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Effects of molecular weight of SEBS triblock copolymer on the morphology, impact strength, and rheological property of syndiotactic polystyrene/ethylene–propylene rubber blends

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Abstract

The effects of molecular weight of atactic polystyrene-b-poly(ethylene-co-butylene)-b-atactic polystyrene [SEBS] triblock copolymers on the morphology, impact strength, and rheological property of syndiotactic polystyrene (sPS)/ethylene–propylene rubber (EPR) blends are examined. From the morphological analysis, it is observed that the size of dispersed EPR phase in sPS/EPR/SEBS blends decreases and the particle size distribution becomes narrower with increasing amounts of SEBS in the blends. It is found that the low molecular weight SEBS is more effective in increasing the impact strength of sPS/EPR blend than the high molecular weight SEBS. The SEM micrographs after impact test show that the sPS/EPR blends compatibilized by the low molecular weight SEBS have a good adhesion between the sPS matrix and dispersed EPR particles, whereas the sPS/EPR blends compatibilized by the high molecular weight SEBS exhibit a poor adhesion between phases. Although the complex viscosity of low molecular weight SEBS is much lower than that of high molecular weight SEBS, the complex viscosity of the sPS/EPR blends compatibilized by the low molecular weight SEBS is higher than that of the sPS/EPR blends compatibilized by the high molecular weight SEBS. It is suggested from these results that the blocks in the low molecular weight SEBS penetrate into the corresponding phase more easily than the blocks in the high molecular weight SEBS. © 1999 Elsevier Science Ltd. All rights reserved.

Keywords: Syndiotactic polystyrene; SEBS copolymer; Compatibilization

1. Introduction

Blending of polymers provides an efficient way of developing new materials with tailored properties and thus has received much attention from both academia and industry [1–19]. However, most of polymer blends are immiscible at the molecular level, because the combinatorial entropy of mixing of two polymers is drastically smaller than that of low molecular weight mixtures, whereas the enthalpy of mixing is often positive or near zero. The incompatibility between polymeric pairs is responsible for poor mechanical properties of most polymer blends due to poor phase morphology. Therefore, modification of polymer blends by interfacially active compatibilizers has extensively been investigated to obtain polymer blends with more desirable properties. Generally, an effective compatibilizer reduces the interfacial tension between the two phases and thus leads to a decrease in the particle size. At the same time, the compatibilizer increases interfacial adhesion by coupling the two phases together, and stabilizes the phase morphology against coalescence. To increase the compatibility of immiscible polymer blends, a number of strategies have been used. One of the most frequently used methods is to add a third component which is totally or partially miscible (or at least compatible) with both phases. The component may be a homopolymer or a suitable block or graft copolymer. It is well known that a block copolymer is an efficient compatibilizer for immiscible polymer blends [9–19]. Here each block of a diblock or triblock copolymer is usually either miscible, or has strong affinities, with one of the two homopolymer phases. Therefore, the block copolymer will preferentially locate at the interface between the two phases, thus reducing the interfacial tension and enhancing adhesion between the phases.

As syndiotactic polystyrene (sPS) with a very high degree of stereospecificity ($> 96\%$) has successfully been synthesized by stereospecific polymerization [20], characterization of this new material has been of considerable interest [21–30]. The sPS is a new crystalline polymer with a high melting temperature ($> 260°C$). The main advantages of sPS are its high heat resistance, excellent chemical

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Table 1
Characteristics of all polymers used in this study

Sample	Source	M_w (g/mol)	M_n (g/mol)	T_g (°C)	T_m (°C)
sPS	Synthesized[a]	122 500[b]	49 000[b]	98	262
EPR[c]	Aldrich	170 000	–	–	–
KG—H[d]	Shell	–	174 000	− 42	–
KG-L[e]	Shell	–	50 000	− 42	–

[a] Polymerized by $CpTiCl_3$/MAO at 25°C.

[b] Determined from GPC at 135°C using 1,2,4-trichlorobenzene.

[c] Poly(ethylene-co-propylene): ethylene content = 60%.

[d] Kraton G1651:SEBS triblock copolymer; styrene block content = 33%.

[e] Kraton G1652:SEBS triblock copolymer; styrene block content = 30%.

resistance, high degree of crystallinity, low specific gravity, and good dielectric properties. Further, the crystallization rate of sPS from the melt is about two orders of magnitude faster than that of isotactic polystyrene under similar supercooling conditions [29], and thus sPS can be used for a number of forming operations such as injection molding and extrusion. Because of the excellent properties described above, sPS is now considered as a potential engineering thermoplastic used for electrical/electronics, automotive, and industrial films. Although sPS has many desirable properties as an engineering thermoplastic, its disadvantage is its low impact strength. Therefore, an improvement of impact strength of sPS is essential to expand its applications. However, studies on this subject are still lacking. Blending of sPS with other rubbers can be a convenient way to increase the impact strength of sPS. As the melt-processing temperature of sPS is very high (usually over 280°C) due to its high melting temperature, the rubbers for blending should be thermally stable at that temperature range. For this reason, ethylene–propylene rubber (EPR), a random copolymer of ethylene and propylene, is chosen as a rubber component. However, sPS is immiscible with EPR due to lack of specific interaction between these two polymers. Hence, an effective compatibilizer for sPS/EPR blends is required to increase the compatibility. It is well known that a triblock copolymer of atatic polystyrene (aPS)-b-poly(ethylene-co-butylene)-b-aPS[SEBS], which is easily prepared by hydrogenation of a triblock copolymer of aPS-b-polybutadiene-b-aPS, has excellent thermal stability at high temperature and that the poly(ethylene-co-butylene) (EB) block in SEBS is compatible with EPR [13,14]. In contrast, it has been very recently reported that sPS is miscible with aPS by means of either enthalpy relaxation and crystallization kinetics [31,32] or diffusion experiments [33]. Therefore, the SEBS is expected to be a potential compatibilizer for sPS/EPR blends, as the aPS and EB block of SEBS is miscible with sPS and EPR, respectively.

