

# Syndiotactic Polystyrene-*b*-Atactic Polypropylene Block Copolymer Alloy as a Compatibilizer for Syndiotactic Polystyrene/Isotactic Polypropylene Blends

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**ABSTRACT:** A polymeric alloy (SP-A) containing syndiotactic polystyrene (sPS), atactic polypropylene (aPP), and about 66 wt % sPS-*b*-aPP diblock copolymer, was prepared by the sequential feed of monomers in the presence of the half-titanocene Cp\*Ti(OBz)<sub>3</sub> (where Cp\* is C<sub>5</sub>Me<sub>5</sub> and Bz is PhCH<sub>2</sub>), modified methylaluminumoxane, and external triisobutylaluminum. The effects of the SP-A alloy as a compatibilizer for sPS and isotactic polypropylene (iPP) blends were evaluated. The blending of sPS and iPP, with and without SP-A, was performed in a single-screw miniextruder with a side channel that allowed the continuous recycling of materials. The influence of SP-A on the mechanical and thermal properties of the immiscible sPS/iPP blends was investigated over a range of composition. The presence of the SP-A alloy resulted in a significant improvement of

the impact strength of the blends compared with that of pure sPS and their pure blends. This improvement was particularly obvious in the sPS/iPP (90/10 wt %) blend containing 5 wt % SP-A. Morphological analysis of the impact-fractured surface of the ternary blends indicated that the sPS-*b*-aPP diblock copolymer contained in the SP-A alloy acted as an efficient compatibilizer by decreasing the dispersed-phase iPP particle size, improving the interfacial adhesion, and generating a stable microphase-separated state. © 2003 Wiley Periodicals, Inc. *J Appl Polym Sci* 89: 1596–1605, 2003

**Key words:** diblock copolymer; syndiotactic; polystyrene; isotactic; poly(propylene) (PP); compatibility

## INTRODUCTION

Syndiotactic polystyrene (sPS), whose phenyl groups show alternating alignment on either side of the chain backbone, has recently generated widespread interest in various studies. Besides its inherent characteristics, such as low specific gravity, low viscosity, hydrolytic resistance, good moldability, and excellent electrical properties, sPS, unlike general purpose polystyrene, has high heat resistance and chemical resistance and a much higher rate of crystallization than isotactic polystyrene. sPS, however, also has some inherent disadvantages, including high brittleness and poor impact and tear resistances, that limit its commercial applications.<sup>1</sup> Some attempts have been made to modify sPS to improve its mechanical properties. Included among these modifications has been the addition of glass fiber. The latter has been used in other polymeric materials. For example, melt blends of poly(phenylene

oxide) (PPO) with glass-fiber-reinforced sPS has been found to improve its properties.<sup>2</sup> Generally, a high amount of inorganic filler is added to impart the desired mechanical properties. For example, glass fibers impart heat resistance and mechanical strength to sPS for application as construction materials and electronic parts. However, the high content of inorganic filler also brings about some deterioration in the material, such as an increase in product density and a loss of tenacity due to the interfacial incompatibility between the organic polymer and the inorganic filler. Melt intercalation of amorphous styrenic polymers into organophilic clays, followed by blending with sPS, also bestows a higher tensile strength.<sup>3</sup> Another way to modify the brittleness of sPS is blending with thermoplastic polymers with lower glass-transition temperatures ( $T_g$ 's) or elastomeric polymers, such as polyethylene, polypropylene, ethylene-propylene rubber (EPR), thermoplastic polyurethane (TPU), poly(2,6-dimethyl-1,4-diphenylene oxide), and so on. However, the problem here is that sPS is immiscible with most polymers because of a lack of specific interactions between sPS and the modifier polymer. In fact, a distinct phase separation with a sharp interface and large particles of the dispersed phase were previously observed, and poor mechanical properties were noted.<sup>4</sup>

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Therefore, in such cases, interfacial active compatibilizers are needed to improve the compatibility between the immiscible polymers in the blend. Block copolymers are well known as effective compatibilizers. Usually, each block segment of a diblock or triblock polymer is either miscible or has strong affinities with one of the two homopolymer phases. The block copolymer resides at the interface of the two phases, thus reducing the interfacial tension, enhancing the adhesion between the phases, and stabilizing the phase morphology against coalescence. The triblock copolymer polystyrene-*b*-poly(ethylene-*co*-butylene)-*b*-polystyrene (SEBS) is frequently chosen as a compatibilizer. Abis et al. reported that a fine dispersion of high-density polyethylene within the sPS matrix was found on the addition of SEBS.<sup>5</sup> Hong and Jo found that the size of the dispersed EPR phase in sPS/EPR/SEBS blends decreased and the particle size distribution became narrower with increasing amounts of SEBS in the blends.<sup>6</sup> In addition, Choi et al. chose reactive polystyrene (RPS) as a compatibilizer in sPS/PPO blends.<sup>7</sup> Increasing the RPS level reduced the rubber particle size due to reactions at the interfaces among the polymer phases. Xu et al. synthesized the diblock copolymer poly(styrene-*b*-4-vinylpyridine) by sequential anionic copolymerization and studied its compatibility in sPS/TPU blends.<sup>8</sup> A compatibilizing efficacy resulted from the polystyrene block in the diblock copolymers entering the noncrystalline region of sPS and the poly(4-vinylpyridine) block interacting with TPU through intermolecular hydrogen bonding.

