed in our laboratory[41,42]. After fractionation from unwanted homopolymer species in the reaction mixture, the copolymer was proved to be a diblock copolymer consisting of isotactic polystyrene and isotactic polypropylene segments (iPS-*b*-iPP) after being subjected to extensive molecular and morphological characterization. The diblock copolymer used in this study contained 50 wt% iPS as determined by ^{13}C n.m.r. and elemental analysis. Gel permeation chromatography experiments gave a copolymer molecular weight of 295 000 g mol^{-1}.

Isotactic polypropylene (iPP, isotactic index $\geqslant$ 98%, $M_w = 300\,000$) and isotactic polystyrene (iPS, isotactic index $\geqslant$ 99%, $M_w = 320\,000$) were prepared in a laboratory-scale reactor by using similar polymerization conditions to that used for the iPS-*b*-iPP diblock copolymer.

*Preparation of binary and ternary blends of iPS-*b*-iPP, iPS and iPS*

Both the binary and ternary blends were prepared by completely dissolving all polymers and an antioxidant into *o*-dicholorobenzene at 170°C, and then using an acetone/methanol (1/1) mixture as precipitant. After thoroughly washing and drying under vacuum at 60°C for 10 h, the precipitated powders were compression-moulded at 300°C into sheets or plates which were suitable for cutting specimens for mechanical testing and morphology studies.

Measurements and characterization of blends

The glass transition point (T_g) and heat capacity (ΔC_p) were measured by differential scanning calorimetry (d.s.c.) (Pekin-Elmer, DSC-2) over the range −100 to 150°C, at a heating rate of 20°C min^{-1}.

The dynamic mechanical properties were determined by using a Rheovibron DDV-HEA dynamic mechanical thermal analyser. Measurements were made at an operating frequency of 100 Hz and a heating rate of 2°C min^{-1}. The measurement temperature of the standard sample covered the range from liquid nitrogen temperature to +170°C.

The morphology of the polymer blends was investigated by using electron microscopy. Fracture surfaces were prepared at either room or liquid nitrogen temperature, and observed by a scanning electron microscope (Hitachi S-570).

Tensile properties, Young's modulus and Izod impact strength of the compression-moulded specimens were measured by the standard procedures described in ASTM D638, D790, and D256, respectively.

RESULTS AND DISCUSSION

Modification of the thermal behaviour of polymer blends, particularly in the temperature change and curve breadths of various phase transitions are often used to show changes in their morphology and miscibility[1,2]. In partially miscibile multiphase polymer blends, useful information about phase equilibrium can be obtained from thermal property measurements such as T_g, or the heat capacity change (ΔC_p) at T_g[44,45]. *Figure 1* shows the d.s.c. thermograms of various iPS/iPS-*b*-iPP/iPP ternary blends. As shown in the figure, two separate T_g values, for the iPS-rich phase (T_{g1}) and the iPP-rich phase (T_{g2}) were observed. The addition of the iPS-*b*-iPP diblock copolymer has a significant effect on the T_gs and the ΔC_ps at T_g of the blends. These results (from *Figure 1*) are summarized in *Figure 2*. It can be seen that the heat capacity change ΔC_{p2} per g of iPP at T_{g2} gradually increases, while the corresponding ΔC_{p1} at T_{g1} decreases (after first increasing) as the content of iPP homopolymer in the blend increases. In addition, from *Figure 2* can be seen a large drop in T_{g1} and a slight increase in the T_{g2} of iPP. These results suggest the occurrence of mutual dissolution of iPS and iPP segments of the homopolymers with the iPS-*b*-iPP block copolymer in the blends. On the other hand, the fact of a large drop in T_{g1} and an increase of ΔC_{p1} at a iPS/iPP ratio of 88/12 (relative to pure iPS) also indicate a partial miscibility or dissolution of the iPS-*b*-iPP diblock copolymer with iPP homopolymer in the iPS-rich phase.

