

Compatibilization effects of block copolymers in high density polyethylene/syndiotactic polystyrene blends

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Abstract

Three triblock copolymers of poly[styrene-*b*-(ethylene-*co*-butylene)-*b*-styrene] (SEBS) of different molecular weights and one diblock copolymer of poly[styrene-*b*-(ethylene-*co*-butylene)] (SEB) were used to compatibilize high density polyethylene/syndiotactic polystyrene (HDPE/sPS, 80/20) blend. Morphology observation showed that phase size of the dispersed sPS particles was significantly reduced on addition of all the four copolymers and the interfacial adhesion between the two phases was dramatically enhanced. Tensile strength of the blends increased at lower copolymer content but decreased with increasing copolymer content. The elongation at break of the blends improved and sharply increased with increments of the copolymers. Drop in modulus of the blend was observed on addition of the rubbery copolymers. The mechanical performance of the modified blends is strikingly dependent not only on the interfacial activity of the copolymers but also on the mechanical properties of the copolymers, particularly at the high copolymer concentration. Addition of compatibilizers to HDPE/sPS blend resulted in a significant reduction in crystallinity of both HDPE and sPS. Measurements of Vicat softening temperature of the HDPE/sPS blends show that heat resistance of HDPE is greatly improved upon incorporation of 20 wt% sPS. © 2001 Elsevier Science Ltd. All rights reserved.

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1. Introduction

Syndiotactic polystyrene (sPS) is a new semicrystalline polymer with a melting temperature of about 270°C and a glass transition temperature similar to that of atactic polystyrene (aPS). Its high heat resistance and modulus of elasticity, low dielectric constant, excellent resistance to chemicals and relatively fast crystallization rate make sPS a potential thermoplastic for a large number of applications in the automotive and electronic industries [1]. It has gained increasing academic and industrial interests since its first successful synthesis using a metallocene catalyst reported by Ishihara [2]. Albeit these desirable properties, a disadvantage of sPS is its inherent brittleness. Thus the application of sPS might be found favorable predominantly

in its blends with other polymers or composites. In patent literature many kinds of polymeric blends based on sPS and thermoplastic or elastomeric polymers have been reported [3–11]. However, only a few papers relating to modification of sPS have been published [12–14]. In order to develop the use of this new polymer, an attempt has been made to make use of sPS to modify other polymers. HDPE, a well-known thermoplastic resin, excellent in moldability and sufficiently high in toughness, is not sufficiently high in heat resistance that limits its use at higher temperatures. Polymers produced by blending sPS and HDPE are certainly an ideal combination to create new products with balanced properties provided that the advantage of one component can compensate the deficiencies of the other. It is well established that simple blends of two immiscible polymers usually have large discreet dispersed phases and weak interfacial adhesion, resulting in poor mechanical properties. Therefore, a compatibilizer is required to enhance the interfacial adhesion between the phases of HDPE and sPS. Generally, an effective compatibilizer should: (1) reduce the interfacial tension between the two phases leading to a finer dispersion

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Table 1
Materials used in the present study

Designation	Source	Molecular characteristics		PS (%)
HDPE 5000 S	Daqing Petrochemical	MI = 0.9 (g/10 min)		–
sPS Questra F2250	Dow Chemical Co.	$M_w = 25 \times 10^4$, $M_n = 9 \times 10^4$		–
		$M_n (\times 10^{-3})$		
		PS block	EB block	
SEBS Kraton 1651 (K–H)	Shell Chemical	29	116	32
SEBS Kraton 1650 (K–M)	Shell Chemical	10.3	53.3	29
SEBS Kraton 1652 (K–L)	Shell Chemical	7.0	37.5	32
SEB (J–D)	Japan Synthetic Rubber	22.5	128	15

of one phase in another, (2) enhance adhesion by coupling the phases together, and (3) stabilizing the dispersed phase against coalescence [15]. The most desirable compatibilizer for HDPE/sPS blends, according to theories [16–19], would be a block copolymer containing sPS block and PE block, but such a block copolymer could not be prepared directly due to limitation in polymerization mechanism.