Recently, Zambelli et al. synthesized a block-like sPS/butadiene copolymer with CpTiX<sub>3</sub>/methylaluminoxane (MAO) and TiX<sub>n</sub>/MAO.<sup>9</sup> Hu et al. prepared partially compatible blends of sPS/isotactic polypropylene (iPP) with TiCl<sub>4</sub>/MgCl<sub>2</sub>/β-diketone activated with MAO.<sup>10</sup> We also reported the syntheses of sPS-*b*-polyolefin (e.g., polyethylene, polypropylene, polybutylene) block copolymers with a sequential feed order in the presence of half-titanocene catalyst systems.<sup>11–13</sup> To the best of our knowledge, the use of compatibilizers bearing sPS blocks in blends based on sPS has not been reported in the literature. We report here an investigation of the compatibility of the diblock copolymer sPS-*b*-atactic polypropylene (aPP), in sPS/iPP blends. A strong connection at the interface could be expected because on one side, the sPS block should have been able to enter the crystalline domain of sPS, and on the other side, the aPP block is miscible with iPP, as already demonstrated.<sup>14</sup>

## EXPERIMENTAL

### Materials

The styrene/propylene block copolymerization was conducted by the sequential feeding of the two mono-

mers into a solution of η<sup>5</sup>-pentamethylcyclopentadienyl tribenzoxyl titanium [Cp\*Ti(Obz)<sub>3</sub>, where Cp\* is C<sub>5</sub>Me<sub>5</sub> and Bz is PhCH<sub>2</sub>]<sup>15</sup> and modified methylaluminoxane (mMAO)<sup>16</sup> together with the external addition of triisobutylaluminum (TIBA).<sup>12</sup> All reagents were purified before use. Solvents were refluxed over sodium under a nitrogen atmosphere and distilled before use. A dried glass reactor equipped with a magnetic stirrer was previously evacuated, thermostated at 40°C in an oil bath, and saturated with gaseous propylene. Dry toluene, mMAO, and Cp\*Ti(Obz)<sub>3</sub> were introduced into the reactor in this exact order. Propylene prepolymerization began under a total pressure of 130 kPa. After half an hour, the feed of propylene was stopped, and the residual propylene in the system was replaced by nitrogen. TIBA and styrene were injected into the reactor, after which the temperature of the reaction mixture was quickly increased to 90°C. After the copolymerization was allowed to proceed for 1 h, a large excess of acidified alcohol was added to quench the reaction. The product, designated as SP-A, was dried completely under vacuum at 80°C.

For reference, pure sPS with a weight-average molecular weight ( $M_w$ ) of 280,000 g/mol was prepared under similar conditions to the styrene copolymerization and with the same catalyst system. Pure aPP ( $M_w$  = 187,000 g/mol) was prepared under the same conditions as the propylene prepolymerization. 99.7 wt % of the aPP was soluble in diethyl ether.

sPS was kindly supplied by Shanghai Research Institute of Petrochemical Technology (Shanghai, China;  $M_w$  = 370,000 g/mol, syndiotacticity ≈ 95%). iPP was kindly supplied by Guangzhou Petrochemical Corp. (Guangzhou, China;  $M_w$  = 170,000 g/mol, isotacticity ≈ 92%).

### Preparation of the blends

All of the polymer materials were completely dried at 60°C in an oven under a vacuum for 3 days. Binary and ternary blends were premixed at the desired compositions, followed by the recycling of the melted components in a single-screw batch-type miniextruder (XJ-01, Jilin University Scientific Instruments, Changchun, China) equipped with a stainless steel screw with a length to diameter ratio 10, a screw diameter of 9.5 mm, and a side channel that allowed the continuous recycling of materials at the head of the mixing chamber. This was done at 300°C and a speed of 15 rpm for 5 min before extrusion. The blends were stabilized with 0.5 wt % antioxidant 1010. The extrudate was injection-molded into 15 mm long, 10 mm wide, and 4 mm thick specimens (for impact and flexural testing) with an injection-molding machine (WZM-1, Jilin University Scientific Instruments). The molding

TABLE I  
Results of Successive Extractions Performed with Boiling Solvents on the Styrene/Propylene Copolymerization Alloy SP-A, Pure sPS, and Pure aPP

	Successive extraction fraction (wt %)				
	Methylethylketone-soluble	Heptane-soluble	THF-soluble	Chloroform-soluble	Chloroform-insoluble
SP-A	5.18	2.47	17.2	48.8	26.4
sPS	2.58	0	0	3.27	94.2
aPP	0	100	—	—	—

conditions were as follows: barrel temperature = 295°C and molding temperature = 180°C.

### Testing and morphology

<sup>13</sup>C-NMR spectra of the samples in CDCl<sub>3</sub> and 1,1,2-trichloroethane were recorded on a Varian INOVA 500NB spectrometer at 60°C. IR spectra of the polymer films were recorded with a Nicolet 205 spectrophotometer.

Unnotched Izod impact and flexural testing were performed at room temperature on a pendulum-type testing machine (VEB, Werkstoffpruefmaschinen). The data were recorded as the average of five tests.

Dynamic mechanical analysis (DMA) was carried out in a bending mode with a dynamic mechanical thermoanalyzer (DuPont 2986). The  $T_g$ , storage modulus ( $E'$ ), and loss tangent ( $\tan \delta$ ) were measured at a frequency of 5 Hz and a heating rate of 5°C/min in the range of -70 to 150°C.

Melting curves of the blends were obtained with a differential scanning calorimeter (PerkinElmer, DSC-7) under a nitrogen atmosphere at a heating rate of 10°C/min.