Dynamic mechanical thermal analysis (d.m.t.a.) is often more sensitive than d.s.c. in closely reflecting the phase behaviour and in identifying the miscibility or compatibility of blend components[21,36,45]. *Figure 3* shows the effect of iPS-*b*-iPP diblock copolymer on the shear modulus E' of 30/70 iPS/iPP blends. In the low-temperature region ($T \leqslant -10°C$), addition of the diblock copolymer leads to an increase in E' for concentrations up to 5%. The shear modulus starts to decease for the 10% modified blend but remains higher than that of the unmodified blend. For temperatures higher than −10°C, a cross-over temperature at around 10°C is observed in the modified blend. From *Figure 3* it can be seen that E' decreases sharply at −8 and 98°C, temperatures which correspond, respectively, to the glass transition temperatures of the iPP and iPS components. In the case of the 70/30 iPS/iPP blends, the effect on E' of adding the diblock copolymer are reported in *Figure 4*. At a level of 5% of added diblock copolymer, the modulus (E') increases, while at the 10% level, E' decreases slightly but is always equal to the values obtained for the 5% added copolymer.

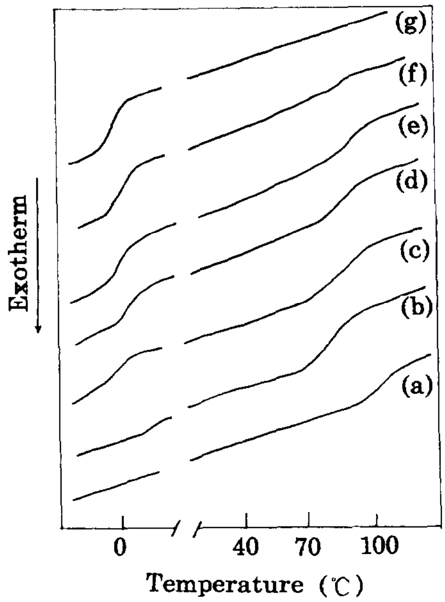

Figure 1 D.s.c. thermograms of iPS-*b*-iPP/iPS/iPP ternary blends: (a) 10/100/0; (b) 10/88/12; (c) 10/70/30; (d) 10/50/50; (e) 10/30/70; (f) 10/12/88; (g) 10/0/100

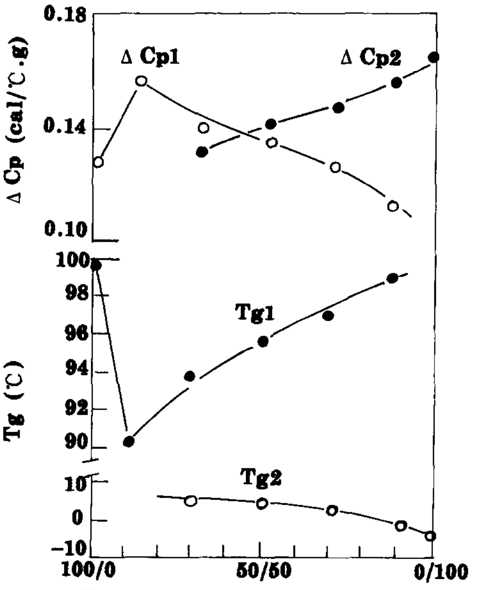

Figure 2 ΔC_p and T_g's data obtained for ternary blends of iPS and iPP after the addition of 10 wt% of iPS-*b*-iPP diblock copolymer