Diblock or triblock copolymers containing aPS block and poly(ethylene-*co*-butylene) (EB) block, such as SEB and SEBS can be synthesized by sequential anionic living polymerization of styrene and butadiene, followed by hydrogenation of the polydiene blocks into polyolefin. These copolymers have been widely used as compatibilizers in PE/aPS blends [20–35]. Studies have shown that sPS is miscible with aPS [36–39]. Therefore, these copolymers are chosen in this work as compatibilizers for PE/sPS blends. The effect of several parameters, such as molecular weight, chemical composition and architecture of SEBS (SEB) block copolymers on the morphology and mechanical properties of PE/aPS blends have been intensively investigated [27–32]. However, little consideration has been given to the influence of the mechanical properties of the *compatibilizer* on the mechanical properties of the blends.

In the present study, three triblock copolymers (SEBS)

with different molecular weight and one diblock were chosen to be compatibilizers for HDPE/sPS blends. The effects of the four block copolymers on the morphology and mechanical properties and of HDPE/sPS blends were examined as a function of copolymer concentration. It is attempted to elucidate, in conjunction with some literature results, the above-mentioned issue of the influence of mechanical properties of the compatibilizer on the properties of the *blends*. Here we use minor amounts of sPS to modify HDPE, aiming at improving the heat resistance of HDPE. The effects of the compatibilizers on melting and crystallization behavior of the blends have also been investigated.

2. Experimental

2.1. Materials

The HDPE (5000S) used in this study is the product of Daqing Petrochemical (China), with melt flow index MI = 0.9 (g/10 min). sPS (Questra F2250) with molecular weight of $M_w = 25 \times 10^4$, $M_n = 9 \times 10^4$ (GPC), was from Dow Chemical. Three triblock copolymers SEBS are products of the Shell Chemical, Kraton G1651, Kraton G1650, and Kraton G1652 (in this paper referred to as K–H, K–M and K–L, respectively). The diblock copolymer SEB, denoted as J–D, is the product of Japan Synthetic Rubber. The characteristics of these copolymers are summarized in Table 1.

2.2. Blends preparation

All the HDPE/sPS = 80/20 (w/w) blends were prepared by melt mixing in a 60 ml batch Plastomill (torque rheometer, TOYOSEKI), at 290°C and 60 rpm for 8 min. The weight percentage of the block copolymer in each blend is based on the total weight of the blend (Table 2). After mixing, the samples were compression molded into sheets with a Ya Don hydraulic press at 290°C and 10 MPa pressure for 5 min and cooled to room temperature. Tensile and morphologic specimens were cut from the molded sheets.

Table 2
Composition of HDPE/sPS (80/20) blends

Blend code	Compatibilizer content (wt%)			
	K–H	K–M	K–L	J–D
H-1	–	–	–	–
H-2a	2	–	–	–
H-6a	6	–	–	–
H-10a	10	–	–	–
H-2b	–	2	–	–
H-6b	–	6	–	–
H-10b	–	10	–	–
H-2c	–	–	2	–
H-6c	–	–	6	–
H-10c	–	–	10	–
H-2d	–	–	–	2
H-6d	–	–	–	6
H-10d	–	–	–	10

2.3. Morphological observation

The morphologies of selected blends were examined by SEM using a Hitachi S-570 microscope at an accelerating voltage of 20 kV. The cryogenically fractured surfaces of the molded specimens were coated with gold before SEM examination.

2.4. Mechanical properties

Tensile tests were performed at room temperature using dumbbell specimens ($20 \times 4 \times 1 \text{ mm}^3$) on Instron 1121 electronic testing instrument at a cross-head speed of 20 mm/min. Each tensile value reported is the average of 10–12 tests.

2.5. Thermal analysis

The melting and crystallization behavior of the blend was studied using a Perkin–Elmer DSC-7 under a nitrogen atmosphere. To ensure comparable thermal history, all samples were first heated to 300°C for 5 min, then cooled down to 50°C and reheated to 300°C, all at a rate of 10°C/min.

The heat of fusion for 100% crystallinity of HDPE and sPS are taken as 293 J/g [40] and 53 J/g [41], respectively, for crystallinity calculations.

2.6. Vicat softening temperature

Vicat softening temperature of HDPE/sPS blends were measured using a Perkin–Elmer TMA-7. The specimens (2.5 mm thick) were loaded with a static force of 100 mN/mm². The temperature was raised from 0 to 200°C at a rate of 5°C/min under a nitrogen atmosphere. The Vicat softening temperature was taken at a penetration of 1 mm.