The morphology of the specimens fractured in impact testing was observed with a Hitachi S-520 scanning electron microscope. The surface was sputter-coated with gold before observation under the microscope. The number-average diameter of the dispersed phase was calculated from the scanning electron microscopy (SEM) microphotographs.

## RESULTS AND DISCUSSION

### Composition of SP-A

The styrene/propylene copolymerization product SP-A was fractionated by successive extraction with boiling methylethylketone, heptane, tetrahydrofuran (THF), and chloroform in a Soxhlet-type extractor. Each fraction of SP-A was characterized by <sup>13</sup>C-NMR and IR spectroscopy. The results from the extraction were compared with those of pure sPS and pure aPP and are reported in Table I. Pure aPP dissolved completely in heptane, whereas pure sPS was insoluble in these solvents; however, 3 wt % of it was slightly

soluble in boiling chloroform. <sup>13</sup>C-NMR characterization demonstrated that the copolymer, extracted into boiling THF and chloroform, was a diblock copolymer composed of aPP and sPS blocks. In fact, the signals of multiplex methyl CH<sub>3</sub> (19.4–21.8 ppm) and methine CH (27.5 ppm) carbons assignable to aPP, those of methine CH (40.7 ppm) and methylene CH<sub>2</sub> (44.0 ppm), and a single sharp peak of phenyl C-6 (145.6 ppm) carbons arising from sPS were clearly observed in the spectra (Fig. 1). The heptane-soluble fraction and chloroform-insoluble fraction were the aPP and sPS homopolymers, respectively. The fraction soluble in methylethylketone was a mixture of atactic polystyrene (aPS) and the aPS-*b*-aPP copolymer. Therefore, the copolymerization product SP-A was *in situ* a chemical blend alloy containing about 2.47 wt % aPP, 26.4 wt % sPS, and 66.0 wt % sPS-*b*-aPP diblock copolymer.

The average molar ratio of the styrene and propylene units in the diblock copolymer, estimated by elemental analysis, was 79:21 in the THF-soluble fraction and 92:8 in the chloroform-soluble fraction (3 wt % sPS was subtracted).

### Mechanical properties

The mechanical properties of the binary sPS/iPP blends are shown in Figure 2. The blends were easily fractured; moreover, the impact strength and flexural strength of sPS decreased greatly in the presence of iPP. This adverse effect of iPP on sPS properties resulted from the immiscibility of sPS and iPP. Similar observation were found in other simple blends.<sup>17</sup> As a consequence, the interface between the matrix and the dispersed phase lacked sufficient adhesion, and fracture took place easily there. These results indicated that sPS could not be toughened by blending only with iPP.

The effect of the addition of the SP-A alloy on the impact properties of the sPS/iPP blends is shown in Figure 2. The SP-A content was fixed at 5 wt %, whereas the blend composition of sPS and iPP was varied. The sPS/iPP/SP-A ternary blends showed a higher impact strength than the mixtures without SP-A, implying that the diblock copolymer con-