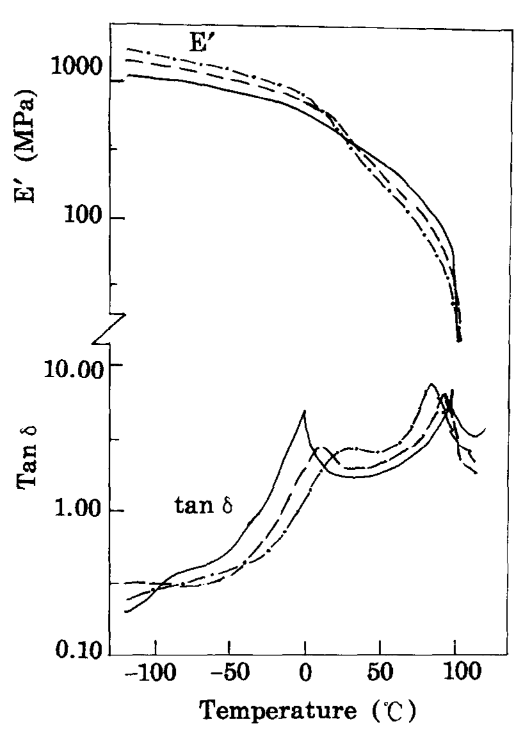

Figure 3 Temperature dependence of the shear modulus (E') and the loss tangent (tan δ) of a 30/70 iPS/iPP blend containing different amounts of diblock copolymer: (a) 0%; (b) 5%; (c) 10%

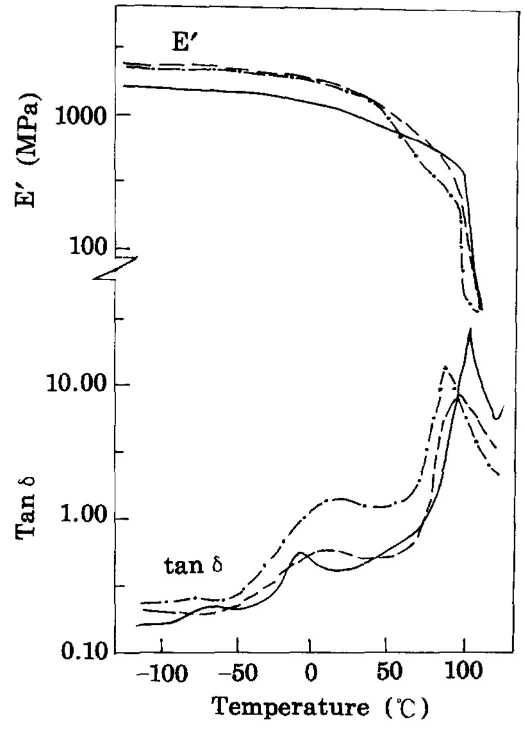

Figure 4 Temperature dependence of the shear modulus (E') and the loss tangent (tan δ) of a 70/30 iPS/iPP blend containing different amounts of diblock copolymer: (a) 0%; (b) 5%; (c) 10%

As shown in *Figures 3* and *4*, the fact that E' varies with the addition of diblock copolymer in both the 30/70 and 70/30 iPS/iPP blend systems suggests that interactions between the two phases are present, which are promoted by the diblock copolymer. These interactions may be attributed to the partition of copolymer micelles between the iPS and iPP phases. In addition, the fact that E' decreases for the blend containing 10% copolymer indicates that the interfacial area is saturated for concentrations up to 10% of added diblock copolymer. However, the precise range of interface saturation concentration needs to be studied in future work.

To better understand the possible interaction between the iPS and iPP phases after addition of the diblock copolymer, we investigated the effect of the diblock copolymer on E' of the pure iPP and iPS components. The effect of the diblock copolymer on E' of pure iPP is shown on *Figure 5*. It was found that at a level of 5% of added diblock copolymer E' of the modified iPP was slightly below that of the pure iPP. For a level of 10% of added diblock copolymer, E' increases to values which are even higher than that of the pure iPP. A similar effect concerning the iPS-*b*-iPP/iPS blends can be observed from examination of *Figure 6*. An increase of E' is observed for 5% of added copolymer whereas a slight decrease is observed for the 10% system. However, E' of the modified iPS even at a level of 10% added copolymer remains greater than that of the pure iPS. In addition, the loss tangent (tanδ) of the homopolymer is found to be affected by the addition of the diblock copolymer. The diblock copolymer also slightly modifies the glass transitions, as shown in *Figures 5* and *6*. This indicates that interactions between iPS-*b*-iPP and both of the iPP and iPS components are possible. Similar effects were also observed in HDPE/high impact polystyrene (HIPS)/(HPB)-*b*-PS blends[21,36]. Therefore, the addition of a iPS-*b*-iPP diblock copolymer added can enhance interphase interactions between the iPS and iPP phases.