3. Results and discussion

3.1. Morphology of the blends

The SEM micrograph of cryogenically fractured surface of HDPE/sPS blend is presented in Fig. 1. The uncompatibilized HDPE/sPS blend shows the typical characteristics of an immiscible blend. The spherical domains of the dispersed sPS phase are large and have a broad size distribution (from ca. 1 to ca. 10 μm). The interface between the HDPE matrix and the dispersed sPS is smooth and clear. Very poor adhesion between the two phases is evidenced by cavities formed in fracturing the specimen.

In order to improve the morphology of the blends, three triblock copolymers having identical structure and similar chemical compositions but very different molecular weight (MW) and one diblock copolymer were employed to compatibilize the HDPE/sPS blends.

Fig. 2 shows SEM micrographs of HDPE/sPS (80/20) modified by varying amounts of the higher MW triblock

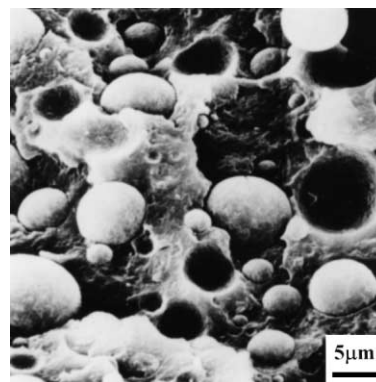


Fig. 1. SEM micrograph of fracture surface of uncompatibilized HDPE/sPS (80/20) blend.

copolymer (K–H). On addition of 2 wt% K–H to the HDPE/sPS blend, the size of the dispersed particles decreased (average size 1.5 μm) and more finely dispersion is obtained. In addition, most sPS particles are fractured,

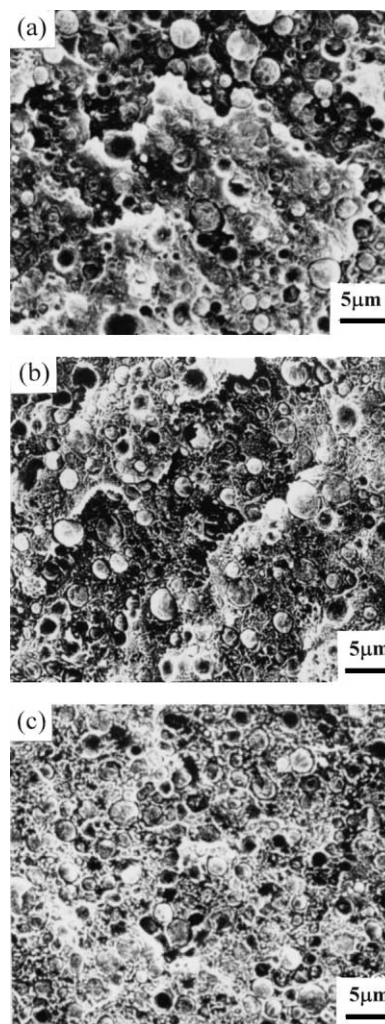


Fig. 2. SEM micrographs of fracture surfaces of HDPE/sPS (80/20) blends compatibilized with various amounts of K–H: (a) 2 wt%; (b) 6 wt%; (c) 10 wt%.

denoting that the interfacial adhesion was enhanced (Fig. 2a). When more K–H was added (6 wt%), no significant change in the size of sPS particles is observed (Fig. 2b). With further increment of K–H to 10 wt%, the interface becomes rougher and still many spherical cavities can be observed, suggesting no very strong interfacial adhesion between the two phases (Fig. 2c). Quite different from morphologies of the HDPE/sPS blends modified with K–H, the HDPE matrix formed an interlocking phase (a continuous network) when 2 wt% medium MW triblock copolymer K–M was added. The sPS particles of about 1 μm are interlocked in the HDPE matrix. Such a structure is commonly observed in the PE/aPS blends containing higher PE contents [22,33]. It is noteworthy that both the matrix and the dispersed sPS particles are fractured, indicating a very strong adhesion between the two phases (Fig. 3a). The particle size continues to decrease when K–M content is increased to 6 wt%. No further reduction in particle size of sPS is observed when K–M content is increased to

10 wt% (Fig. 3c). The lower MW triblock copolymer K–L and the diblock copolymer are also effective in compatibilizing the HDPE/sPS blends and their morphology changes are very similar with that modified by K–M, except at lower copolymer content where the domains are relatively larger than that modified with K–M, as shown in Figs. 4 and 5.