In this study, the effects of SEBS triblock copolymer on the morphology, impact strength and rheological property of sPS/EPR blends are examined as functions of the amount of addition and molecular weight of SEBS triblock copolymers.

2. Experimental

2.1. Materials

The sPS was synthesized by polymerizing styrene (Junsei Chemical Co., 99.5%) using $CpTiCl_3$(Cp, cyclopentadienyl)/methylaluminoxane (MAO) catalyst [20,21]. $CpTiCl_3$ and MAO were purchased from Aldrich Chemical Co. MAO was provided as a 10 wt.% solution in toluene and was used as received without further purification. The polymerization was performed in a stirred glass flask at 25°C under vacuum with an [Al]/[Ti] molar ratio of 2000, and stopped by adding acidified methanol. The sPS was washed several times with fresh methanol and dried in vacuo at 40°C. As-polymerized powders were purified by extraction with a boiling acetone for 10 h in a Soxhlet extractor, and then dried in vacuo at 40°C. The acetone insoluble fraction of sPS was 96%. The number (M_n) and weight-average molecular weights (M_w) of sPS were measured by gel permeation chromatography (Waters 150C) at 135°C using 1,2,4-trichlorobenzene as a solvent. EPR is a commercial polymer containing about 60% of ethylene and was purchased from Aldrich Chemical Co. Two commercial SEBS triblock copolymers (Kraton G1651 and Kraton G1652; hereafter denoted as KG-H and KG-L, respectively) were supplied by Shell Co. The number-average molecular weights of KG-H and KG-L are 174 000 and 50 000 g/mol, respectively [13]. They are essentially monodisperse. The KG-H and KG-L contain about 33 and 30% styrene, respectively. Characteristics of all polymers used in this study are listed in Table 1.

2.2. Blends preparation

All the polymer samples were completely dried in a vacuum oven at 60°C for at least five days before blending. The samples were melt-blended at 300°C for 15 min and

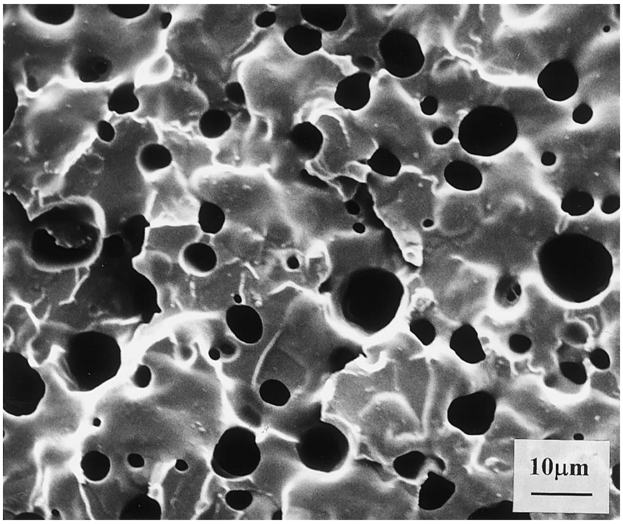

Fig. 1. SEM micrograph of cryogenically fractured surface of uncompatibilized sPS/EPR blend (magnification: 1000×).

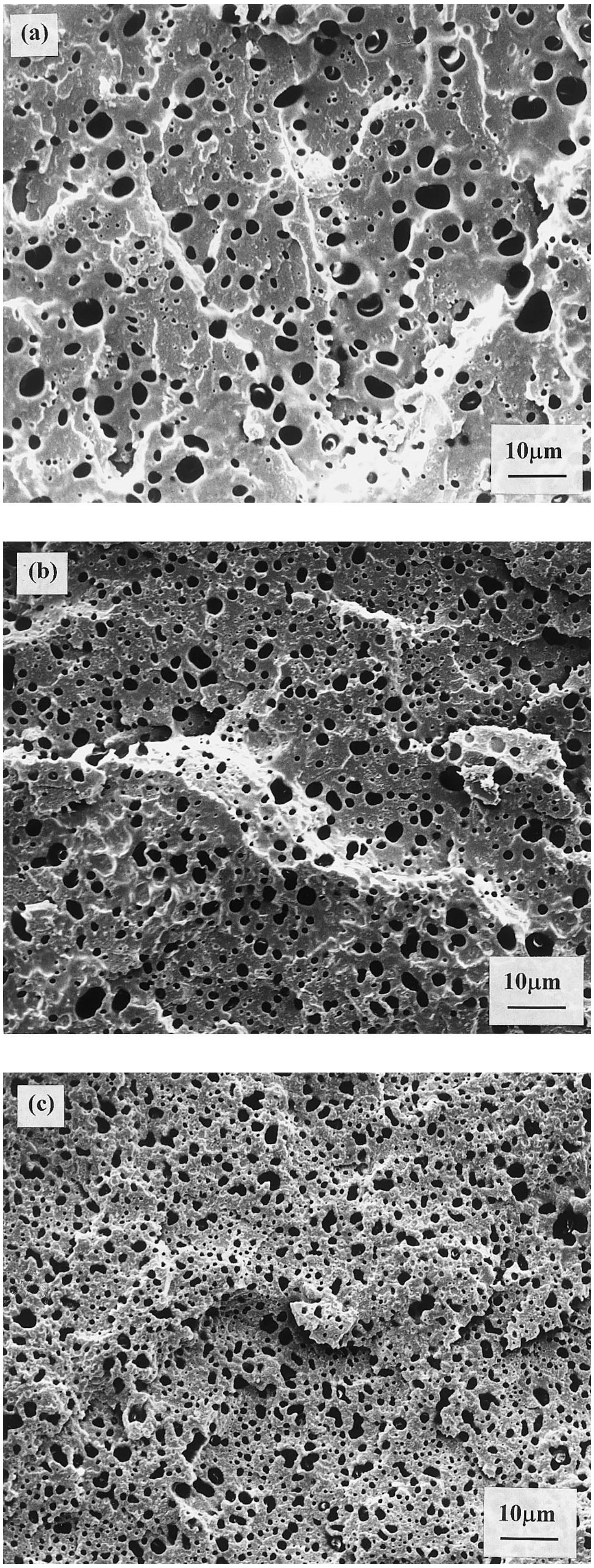

Fig. 2. SEM micrographs of cryogenically fractured surfaces of sPS/EPR/KG-L blends: (a) 2 wt.% KG-L added; (b) 5 wt.% KG-L added; (c) 10 wt.% KG-L added (magnification: 1000×).