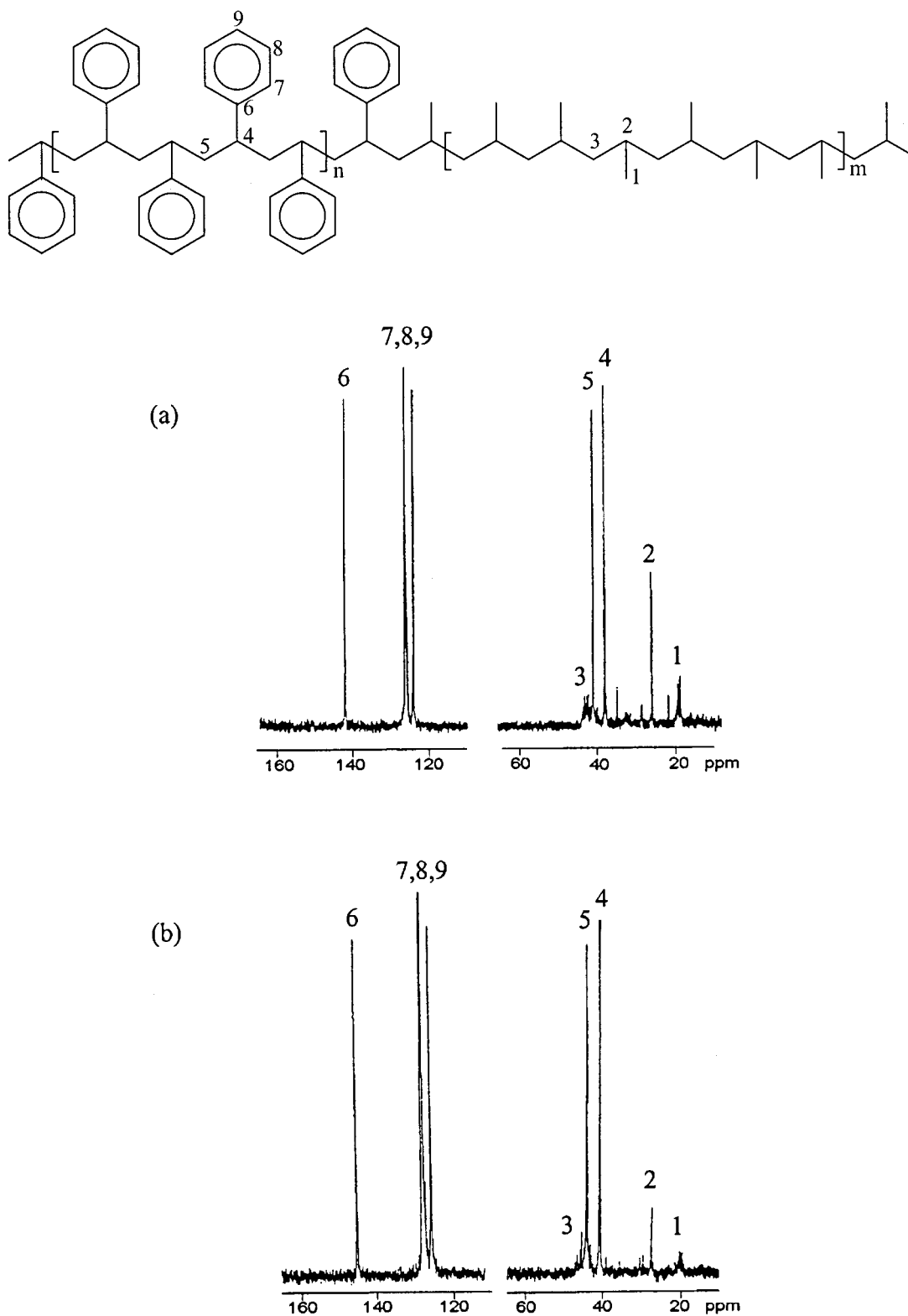
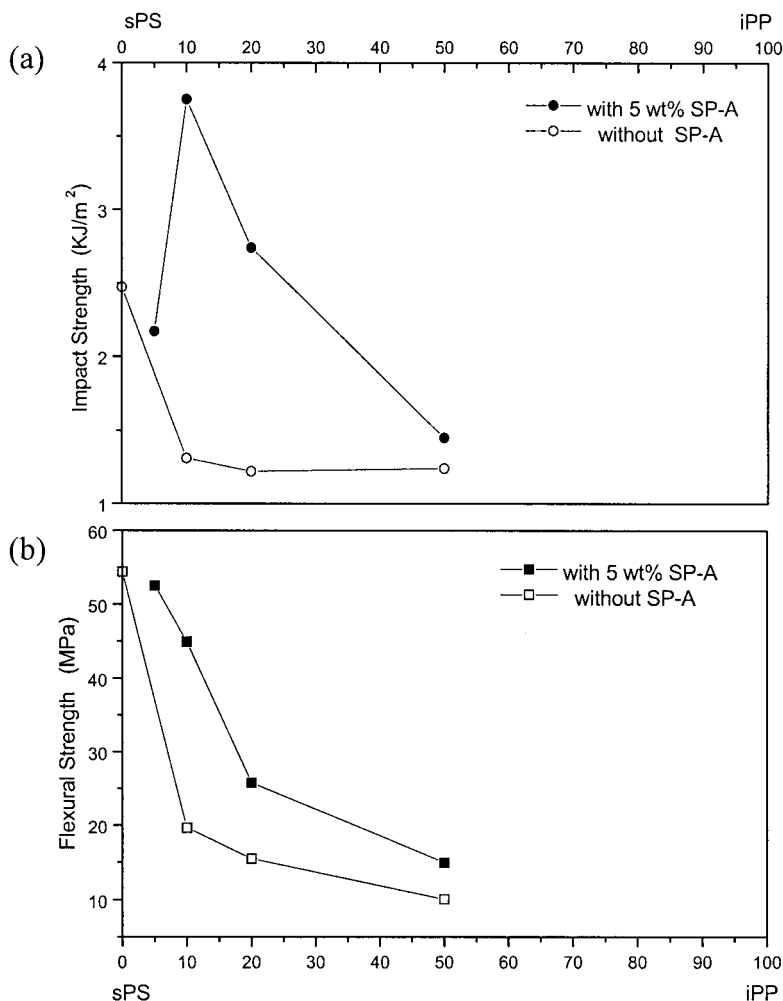


Figure 1  $^{13}\text{C}$ -NMR spectra of SP-A: (a) THF-soluble and (b) chloroform-soluble fractions.

tained in the SP-A alloy effectively increased the interfacial adhesion between the sPS and iPP phases. The sPS/iPP (90/10 wt %) blend with SP-A (5 wt %) added was extremely tough, as the impact

strength increased about three times. The blend containing about 50 wt % iPP was still brittle, and it was likely that a higher amount of SP-A was required for compatibilization.



**Figure 2** Effect of SP-A addition on the mechanical properties of sPS/iPP blends: (a) impact and (b) flexural strengths.