Comparing the effect of the different copolymers on blend morphology, it is clear that the medium MW triblock copolymer K–M is more effective in compatibilizing HDPE/sPS in reducing the particles size of the dispersed sPS and enhancing interfacial adhesion than either the lower or higher MW triblock copolymer and even more effective than the diblock copolymer J–D. Such a result is in accord with previously results reported by Taha [31], where the same three triblock copolymers (Kraton 1651, 1650 and 1652) were used in compatibilizing LDPE/aPS blends. Relatively poor compatibilizing ability of the higher MW copolymer, K–H, is due to the fact that its high melt

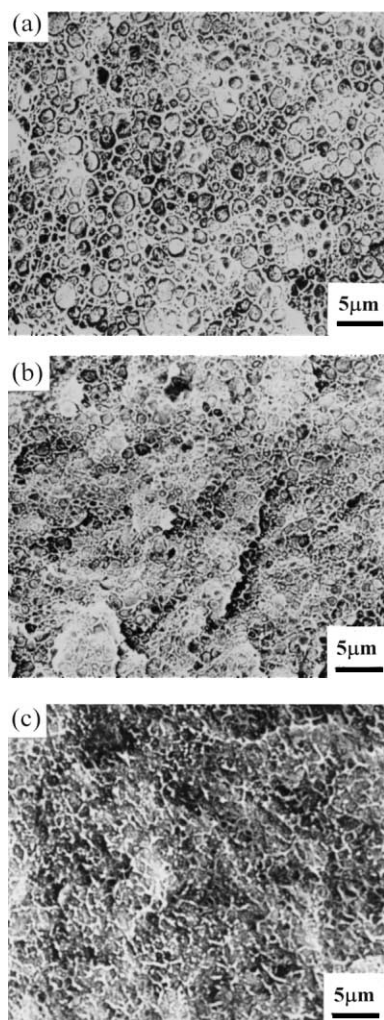


Fig. 3. SEM micrographs of fracture surfaces of HDPE/sPS (80/20) blends compatibilized with various amounts of K–M: (a) 2 wt%; (b) 6 wt%; (c) 10 wt%.

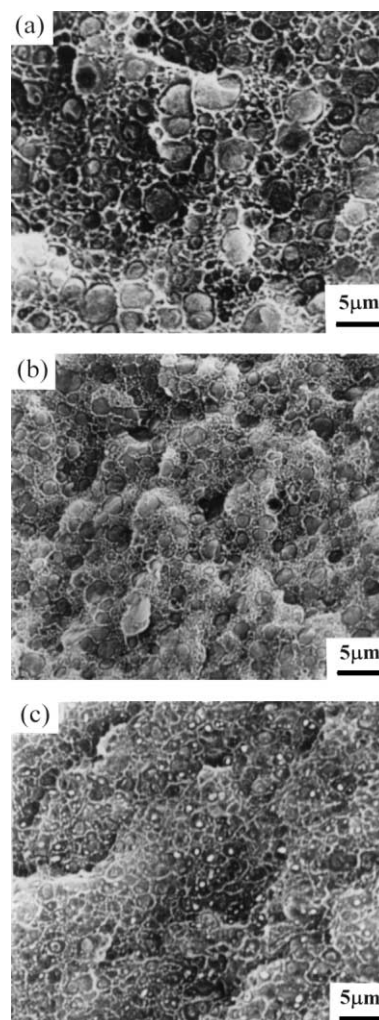


Fig. 4. SEM micrographs of fracture surfaces of HDPE/sPS (80/20) blends compatibilized with various amounts of K–L: (a) 2 wt%; (b) 6 wt%; (c) 10 wt%.