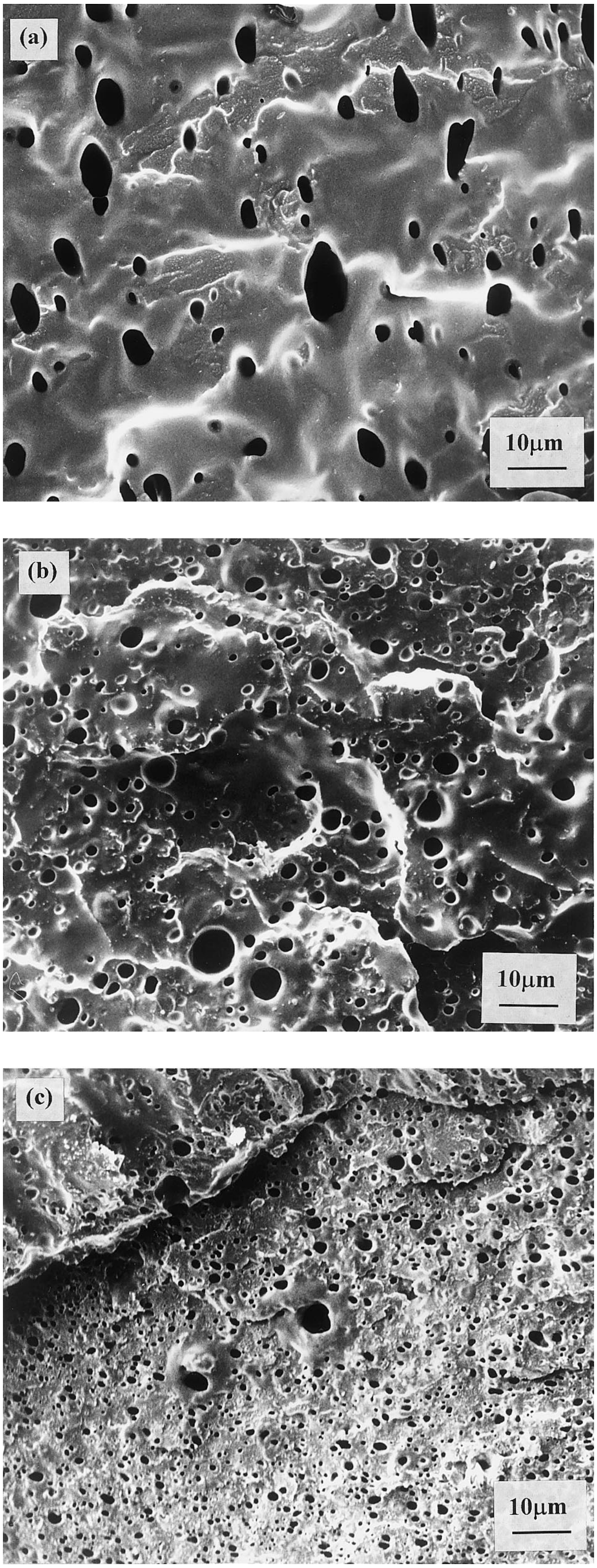

Fig. 3. SEM micrographs of cryogenically fractured surfaces of the sPS/EPR/KG-H blends: (a) 2 wt.% KG-H added; (b) 5 wt.% KG-H added; (c) 10 wt.% KG-H added (magnification: 1000×).

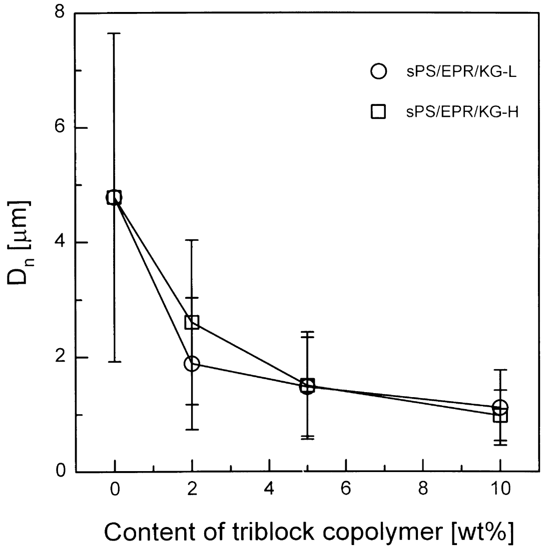

Fig. 4. Number-average diameter (D_n) of dispersed EPR particles versus content of triblock copolymer in sPS/EPR blends. Circles and squares indicate the sPS/EPR blends compatibilized by KG-L and with KG-H, respectively.

injection-molded into dumbbells (for the impact test) or disks (for rheological measurement) in a Mini-Max molder (CS-MMX, Custom Scientific Instruments, Inc.). The composition of sPS/EPR blend was fixed at 80/20 (w/w) in this study. The weight percentage of the added triblock copolymers with respect to the total weight of sPS/EPR blend was varied 0, 2, 5 or 10 wt.%.

2.3. Morphological observation

The morphology of samples was observed using a Hitachi S-2500C scanning electron microscope (SEM) at an accelerating voltage of 10 kV. The dumbbells were fractured perpendicular to the machine flow direction in liquid nitrogen. The cryogenically fractured surface was etched with *n*-hexane to remove the EPR phase and coated with gold to avoid charging. The SEM micrographs were analyzed to determine the average particle size and particle size distribution of the dispersed EPR phase by an image analyzer with the Ultimage software. The Waddel diameter (diameter of a circle having equivalent area of a non-round shape) of each particle was computed. An average of 300–500 diameter measurements per sample was reported. The number-average diameter (D_n) of dispersed EPR particles was calculated from the following relation:

$$D_n = \frac{\sum N_i D_i}{\sum N_i},$$

where N_i is the number of particles having a diameter D_i. The morphology of samples after the impact test was examined without etching. All micrographs were taken at the core region of samples.