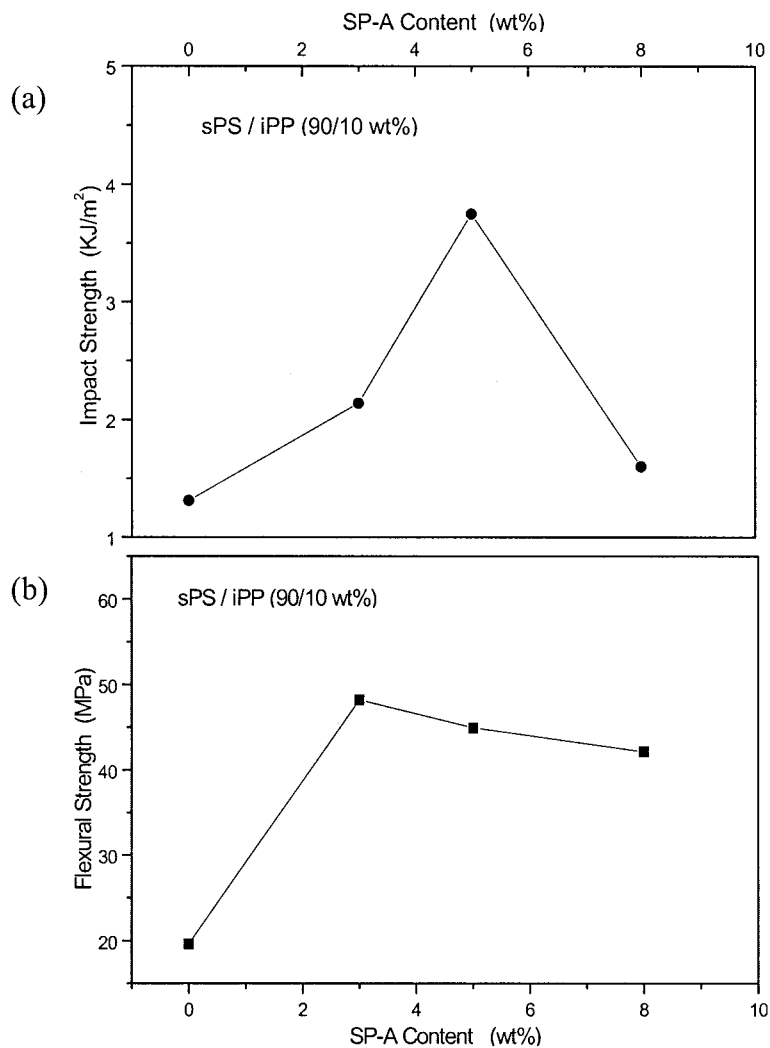
The impact strength of these ternary blends was also investigated as a function of SP-A content. Figure 3 shows that for a fixed composition of sPS/iPP (90/10 wt %), the impact strength increased initially with increasing SP-A content, followed by a subsequent decrease when the SP-A content was over 5 wt %. Such behavior may be explained by consideration of the fact that an increase in the SP-A alloy content, that is, an increase in the amount of the diblock copolymer, was expected to decrease the dispersed iPP particle size and improve the impact resistance if the iPP particles were too large for effective toughening. However, the impact strength of blends would become weaker if the dispersed-phase particle size fell below the lower limit for toughening.

Also, the flexural strength of sPS got dramatically worse with only the blending with iPP but was greatly enhanced by the addition of the SP-A alloy into the blends of sPS and iPP. This was further evidence that the interfacial adhesion was improved by the diblock copolymer contained in SP-A. However, for a fixed content of SP-A, the flexural strength of the ternary

blends descended rapidly with increasing iPP portion, whereas it was only slightly affected by variation of the SP-A content in the blends.

### Phase morphology

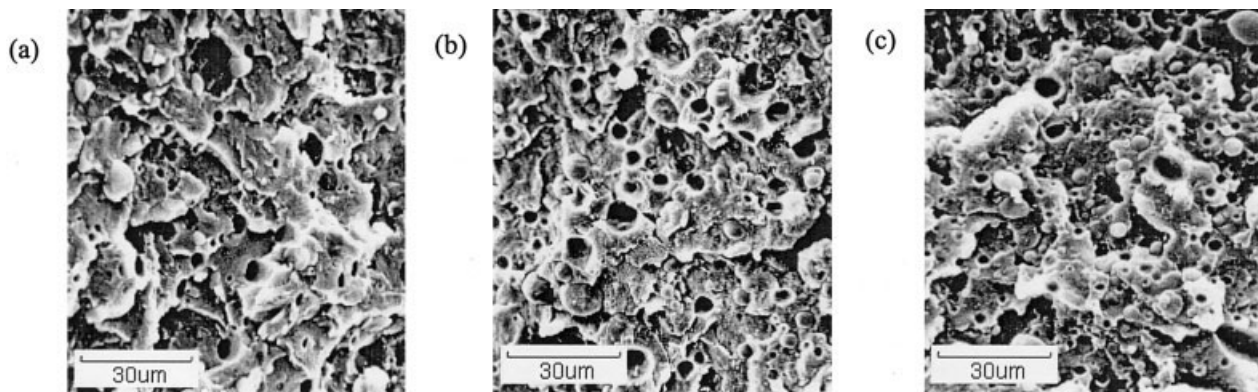
It is generally accepted that the mechanical properties of blends are controlled by their phase morphology. Therefore, the effect of the SP-A alloy on the impact resistance of sPS/iPP blends could be illustrated by the morphology of their impact-fractured surfaces. SEM micrographs of the impact-fractured surfaces of the blends containing variable amounts of SP-A (Fig. 4) showed that the dispersed-phase iPP particle size significantly decreased and became more regular with increasing amounts of SP-A alloy. Furthermore, the optimum size of the dispersed iPP particles ( $\sim 5 \mu\text{m}$ ), corresponding to the higher impact strength, was reached in the sPS/iPP (90/10 wt %) blend with 5 wt % SP-A, as shown in Figure 5. The existence of an optimum particle size was also reported in other toughening blends.<sup>18,19</sup> These results revealed that the sPS-*b*-aPP diblock copolymer con-