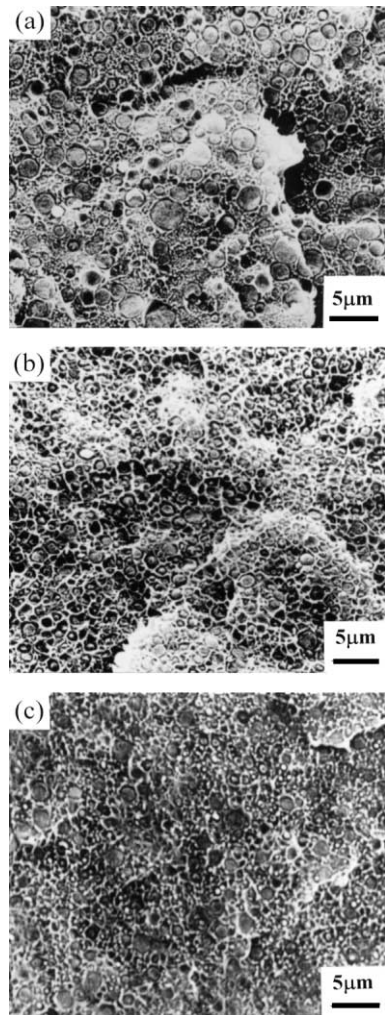


Fig. 5. SEM micrographs of fracture surfaces of HDPE/sPS (80/20) blends compatibilized with various amounts of J–D: (a) 2 wt%; (b) 6 wt%; (c) 10 wt%.

viscosity prevents it from migrating effectively to the interface. Another factor is that the higher MW copolymer tends to form micelles more readily [42], which would decrease the compatibilizing effect.

3.2. Tensile properties of the blends

Fig. 6 shows the tensile properties of HDPE/sPE (80/20) blends modified by varying amounts of the four copolymers. The binary HDPE/sPS exhibits relatively low tensile strength due to the very poor adhesion between sPS particles and HDPE matrix. Addition of 2 wt% of all the copolymers except K–H results in a significant improvement in tensile strength (Fig. 6a). Of the four copolymers, the diblock copolymer J–D gives the highest increase in tensile strengths although the triblock K–M is the most effective in compatibilizing as shown in Figs. 2 and 4. This may possibly arise from the more effective entanglements by the diblock copolymer in the interfacial region. It is the

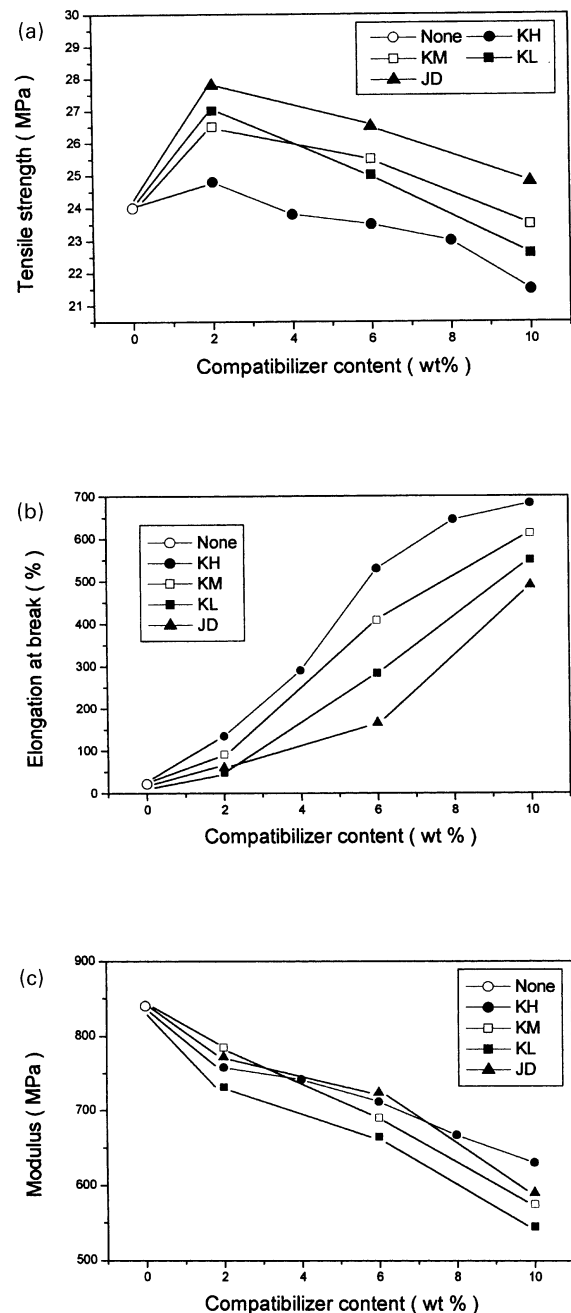


Fig. 6. Effect of compatibilizers on tensile properties of HDPE/sPS (80/20) blends: (a) tensile strength; (b) elongation at break; (c) modulus.

enhancement in interfacial adhesion between the two phases that brings about increase in tensile strength. The higher MW K–H, however, has little effect on tensile strength of the blends because of its relatively poor ability in compatibilizing the blends.