2.4. Impact test

The impact strength of dumbbell-type specimens was measured by a pendulum-type impact tester (CS-183TI-086, Custom Scientific Instruments, Inc.) at room temperature. The dimensions of the dumbbell-type specimens were 9 mm in length and 1.6 mm in diameter. For all measurements, the data were taken as averages of at least 12 tests (maximum and minimum values were discarded).

2.5. Rheological measurement

The rheological property of samples was measured at 300°C using a Rheometrics Mechanical Spectrometer (RMS-800) with parallel-plate fixture under a nitrogen atmosphere. Dynamic experiments were performed in an oscillatory shear with dimensions of 25 mm (diameter) and 1.5 mm (gap size). The frequency sweep tests were carried out from 0.1 to 100 rad/s. Before measuring the rheological property of samples, strain sweep tests at various frequencies were carried out to confirm that the applied strain did not exceed the limit of linear viscoelastic behavior.

3. Results and discussion

3.1. Morphology of cryogenically fractured surface

The morphology of cryogenically fractured surface of uncompatibilized sPS/EPR blend is shown in Fig. 1. The uncompatibilized sPS/EPR blend exhibits a typical morphology of an immiscible mixture: very large, coarse, and irregular domains are formed. Changes in morphology of sPS/EPR/KG-L blends with addition of KG-L, a low molecular weight triblock copolymer, are observed in Fig. 2. The particle size of the dispersed EPR phase in sPS/EPR/KG-L blends is decreased significantly with increasing amounts of KG-L in the blends. Further, when compared with the morphology of uncompatibilized sPS/EPR blend in Fig. 1, the sPS/EPR/KG-L blends show more regular and a finer dispersion of particles, indicating that the added KG-L block copolymer acts as an efficient compatibilizing agent.

When the KG-H, a high molecular weight triblock copolymer, is used as a compatibilizer, a change in morphology of sPS/EPR/KG-H blends is shown in Fig. 3. When 2 wt.% of KG-H is added to sPS/EPR blends, the particle size of the dispersed EPR phase is decreased, as shown in Fig. 3(a). However, when compared with the corresponding sPS/EPR/KG-L blends in Fig. 2(a), it reveals that the size of the dispersed EPR phase in sPS/EPR/KG-H blends is larger and more irregular. As the amount of KG-H block copolymer is increased more than 2 wt.%, the dispersed particle size of both sPS/EPR/KG-L and sPS/EPR/KG-H blends is decreased drastically, as shown in Figs. 2 and 3.

The variation in number-average particle diameter for

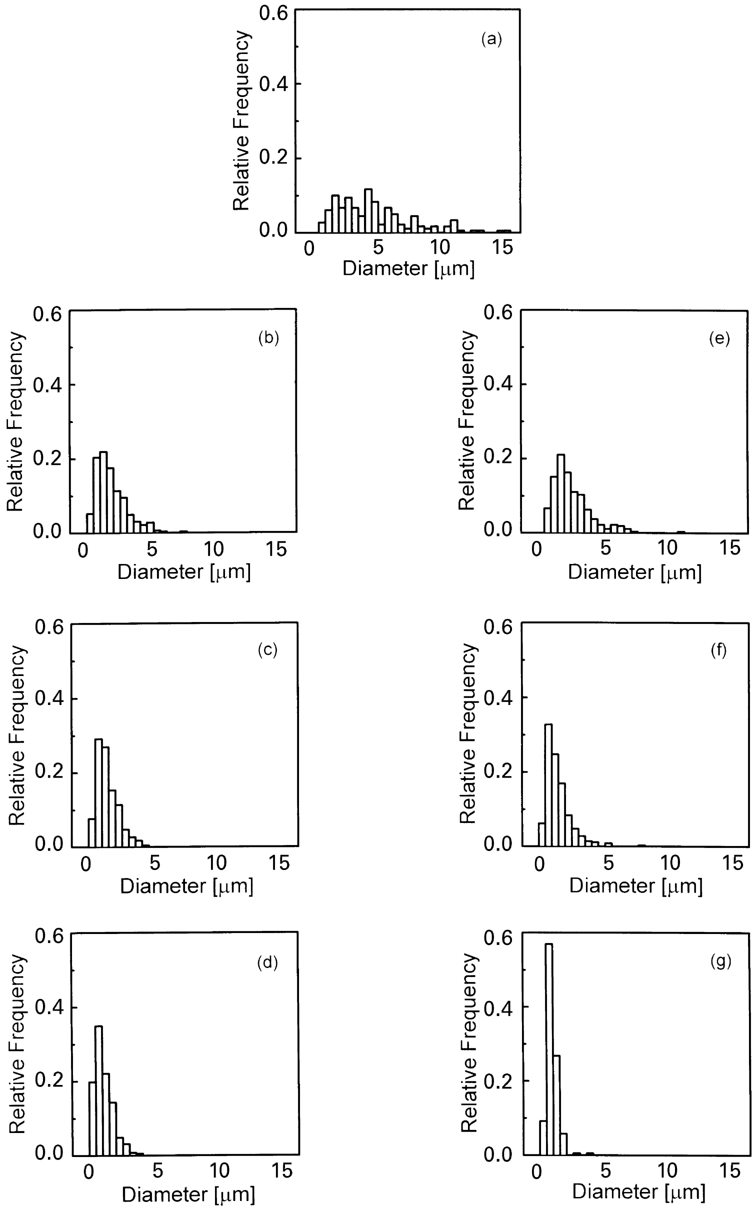

Fig. 5. Particle size distribution of dispersed EPR phase for sPS/EPR blends: (a) without compatibilizer; (b) 2 wt.% KG-L added; (c) 5 wt.% KG-L added; (d) 10 wt.% KG-L added; (e) 2 wt.% KG-H added; (f) 5 wt.% KG-H added; (g) 10 wt.% KG-H added.