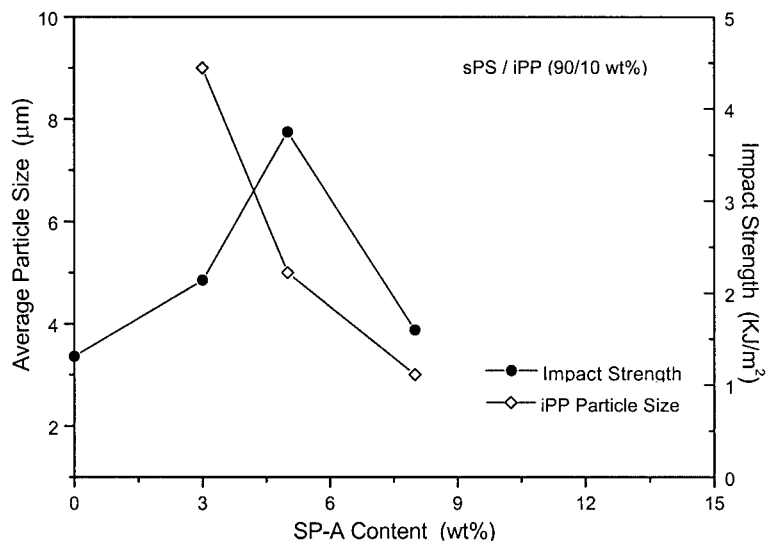
**Figure 3** Effect of SP-A content on the mechanical properties of the 90/10 wt % sPS/iPP blend: (a) impact and (b) flexural strengths.

tained in the SP-A alloy acted as an efficient compatibilizing agent, reducing the interfacial tension and decreasing the dispersed-phase particle size.

In addition to the reduction of the dispersed particle size, the diblock copolymer also played a major role in the control of impact resistance by bridging at the



**Figure 4** SEM photomicrographs of the impact-fractured surfaces of 90/10 wt % sPS/iPP blends containing variable amounts of SP-A: (a) 3, (b) 5, and (c) 8 wt % (magnification = 1000×).

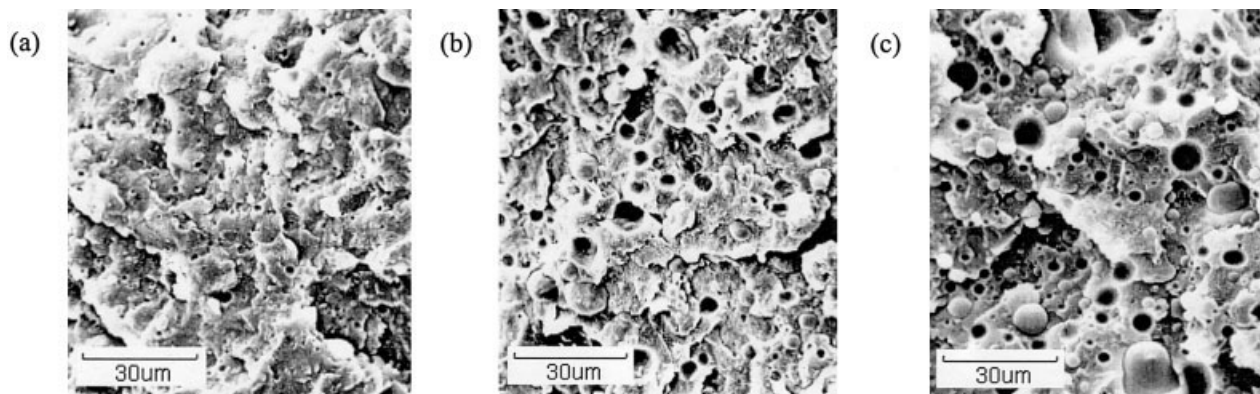


**Figure 5** Effect of SP-A content on the impact strength and the number-average diameter of iPP particles for the 90/10 wt % sPS/iPP blends.

interface of the two polymeric phases through the percolation of aPP and sPS blocks into the dispersed-phase iPP and the sPS matrix. Figure 6 shows, for a fixed SP-A content (5 wt %), a variation in the morphology of the impact-fractured surface of the ternary blends as a function of the ratio of sPS to iPP. A fine dispersion of iPP particles was found for sPS/iPP/SP-A (95/5/5 wt %), but it turned into an irregular dispersion for sPS/iPP/SP-A (80/20/5 wt %). Moreover, in the latter blend, the particle size of the iPP phase became larger, whereas the shape of the iPP particles grew round. In addition, the interface between the sPS matrix and the dispersed iPP became smooth and clear, indicating a poorer interfacial adhesion between the two phases due to an insufficient amount of SP-A, that is, the sPS-*b*-aPP diblock copolymer.

### Thermal properties

The differential scanning calorimetry (DSC) plot of the sPS-*b*-aPP diblock copolymer contained in the SP-A alloy (Fig. 7) exhibited two melting peaks of the sPS block at about 266 and 257°C, corresponding to  $\alpha$  and  $\beta$  crystalline forms containing a zigzag plane. This polymorphic pattern was also observed in the pure and mixed states.<sup>20</sup> Thus, we reasonably deduced that the interfacial adhesion found in the ternary blends was improved not only by the entanglement of chains at the interface but also by the entry of crystalline sPS blocks into the crystalline domain of the sPS matrix. The  $T_g$  derived from the aPP block was not as sharp as that of the sPS block because of the much shorter aPP block, which might have been responsible for the limited improvement in toughness.



**Figure 6** SEM photomicrographs of the impact-fractured surfaces of (a) 95/5, (b) 90/10, and (c) 80/20 wt % sPS/iPP blends containing 5 wt % SP-A (magnification = 1000 $\times$ ).