The variation of loss tangent (tan δ) *versus* temperature for different copolymer concentrations in both of the 30/70 or 70/30 iPS/iPP blend systems are shown in *Figures 3* and *4*, respectively. The unmodified blend shows two distinct transitions (i.e. two tan δ peaks) for both of the blend systems. The lower one is due to the glass transition (T_g) of iPP while the peak at 98°C is due to the T_g of iPS. However, addition of diblock copolymer can modify the transition temperatures in both blend systems. After adding the diblock copolymer, the lower transition peak increases slightly whereas the higher one gradually decreases. As the added diblock copolymer content increases, the glass transition peaks (tan δ peaks) of the two individual components shift progressively toward each other, and the peaks become broader. Those results confirm that there exists partial miscibility or compatibility of the diblock copolymer with each component of the blend in the iPS/iPP blends examined. These results also suggest that this miscibility or compatibility may occur at the interphase between the components of the blend, thus allowing for enhanced interphase interactions.

The effect of the diblock copolymer on the morphology of the 30/70 iPS/iPP blends is shown in the scanning electron micrographs (obtained by fracturing specimens in liquid nitrogen) in *Figure 7*. The fracture surface shows that isotactic polystyrene particles are embedded in the iPP matrix, and slippage between the two dissimilar components can be observed (*Figure 7a*). The smooth surface of the unmodified iPS/iPP blend is seen on the

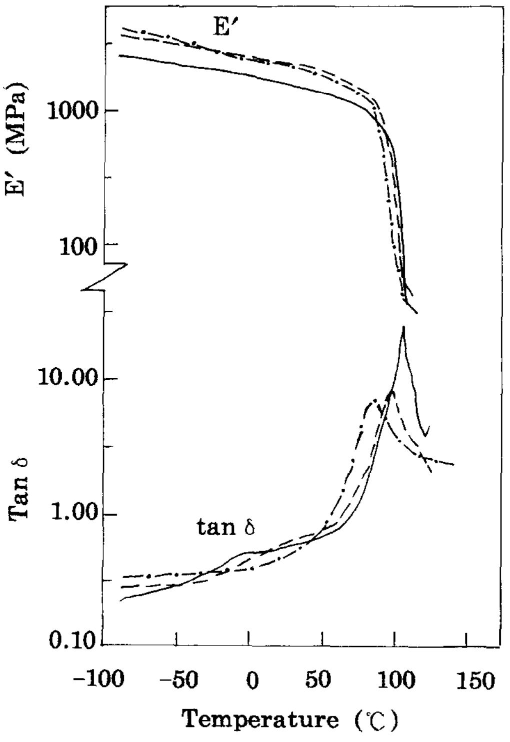

Figure 5 Temperature dependence of the shear modulus (E') and the loss tangent (tan δ) of pure iPS on different amounts of added diblock copolymer: (a) 0%; (b) 5%; (c) 10%

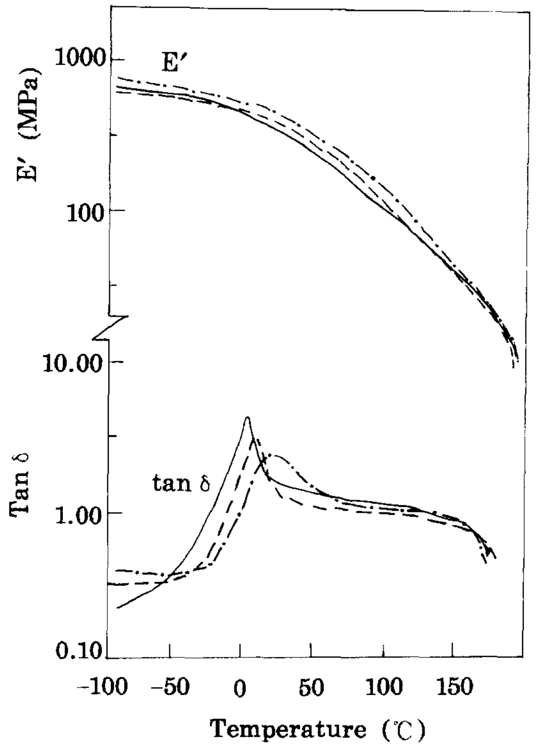

Figure 6 Temperature dependence of the shear modulus (E') and the loss tangent (tan δ) of pure iPP on different amounts of added diblock copolymer: (a) 0%; (b) 5%; (c) 10%