sPS/EPR blends with the amount of block copolymer is shown in Fig. 4. It is found that the addition of both KG-L and KG-H block copolymers to the sPS/EPR blend results in a decrease in the dispersed particle size. In the case of sPS/EPR/KG-L blends, an addition of 2 wt.% KG-L causes a significant reduction in the domain size by about 61%. Further addition of KG-L block copolymer does not reduce the domain size considerably, but levels off above 2 wt.%. In contrast to sPS/EPR/KG-L blends, sPS/EPR/KG-H blends show a continuous decrease in D_n up to the addition of 10 wt.% KG-H. This indicates that, at low concentration of block copolymer, the KG-L is more efficient in reducing the dispersed particle size than the KG-H.

The effect of triblock copolymer addition on the particle size distribution of sPS/EPR blends is also examined. The uncompatibilized blend shows a broader distribution in particle size, as shown in Fig. 5(a). When 2 wt.% of KG-L or KG-H is added to the blend, the particle size distribution becomes narrower. By increasing the concentration of the block copolymers (KG-L and KG-H) up to 10 wt.%, the polydispersity of size distribution decreases as evident from the decrease in the width of the distribution curve. It has been also reported in the literature [6,34] that the addition of a compatibilizer to immiscible polymer blends not only reduces the size of the minor phase, but it also results in uniform size distribution.

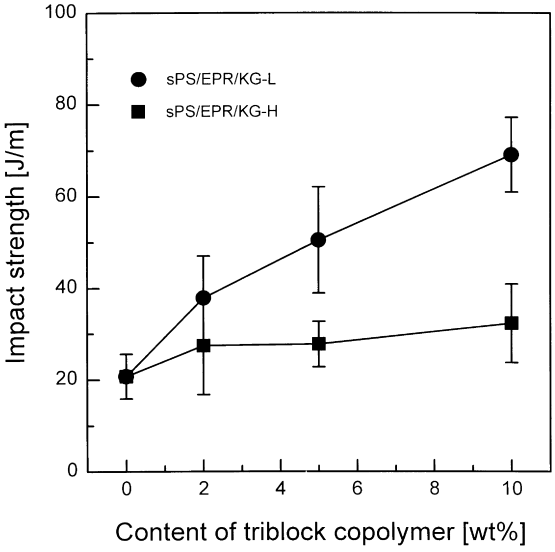

Fig. 6. Impact strength versus content of triblock copolymer for sPS/EPR blends. Circles and squares indicate the sPS/EPR blends compatibilized by KG-L and by KG-H, respectively.

3.2. Impact strength

The effect of triblock copolymer addition on the impact strength of sPS/EPR blends is examined. As shown in Fig. 6, the impact strength of sPS/EPR blends compatibilized with KG-L increases about 3.3 times higher than that without block copolymer, implying that the KG-L effectively improves the interfacial adhesion between sPS and EPR phases. This result leads us to speculate that three blocks in the SEBS triblock copolymer (in this study, KG-L) form strong entanglements, such as ‘hairpin loops’, at the interface region [18,35]. When the KG-H is added to sPS/EPR blend, however, the impact strength of the blends increases marginally. This indicates that the high molecular weight triblock copolymer (KG-H) is not so effective in increasing the impact strength as the low molecular weight one (KG-L).

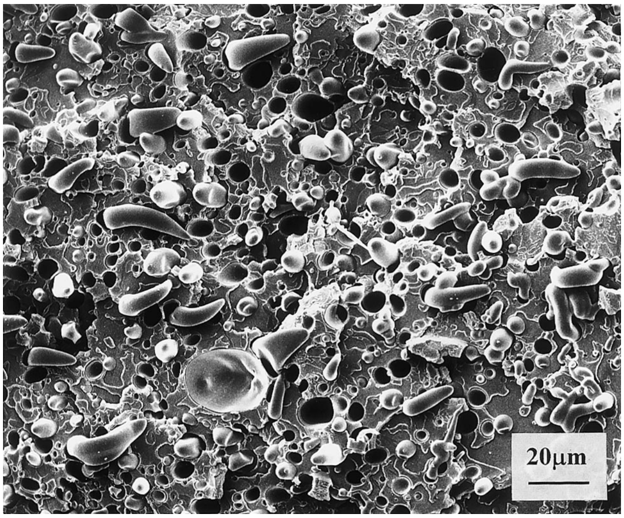

Fig. 7. SEM micrograph of fractured surface of uncompatibilized sPS/EPR blend after impact test.

The morphology of fractured surface of the uncompatibilized sPS/EPR blend after impact test is shown in Fig. 7. The fractured surface of the uncompatibilized blend exhibits a typical morphology of blend with low impact strength: the shape of particles is round and the particles are easily pulled out, implying a poor adhesion between sPS matrix and dispersed EPR phases. Fig. 8 shows a variation in morphology of fractured surfaces of sPS/EPR/KG-L blends after impact test as a function of triblock copolymer content. As the amount of KG-L in the sPS/EPR/KG-L blends increases, the fractured surface of the blends becomes rougher. Moreover, the EPR particles seem to adhere strongly to the sPS matrix, resulting in an improved impact strength of compatibilized blends. Fig. 9 shows SEM micrographs of fractured surfaces of sPS/EPR/KG-H blends after impact test. In contrast to sPS/EPR/KG-L blends, the sPS/EPR/KG-H blends show that the shape of the dispersed EPR particles is round and the particles are easily pulled out, indicating a poor adhesion between the sPS matrix and dispersed EPR particles. This may be responsible for the low impact strength of the sPS/EPR/KG-H blends in comparison with sPS/EPR/KG-L blends.