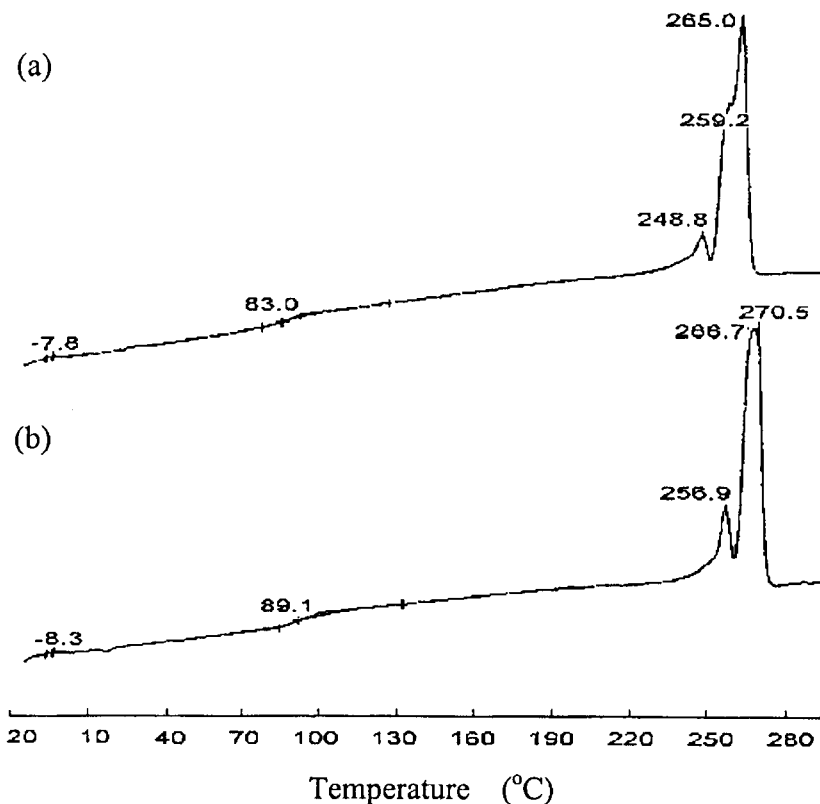


Figure 7 DSC curves of the (a) THF-soluble and (b) chloroform-soluble fractions of the sPS-*b*-aPP diblock copolymer.

For the binary blends of sPS and iPP, each blend component showed the same melting temperature ( $T_m$ ) as its pure material (Table II), indicating the immiscibility of the two components and the existence of macroseparated phases from coalescence. Thermal analysis curves of the ternary blends containing a fixed amount of SP-A (5 wt %) (Fig. 8) showed that with increasing iPP content, the  $T_m$  of sPS decreased

and the melting peaks became wider and lower. It seemed that initially, the microphase iPP, which was well dispersed in the matrix because of the SP-A compatibilizing effect, affected the crystal growth of sPS slightly. However, when the content of iPP was increased, the crystallization of sPS was strongly interfered with by the large iPP particles arising from insufficient compatibilization.

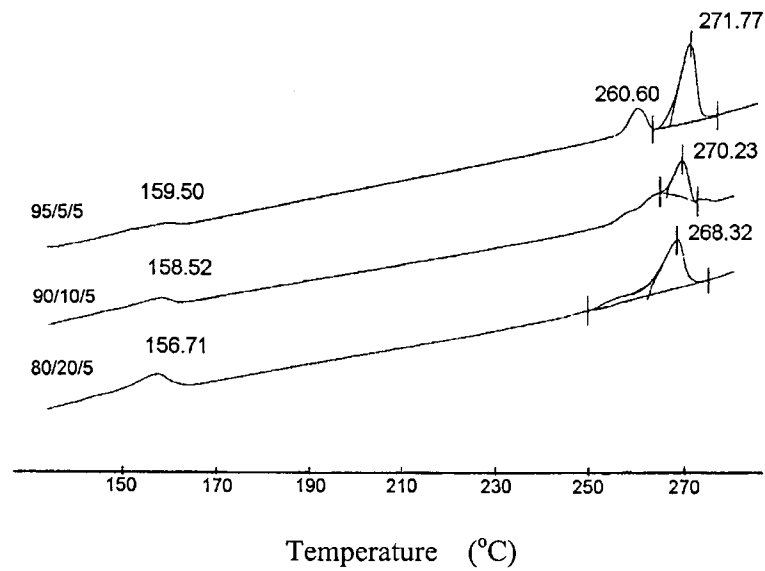
TABLE II  
DSC and DMA Data Obtained for sPS/iPP Blends with and without the Addition of SP-A

Sample	Composition (sPS:iPP:SP-A)	$E'$ <sup>a</sup> (MPa)	$T_g$ (°C) <sup>a</sup>		$T_m$ (°C) <sup>b</sup>	
			PP	sPS	iPP	sPS
SP-A		15,600	12	106		273
sPS		23,400		103		274
iPP		12,500	28		157	
sPS/iPP	90:10:0				156	273
sPS/iPP	80:20:0				157	273
sPS/iPP/SP-A	95:5:5	20,900	11	114	159	271
sPS/iPP/SP-A	90:10:3				157	270
sPS/iPP/SP-A	90:10:5	18,600	10	116	158	270
sPS/iPP/SP-A	90:10:8				158	270
sPS/iPP/SP-A	80:20:5	17,800	13	113	156	268
sPS/iPP/SP-A	50:50:5	16,300	11	110		