larger protrusions, plus craters formed during the fracturing process when copolymer is absent, indicating little or no adhesion between iPS and iPP. After adding the diblock copolymer the surface morphologies of the modified blends show different features, i.e. a pattern of an ever smaller (i.e. finer) scale (or roughness) is observed as the diblock copolymer content increases (*Figures 7b* and *c*). The diblock copolymer leads to an enhanced polystyrene phase dispersion. The increase of the number of finer particles in the blend, due to the presence of the copolymer, results in an increase of the interfacial area, thus allowing for a better stress transfer. Even at higher magnifications the surface morphology of the iPS/iPP blend containing 10% of the diblock copolymer reveals no slippage occurring during the fracturing process (*Figure 8*). The morphology suggests that the diblock copolymer appears to span the interfaces between regions of iPS and iPP, thus enhancing adhesion and compatibilization of the blend. Similar observations have been made by Cohen and coworkers in an identical blend system[40].

The decrease of particle size of the dispersed polymer upon adding the diblock copolymer in the 70/30 iPS/iPP blend system was also substantiated by scanning electron microscopy of fracture surfaces (*Figure 9*). Note that also in this case, the fracture surfaces show the presence of the diblock copolymer by the phenomenon of adhesion when the diblock copolymer is added, and smooth clean surfaces of matrix and dispersed spheres when the copolymer is absent. It can be seen on the micrographs of the modified blends that the phase dimensions become smaller and smaller, and cannot readily be distinguished from each other as the diblock copolymer content increases. In addition, the intricate formation of a fine semicontinuous phase upon adding the copolymer can be seen in the micrograph of the impact fracture surface of the 70/30 iPS/iPP blend shown in *Figure 10*. The decreasing of the characteristic dimensions of the two semicontinuous phases in the presence of diblock copolymer can be observed by comparing the two pictures. In addition, SEM observation of the impact fracture surface

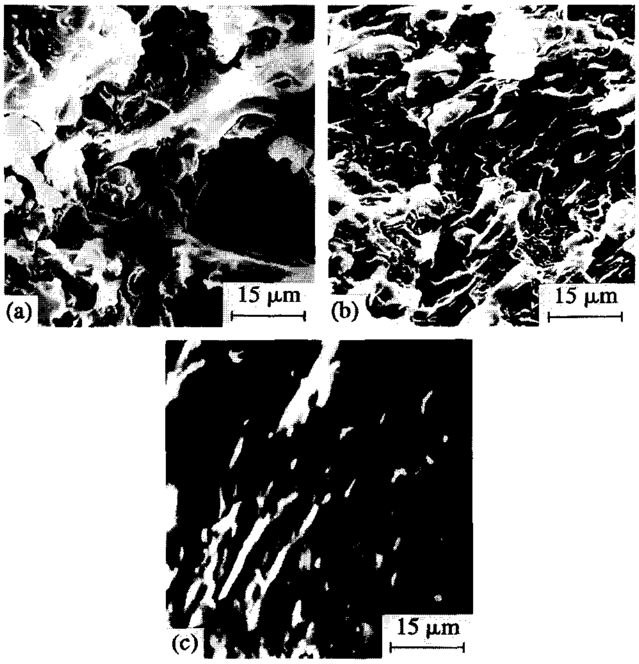

Figure 7 Scanning electron micrographs of cryofractured surfaces of 30/70 iPS/iPP blends with different amounts of added diblock copolymer: (a) 0%; (b) 5%; (c) 10%