3.3. Rheological property

The flow properties of polymer blends depend, among other factors, on the morphology and on the interfacial properties. A modification of morphology by added compatibilizers, e.g., reduction in particle size, can result in a change in flow properties of the blends. As the rheological behavior of immiscible polymer blends is generally very complex, the effect of copolymer addition on rheological properties of such blends remains controversial [9,36,37]. To examine the effect of addition of KG-L on the rheological property of sPS/EPR blends, the magnitude of complex viscosity of the blends at 300°C is plotted against frequency, as shown in Fig. 10. It is observed that the magnitude of complex viscosity of sPS/EPR/KG-L blends increases significantly with increasing the amount of KG-L in the blends. This is probably due to a coupling effect of the block copolymer—when the KG-L is added to the sPS/EPR blend, it gives better interfacial adhesion between the sPS matrix and dispersed EPR phase, as the aPS and EB blocks in KG-L penetrate into the sPS matrix and dispersed EPR phase, respectively. Therefore, the EPR phase, which has higher viscosity than sPS at 300°C, contributes significantly to the blend viscosity and thus results in an increase in complex viscosity of the blend. It is also noted that the uncompatibilized blend shows a typical Newtonian behavior, whereas the compatibilized blend containing 10 wt.% KG-L exhibits a non-Newtonian behavior.

The effect of KG-H addition on the magnitude of complex viscosity of sPS/EPR blends at 300°C is shown

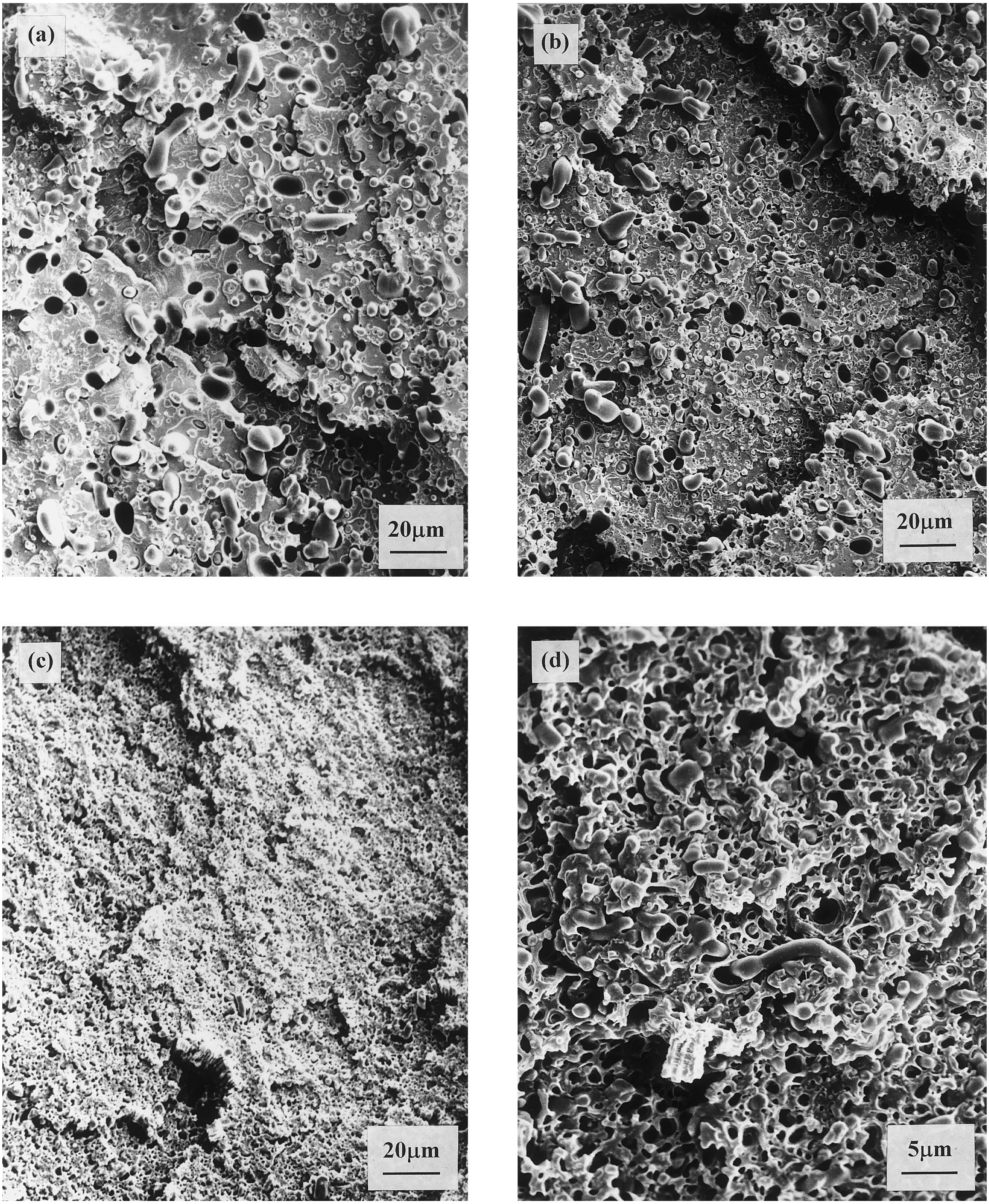

Fig. 8. SEM micrographs of fractured surfaces of sPS/EPR/KG-L blends after impact test: (a) 2 wt.% KG-L added; (b) 5 wt.% KG-L added; (c) 10 wt.% KG-L added; (d) 10 wt.% KG-L added (high magnification).