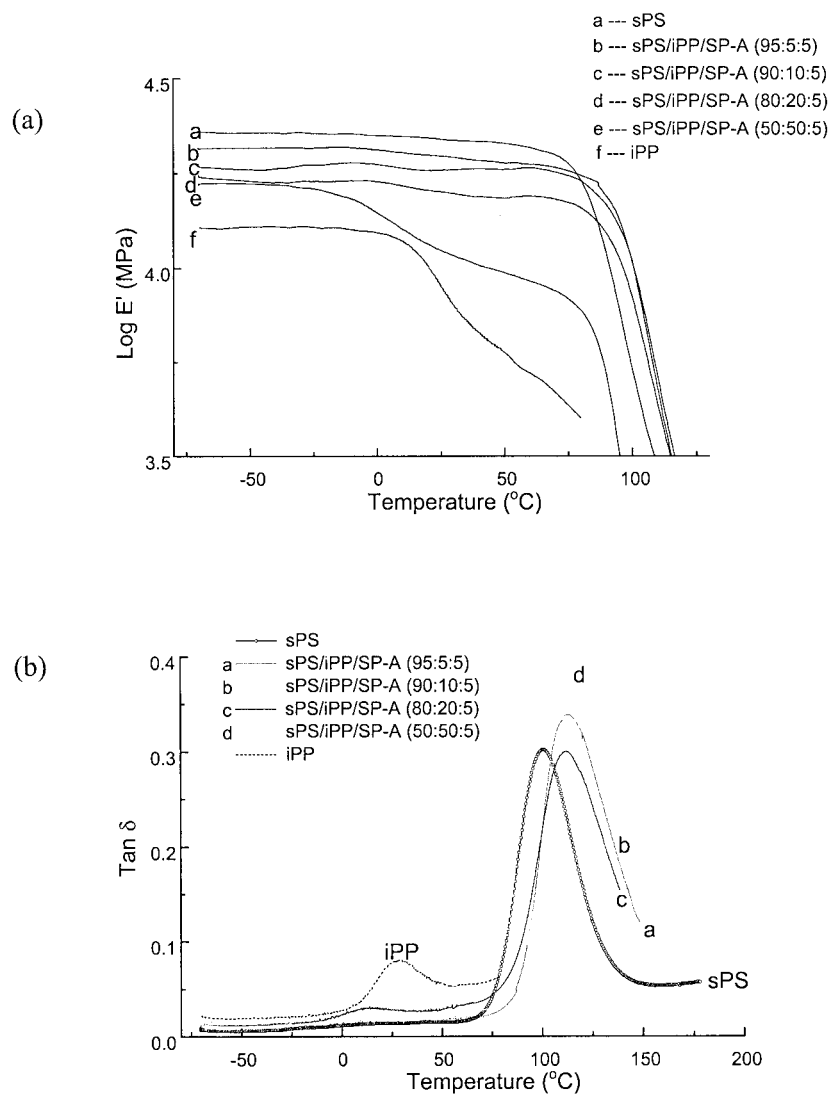
<sup>a</sup>Estimated from DMA.

<sup>b</sup>Determined by DSC.





**Figure 8** DSC curves of the sPS/iPP/SP-A blends with different sPS/iPP ratios.



**Figure 9** Variation of (a)  $\log E'$  and (b)  $\tan \delta$  of sPS/iPP/SP-A blends with different sPS/iPP ratios.

### Dynamic mechanical properties

DMA for sPS/iPP/SP-A blends was performed to investigate the temperature dependence of the dynamic  $E'$  and  $\tan \delta$ . Figure 9 shows that the  $E'$  of the ternary blends decreased slightly in the temperature range of  $-70$  to  $70^\circ\text{C}$  up to 20 wt % iPP, suggesting that in this range of composition, the  $E'$  of the blends was related to the sPS matrix. When the portion of iPP was increased up to 50 wt %, the  $E'$  of the blend decreased greatly. This was already significant at  $0^\circ\text{C}$  and reached a value close to that of pure iPP.

Two relaxation peaks appeared in the  $\tan \delta$  curves of the ternary blends [except for curve (a)]. The dispersion occurring at about  $10^\circ\text{C}$  was ascribed to the glass-rubber transition of iPP, but it was lower than that of pure iPP; the dispersion at about  $110^\circ\text{C}$  was ascribed to that of sPS, but it was higher than that of pure sPS. This trend is uncommon because the two peaks are expected to shift inward.

### CONCLUSIONS

Styrene/propylene block copolymerization was achieved with a titanocenic catalyst system. The resulting SP-A product was a chemical blend alloy containing about 66 wt % sPS-*b*-aPP diblock copolymer together with the sPS and aPP homopolymers.

SP-A proved to be effective in compatibilizing the immiscible blend of sPS and iPP. This was achieved by the percolation of the aPP and sPS blocks of the diblock copolymer making up the SP-A alloy into the dispersed-phase iPP and the sPS matrix of the blend. The SP-A alloy thus acted as a bridge between the two immiscible phases. SEM micrographs of the impact-fractured surfaces of the ternary blends showed improved phase morphologies with a decrease in iPP phase particle size. An effective and stable dispersion of iPP in the sPS matrix and stronger interfacial adhe-

sion were also observed. The investigation into the effect of SP-A on the mechanical properties of sPS/iPP blends showed that the impact strength and flexural strength were significantly improved over the blends without SP-A. This was more pronounced when the SP-A content was 5 wt % and the ratio of sPS/iPP was 90/10 wt %. The compatibility of sPS and iPP increased with increasing SP-A content and decreased with increasing iPP portion.

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