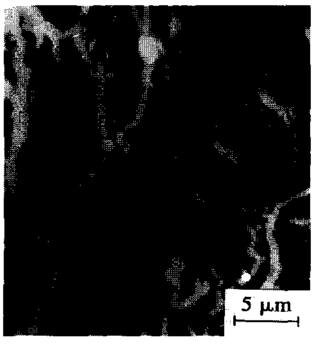

Figure 8 Scanning electron micrographs of cryofractured surfaces of the 10/30/70 iPS-*b*-iPP/iPS/iPP blend at high magnification

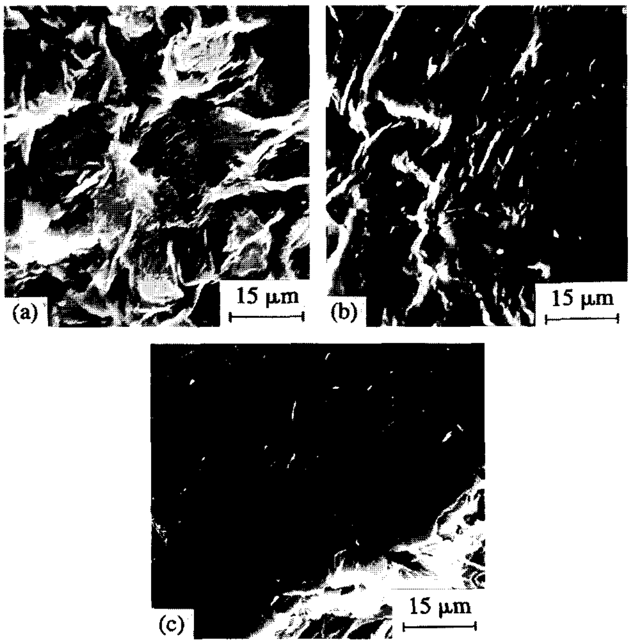

Figure 9 Scanning electron micrographs of cryofractured surfaces of 70/30 iPS/iPP blends with different amounts of added diblock copolymer: (a) 0%; (b) 5%; (c) 10%

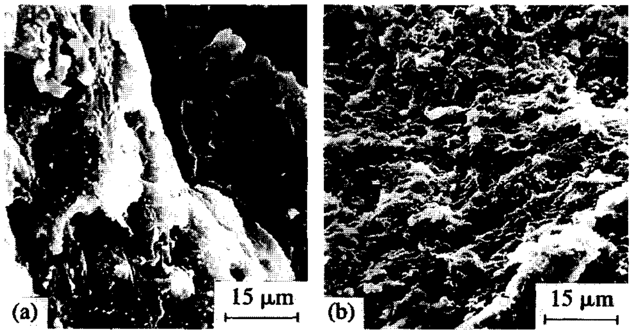

Figure 10 Scanning electron micrographs of impact fracture surfaces (at room temperature) of 70/30 iPS/iPP blends: (a) no copolymer; (b) with 10% added copolymer

of the iPS/iPP blend to which copolymer was added shows crazing and microshear features, which can make a significant contribution to the impact strength of the blends.

Morphological examinations of 50/50 iPS/iPP to which copolymer was added further shows clear evidence of the interfacial or emulsifying activity of the diblock copolymer (*Figure 11*). The unmodified iPS/iPP blend exhibits a rather large domain structure, wherein many domains are pulled away from their previous positions and where the surface of the holes left by removal of the PS phases appear to be very smooth. By adding as little as 10 wt% of diblock copolymer the iPS domains in the blend are not pulled away from their previous positions, and the iPP component no longer forms dispersed particles, but a continuous fine lamellar bundles which is firmly anchored into the iPS matrix. The emulsifying effect of the diblock copolymers therefore appears unambiguous, and correlates with their preferential location at the interface between the iPS and iPP phases, as previously reported[43].