The tensile strength decreases with further addition of the copolymers (Fig. 6a). At 10 wt% of the copolymer, the tensile strengths of the K–H and K–L modified blends are even lower than that of the unmodified blend. Similar results were obtained by Xu [34] and Karrad [28] for HDPE/aPS blend compatibilized by a SEBS triblock copolymer

(Kraton 1652). This suggests that the tensile strength of a compatibilized blend is determined not only by the interfacial adhesion but also by the strength of the matrix that is highly affected by the amount of the compatibilizer.

It is well established that only a small part of the compatibilizer is located at the interfacial area between the dispersed phase and the matrix, the rest distributing as micelles or micro-particles in either the matrix or the dispersed phase. Since the chosen block copolymers used in the present study contain about 30% PS, these copolymers selectively distribute in HDPE matrix [43]. The tensile strength of this rubbery block copolymer is much lower than either the HDPE matrix or the dispersed sPS phase, so the part that distributes in HDPE matrix causes a decrease in tensile strength of the blends. The results of early work [20] that the tensile strength of aPS/LDPE blend compatibilized by graft copolymer PE-*g*-PS, whose tensile strength is much higher than that of the corresponding PS/LDPE blend, increased with increments of the graft copolymer up to 30% also support our explanation. Thus, a compatibilizer could either improve or lower the tensile strength of a blend, depending on the compatibilizing ability of the compatibilizer as well as the tensile strength of itself.

The elongation at break of HDPE/sPS blends as a function of the compatibilizer content is shown in Fig. 6b. The unmodified HDPE/sPS blend exhibits a very low elongation at break (only about 10%), owing to the large size of the dispersed sPS and the very weak interfacial adhesion between the two phases. However, the elongation at break is significantly improved on addition of either of the four copolymers and increases sharply with increasing copolymer content. The higher MW triblock copolymer (K–H) clearly improves the elongation at break of the blend to a greater degree than do the other three copolymers, in spite of the fact that K–H exhibits a relatively lower ability in improving the morphology of the blend (Fig. 2).

The sensitivity of elongation at break to copolymer content is seen in the fact that, though only 50–150% at a low copolymer contents, it increases dramatically to a high level of 550–700% at higher copolymer contents. This demonstrates that ductility of the compatibilized HDPE/sPS blends depends not only on the morphology of the blend, which determines better interfacial adhesion and finer domain size of the dispersed component, but also on the content of the rubbery compatibilizer added, which acts as both a compatibilizer and a toughening modifier.

The best ductility, attained in K–H modified HDPE/sPS blend, is probably attributable to a finer dispersion of sPS particles in HDPE matrix and at the same time a relatively poorer interfacial adhesion caused by K–H. Burau [44] has shown that strong adhesion between the HDPE matrix and the dispersed aPS produced by SEBS copolymer has a negative effect on the ductility of the HDPE/aPS (85/15) blend. The particle size of the dispersed K–H in HDPE matrix also favors an increase in the elongation of the blend. Paul [45] has proposed that there existed an optimum

size of elastomer to achieve maximum improvement in toughening a polymer.

It should also be pointed out that only when the addition of a rubbery copolymer whose elongation at break is high a significant improvement in elongation at break of the modified blend results. A crystalline block or graft copolymer whose elongation at break is low would cause either a drop or only a little increase in elongation at break of the blend.

Fig. 6c shows the effect of the quantity of the four different block copolymers on the moduli of HDPE/sPS blends. Addition of copolymers to the blends results in a decrease in moduli, decreasing with increasing copolymer content. The phenomenon of decrease in modulus on addition of compatibilizer has been also observed in other blends [26,34]. The modulus of a blend depends mainly on that of the blend components besides the factor of interfacial adhesion. The copolymers used here are all elastomers whose modulus are almost two orders of magnitude lower than that of the blend components; hence, small increases in the weight fraction of this phase could significantly reduce the moduli of the blends.