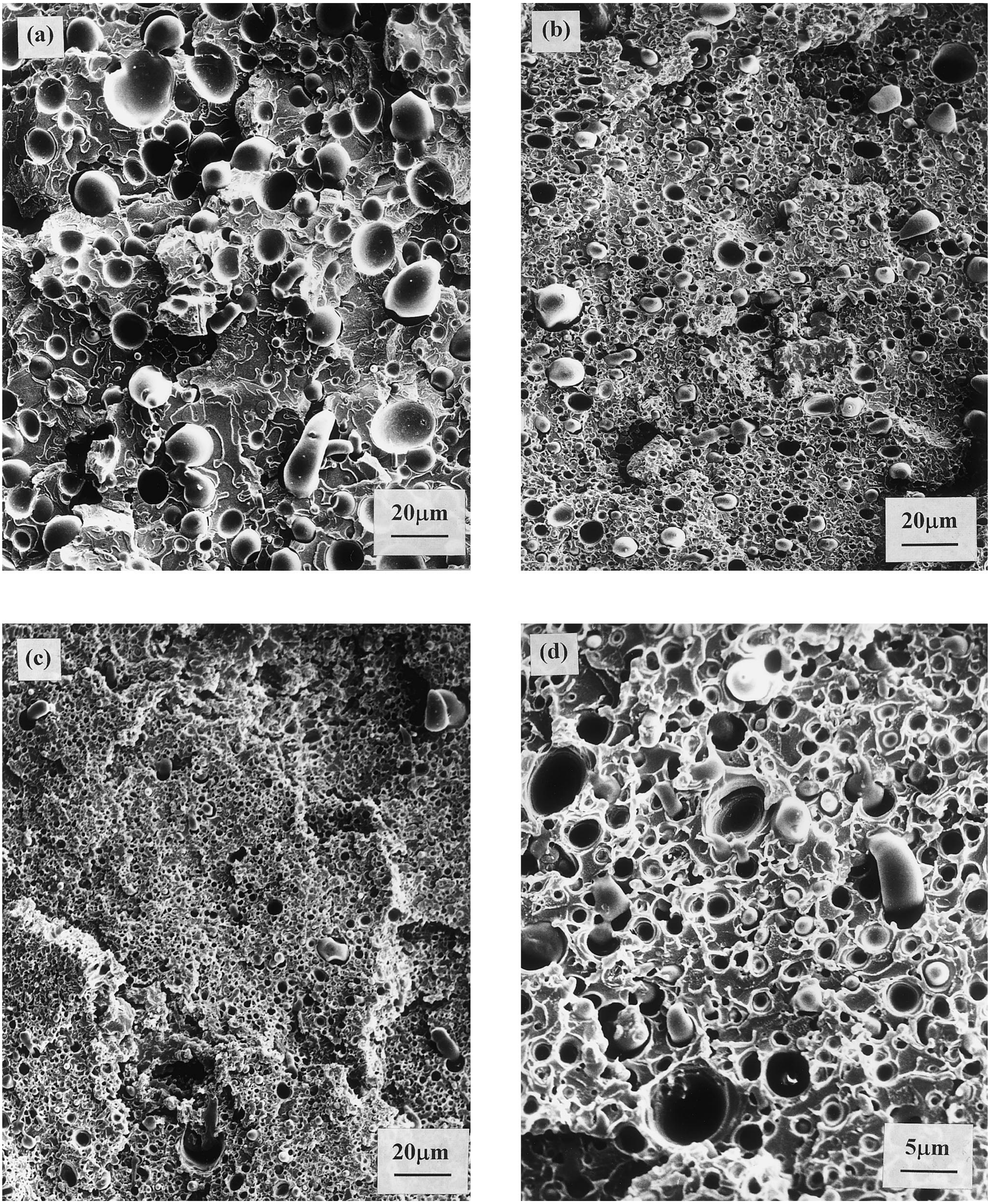

Fig. 9. SEM micrographs of fractured surfaces of sPS/EPR/KG-H blends after impact test: (a) 2 wt.% KG-H added; (b) 5 wt.% KG-H added; (c) 10 wt.% KG-H added; (d) 10 wt.% KG-H added (high magnification).

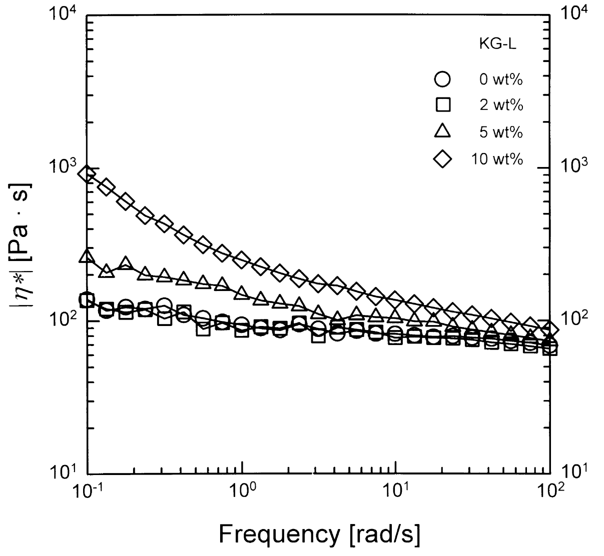

Fig. 10. Magnitude of complex viscosity versus frequency at 300°C for sPS/EPR/KG-L blends: (○) 0 wt.% KG-L added; (□) 2 wt.% KG-L added; (△) 5 wt.% KG-L added; (◇) 10 wt.% KG-L added.

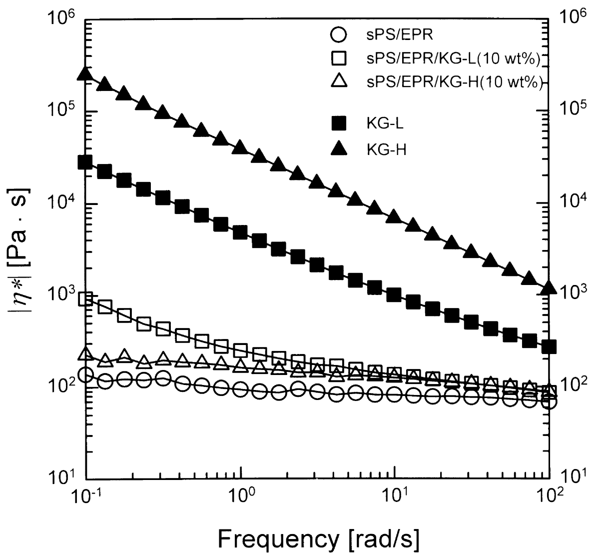

Fig. 12. Magnitude of complex viscosity versus frequency at 300°C for samples: (○) sPS/EPR blend; (□) sPS/EPR/KG-L(10 wt.%) blend; (△) sPS/EPR/KG-H(10 wt.%) blend; (■) neat KG-L; (▲) neat KG-H.

in Fig. 11. When 5 wt.% of KG-H is added the blend, the magnitude of complex viscosity slightly increases. However, unlike KG-L addition, a further increase in the content of KG-H does not increase significantly the viscosity of the blends. Further, the sPS/EPR/KG-H blends show a typical Newtonian behavior independent of the block copolymer content.