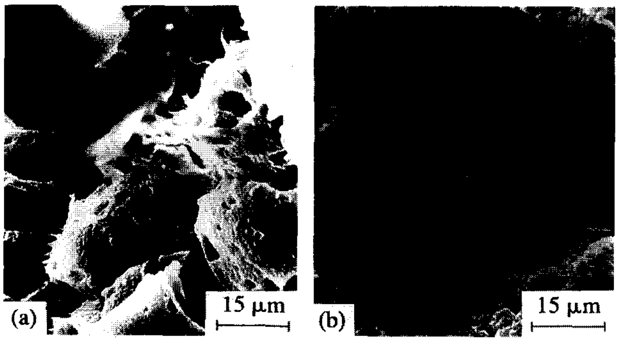

Figure 11 Scanning electron micrographs of impact fracture surfaces (at room temperature) of 50/50 iPS/iPP blends: (a) no copolymer; (b) with 10% added copolymer

Results obtained from the d.s.c., d.m.t.a. and morphological studies, as discussed above confirm the expectation of miscibility or compatibility, with interphase interaction occurring at the interphases between the components of the blend, which probably results from the anchoring of each sequence of the block to its corresponding component in the blend. Similar conclusions have been drawn by Teyssie and coworkers for PS/HPB-*b*-PS/PE blends[5,22]. These conclusions are in very good accordance with the mechanical properties (e.g. Izod impact and tensile strength) of the blends, which are discussed in the following.

Figure 12 shows the mechanical properties (i.e. Young's modulus, tensile strength, elongation at break, Izod impact strength) of iPS-*b*-iPP/iPS/iPP ternary blends. Stress–strain measurements demonstrate the (expected) poor ultimate behaviour of the unmodified iPS/iPP blend. Due to the brittleness of the iPS homopolymer and its poor compatibilization in the iPS/iPP blend, as shown already in the morphology studies, very low values for the Izod impact parameters are noted accordingly. However, adding diblock copolymer to the blends significantly enhances the impact strength of the blends (*Figure 12a*). It is believed that the improved adhesion in blends with a small amount of diblock copolymer gives rise to an increase of impact