Using a series of crystalline and noncrystalline SEB (SEBS) copolymers, whose EB block are crystalline or rubbery to modify LLDPE/aPS blends, Li [30] found that the former caused an increase in modulus of the blend, whereas the latter had the opposite effect. This result has been attributed to the different interfacial structures formed by the two kinds of the copolymers. Crystalline copolymers would result in a stronger interfacial adhesion than noncrystalline copolymers. Another factor, probably more important, is that the crystalline copolymers have much higher modulus than that of the noncrystalline, leading to an increase in moduli of the blends. Heikens [20] found that elastomeric block copolymer HPB-*b*-PS copolymer whose modulus is lower than the blend caused a decrease in modulus of the blend; the graft copolymers PS-*g*-PE whose modulus is corresponding with the blend did not have this effect. This is another evidence to support the above explanation.

3.3. Thermal analysis

The DSC crystallization and melting thermograms of HDPE/sPS blends compatibilized with varying amounts of K–M copolymer are shown in Figs. 7 and 8 with thermograms of neat HDPE and sPS and the uncompatibilized blends as references.

It is shown that the crystallization temperature, T_c , is 116°C for pure HDPE and 235°C for sPS. The T_c of HDPE remains a constant on blending with sPS, but the crystallization peak of HDPE component becomes narrower, suggesting a reduction in size distribution of HDPE crystallites. Addition of compatibilizers seems to have no influence on the T_c and half-width of HDPE in the blends.

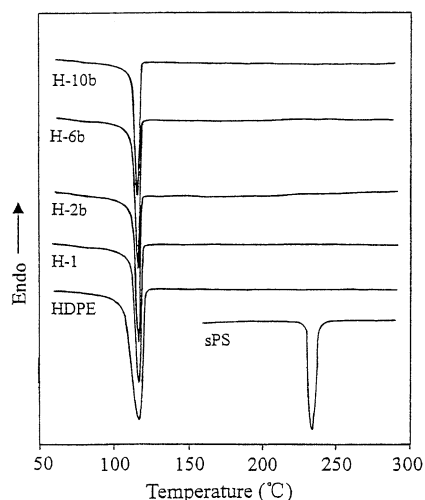


Fig. 7. DSC crystallization thermograms of HDPE/sPS (80/20) blends compatibilized with varying amounts of K–M. Cooling rate: 10°C/min.

A striking fact is that on the crystallization curves of all the blends only one crystallization exotherm at 116°C appeared with no sign of sPS crystallization at 235°C, though reheating curves showed the usual melting endothermic peaks of both HDPE and sPS. Such a phenomenon will be discussed elsewhere.

Table 3 presents the DSC data of non-isothermal crystallization and melting of unmodified and compatibilized HDPE/sPS blends as well as the plain HDPE and sPS. The melting points of HDPE and sPS in the HDPE/sPS blends are slightly depressed by 1–2°C compared to the plain HDPE and sPS. On blending, the crystallinity of HDPE increased significantly while that of sPS is lowered slightly. This suggests that incorporation of sPS into HDPE can promote crystallization of HDPE, i.e. HDPE crystallites

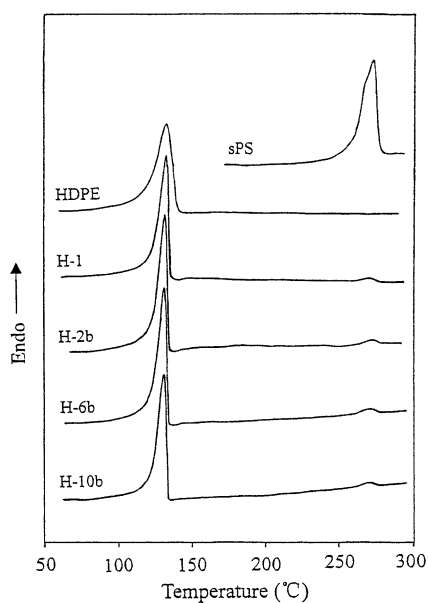


Fig. 8. DSC second heating thermograms of HDPE/sPS (80/20) blends compatibilized with varying amounts of K–M. Heating rate: 10°C/min.