To examine the effect of molecular weight of triblock copolymer on the rheological property of sPS/EPR blends, the magnitude of complex viscosity for samples is plotted against frequency, as shown in Fig. 12. For comparison, the magnitudes of complex viscosity of neat triblock copolymers (KG-L and KG-H) are also shown. When the same amount of block copolymers (10 wt.%) is added to the sPS/EPR blend, it reveals that the complex viscosity of the blend compatibilized with KG-L is higher than the blend compatibilized with KG-H, particularly at low frequency range, although the complex viscosity of KG-L is much lower than that of KG-H. This behavior can be attributed either to one of two factors or to their combination: (i) the rheological properties of block copolymers itself, (ii) the morphology change of the blend due to the addition of the block copolymers. In the present system, the factor associated with the blend morphology seems to be dominant, because the magnitude of complex viscosity of block copolymers adversely affects the viscosity of the blends. From these results, it is suggested that the coupling effect of the low molecular weight compatibilizer (KG-L) is greater than that of the high molecular weight one (KG-H).

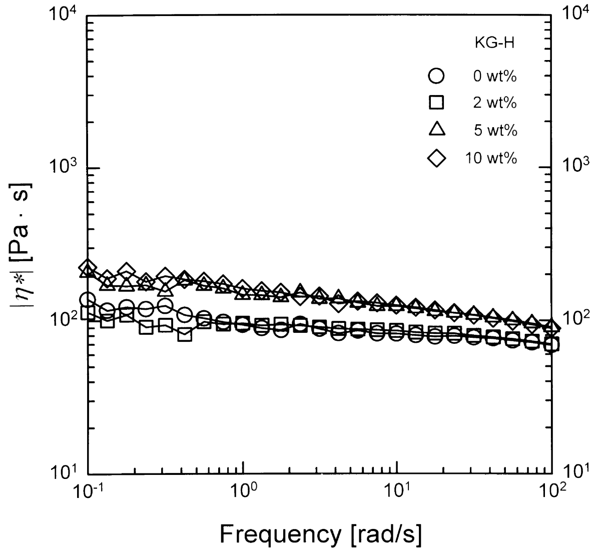

Fig. 11. Magnitude of complex viscosity versus frequency at 300°C for sPS/EPR/KG-H blends: (○) 0 wt.% KG-H added; (□) 2 wt.% KG-H added; (△) 5 wt.% KG-H added; (◇) 10 wt.% KG-H added.

From the rheological property and impact strength of sPS/EPR/KG blends, it is suggested that the efficacy of KG-L to improve interfacial adhesion between the sPS and EPR phases is much greater than that of KG-H. Although it is generally expected that higher molecular weight block copolymer would give higher interfacial adhesion due to deeper anchoring of each block into each of the phases, the opposite behavior is observed in this study. The better efficacy of low molecular weight compatibilizers to a high molecular weight one was also reported previously for aPS/EPR/SEBS blends [14]. Favis et al. [14] reported that the low molecular weight SEBS is more effective for increasing the impact strength of aPS/EPR blend than the high molecular weight SEBS, and concluded from morphological analysis that the low molecular weight SEBS migrates more effectively to the interface than does the high molecular weight SEBS. In the present sPS/EPR/SEBS system, however, this could not explain the observed behavior, as the ability of high molecular weight SEBS (KG-H) to migrate to the interface between sPS and EPR phases is nearly the same as that of low molecular weight SEBS (KG-L), as shown in Fig. 4. Therefore, the results in this study might be related to the difference in the 'penetration' ability of each block into the corresponding phase rather than the migration ability of the block copolymers to the interface. For sPS/EPR/KG-L blends, the KG-L, despite its short chain and low molecular weight, seems to readily penetrate into each phase of sPS and EPR due to its lower viscosity. Thus, the KG-L gives higher interfacial adhesion between phases, resulting in a blend with high impact strength. In the case of the sPS/EPR/KG-H blends, however, it is believed that the KG-H has too high molecular weight to penetrate effectively into each phase. As a result, the sPS/EPR/KG-H blends exhibit a poor interfacial adhesion and thus has low impact strength.

4. Conclusions

The effect of molecular weight of SEBS triblock copolymers on the morphology, impact strength, and rheological property of sPS/EPR blends is examined. From the morphological analysis, it is found that, for both blends compatibilized by low (KG-L) and high molecular weight SEBS (KG-H), the size of the dispersed EPR phase in the sPS/EPR blends decreases and the particle size distribution becomes narrower with increasing the amount of the block copolymers in the blends. The impact strength of sPS/EPR/KG-L blends is increased significantly by increasing the content of KG-L, whereas the impact strength of the sPS/EPR/KG-H blends is increased slightly by increasing the content of KG-H. The SEM micrographs after the impact test show that the sPS/EPR/KG-L blends have a good adhesion between the sPS matrix and dispersed EPR particles, whereas the sPS/EPR/KG-H blends exhibit a poor adhesion between the two phases. Although the complex viscosity of neat KG-L is lower than that of neat KG-H, the complex viscosity of sPS/EPR/KG-L blend is higher than that of the sPS/EPR/KG-H blend, indicating that the coupling effect of KG-L is greater than that of KG-H. From these results, it is concluded that the blocks in the low molecular weight SEBS (KG-L) penetrate into each corresponding phase more easily.

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