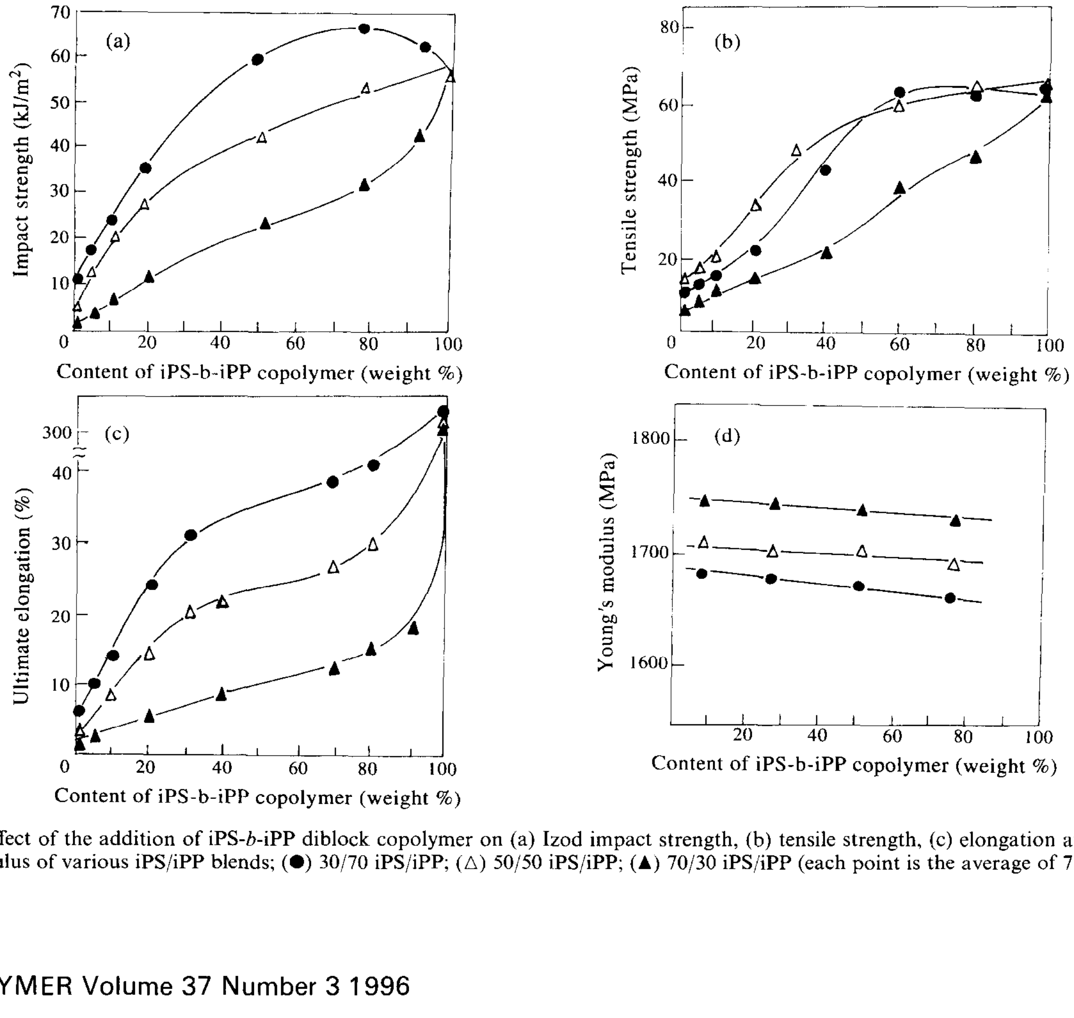

Figure 12 Effect of the addition of iPS-*b*-iPP diblock copolymer on (a) Izod impact strength, (b) tensile strength, (c) elongation at break and (d) Young's modulus of various iPS/iPP blends; (●) 30/70 iPS/iPP; (△) 50/50 iPS/iPP; (▲) 70/30 iPS/iPP (each point is the average of 7 tests)

strength. Addition of the diblock copolymer to the iPS/iPP blends increases the elongation at break and also the tensile strength. As both iPS and iPP have a higher tensile strength and elongation at break than the unmodified iPS/iPP blend, it seems feasible to suppose that the fracture path preferentially follows the weak interface between the polymer phases, or that fracture is initiated at the interface. This is confirmed by scanning electron microscopy of the fracture surfaces (see *Figures 7–11*). *Figure 12* shows a dependence of the tensile strength, elongation at break and Izod impact strength on the diblock copolymer content. The fact that adding copolymer to the iPS/iPP blends does contribute significantly to improving these mechanical properties indicates that the diblock copolymer concentrates at the interface between the two homopolymers, thus acting both as an adhesive and compatibilizer. To ensure tough behaviour, it is reasonable to suggest that the dispersed phase must not only adhere to the surrounding glassy polymer but must also be crosslinked through interaction or miscibility of individual diblock copolymer segments with corresponding iPS/iPP blend components. The anchoring/crosslinking-type effect of the copolymer in the blends retards any mechanically induced slippage between the iPS and iPP phases, thus increasing the Izod impact and tensile strength parameters. These results are in agreement with the results obtained from the d.s.c., d.m.t.a. and SEM studies, i.e. iPS-*b*-iPP copolymer is a very effective interfacial agent or compatibilizer for iPS/iPP blends.

Most methods of increasing the impact strength also reduce the stiffness[18,46]. However, this is not the case for the iPS-*b*-iPP/iPS/iPP ternary blends. The moduli of the modified iPS/iPP blends do not differ greatly from those of the unmodified blend (*Figure 12d*). It is clear that the enhancement in toughness described above does not have a corresponding loss of stiffness, and the latter is essentially unaffected by the compatabilizer. This improvement in impact strength of the blend with no apparent loss of stiffness is beneficial to the concept of tailor-making materials for specific applications. The impact-modulus behaviour seems to be related to the 'tough' or rigid characteristics of the semicrystalline isotactic structure of each block in the iPS-*b*-iPP diblock copolymer[41,42].

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