Table 3

Thermal characterization of HDPE, sPS and HDPE/sPS (80/20) blends

Blend code	T_m (°C)		χ_c (%) ^a		T_c (°C)	
	HDPE	SPS	HDPE	sPS	HDPE	sPS
sPS	–	271.3	–	50.8	–	235.1
HDPE	132.2	–	64.7	–	116.3	–
H-1	131.1	269.7	70.0	49.5	116.3	–
H-2a	130.8	269.2	63.9	49.3	116.6	–
H-6a	130.9	269.1	61.5	49.3	116.2	–
H-10a	130.2	269.6	58.3	46.2	116.5	–
H-2b	131.2	269.1	67.1	50.4	116.1	–
H-6b	131.2	269.7	63.1	47.4	116.2	–
H-10b	131.2	269.9	59.4	34.9	115.8	–
H-2c	131.3	269.5	66.1	50.5	116.1	–
H-6c	131.4	269.4	64.4	47.2	116.0	–
H-10c	131.3	269.3	60.2	33.0	116.2	–
H-2d	131.0	269.2	66.9	51.9	116.4	–
H-6d	131.0	269.2	64.4	48.9	116.2	–
H-10d	131.0	269.5	59.4	38.5	116.1	–

^a Normalized crystallinity.

induce the simultaneous crystallization of sPS, which in turn promotes the crystallization of HDPE. The drop in crystallinity of sPS is obviously due to the presence of sPS as dispersed particles in HDPE where the crystallization ability of the former was suppressed. The normalized crystallinity of both HDPE and sPS in the blend decreases on addition of increments of the compatibilizers. The decrease in crystallinity of HDPE in the compatibilized blend is probably due to the penetration of the block copolymer into HDPE phase from the interface area when the compatibilizer concentration is lower. At higher compatibilizer concentration, the copolymer not only locates at the interface area but also disperses in HDPE matrix, which acts as a polymeric diluent, interfering with the crystallization of HDPE. The remarkably reduced size and the finer dispersion of sPS in the HDPE matrix on addition of the compatibilizer retards and suppresses the crystallization of sPS. Alternatively, the lowering of crystallinity of sPS may be caused by the penetration of the compatibilizer from the interface into the sPS domains. The synchronous reduction in crystallinity of both HDPE and sPS on addition of the compatibilizer is in favor of improvement in toughness of the blend. This is in agreement with the results discussed above. This effect is not of major importance in achieving high toughness of the blend, but could also make some contribution to the overall performance of these blends.

4. Heat resistance of the blend

The object of blending HDPE with sPS is to upgrade the useful temperature range of the blend. Vicat softening temperature of HDPE/sPS blends containing different amount of sPS and the HDPE/sPS (80/20) blends modified by varying amounts of the copolymers are presented in

Table 4
Vicat softening temperature of HDPE/sPS (with addition of 6 wt % K–H) blends

HDPE/sPS	Vicat softening temperature (°C)
100/0	121
90/10	130.5
80/20	142.5
70/30	163
0/100	254

Tables 4 and 5. The softening temperature of pure HDPE is only 121°C while that of the HDPE/sPS(80/20) blend is 142.5°C, which decreased only slightly with increasing compatibilizers content, indicating that the heat resistance of the blend is much higher than the neat HDPE. Obviously, the improvement in heat resistance of the blend arose from the higher softening temperature of sPS. The blends compatibilized by different copolymers exhibited roughly the same values of softening temperatures.

5. Conclusion

The four block copolymers of SEBS and SEB are effective in reducing the sPS domain size and improving the interfacial adhesion in immiscible HDPE/sPS blend. The medium molecular weight triblock copolymer K–M is the most effective, while the higher molecular weight triblock K–H displays relatively poor compatibilization. A dramatic improvement in tensile strength of HDPE/sPS (80/20) blends is observed on addition of a small amount of any of these copolymers except K–H, followed by a decrease as the copolymer concentration is increased. The elongation at break of the blends is significantly improved on addition of any the copolymers, most effective being that with K–H though the least in compatibilizing the blends. The modulus of the blends suffered a decrease on addition of the low modulus compatibilizers.

The mechanical properties of the HDPE/sPS blend depend not only on the size of the dispersed sPS and interfacial adhesion between the two phases but also on the compatibilizer content and the mechanical properties of the compatibilizers.

Addition of the compatibilizer to the HDPE/sPS blends

Table 5
Effect of compatibilizers on Vicat softening temperature of HDPE/sPS (80/20) blends

Blend code	Vicat softening temperature (°C)
H-1	144
H-2b	143
H-6b	142
H-10b	141.5
H-6c	141.5
H-6d	142

has little influence on the crystallization temperature of the HDPE but results in a dramatic reduction in crystallinity of both HDPE and sPS. Vicat softening temperature indicates that the heat resistance of HDPE has been improved by ~20°C on incorporation of 20 wt% sPS.

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