Grafted Syndiotactic Polystyrene Synthesized via Melting Graft Copolymerization Used as a Compatibilizer for Immiscible Syndiotactic Polystyrene/Polyadimide 66 Blends

Youming Cao,[1] Zhigang Shen,[2] Shang'an Lin[2]

[1]*Department of Polymer Science, School of Materials and Energy, Guangdong University of Technology, Guangzhou 510263, People's Republic of China*
[2]*Institute of Polymer Science, School of Chemistry and Chemical Engineering, Zhongshan University, Guangzhou 510275, People's Republic of China*

ABSTRACT: A new grafted syndiotactic polystyrene (*g*-sPS), to be used as a compatibilizer for syndiotactic polystyrene (sPS)/polyadimide 66 blends, was prepared by the melting graft copolymerization of sPS and monomers composed of itaconic acid and dibutyl maleate with dicumyl peroxide as an initiator. The resulting *g*-sPS possessed a side-chain structure identified by IR spectra, and the results of mechanical testing show that a good impact strength and tensile strength were obtained for *g*-sPS at a 7.16-phr addition of monomer with a 3:1 proportion of dibutyl maleate and itaconic acid. Differential scanning calorimetry and scanning electron microscopy analysis indicated that the *g*-sPS maintained a high glass-transition temperature and a crystalline structure. © 2005 Wiley Periodicals, Inc. J Appl Polym Sci 97: 1659–1666, 2005

Key words: syndiotactic; polystyrene; graft copolymers

INTRODUCTION

Polymer blending has been the subject of intense research in both academics and industry. In recent years, the new semicrystalline polymer syndiotactic polystyrene (sPS) has attracted much interest because of its unique physical properties.[1–6] It has a completely ordered chain configuration, which is endowed with (1) a high melting temperature ($T_m \approx 270$°C), (2) a rapid crystallization rate, and (3) a controllable crystallinity (X_c). Therefore, sPS exhibits not only good chemical resistance but also enhanced mechanical performance at elevated temperatures.[7–12] However, it has some shortcomings, such as its brittleness and poor impact and tear resistances. For sPS to be of commercial use, the practice of blending with an elastomer, just as in the toughening of polypropylene,[13,14] seems to be the a good method.

Many commercial rubbers could be used to the improve impact strength (σ_I) at low temperatures and the environmental stress cracking resistance of a brittle polymer. Thermoplastic polyurethanes are an important class of elastomers that has found many novel and specialized applications where high mechanical and chemical performances are prerequisites. They are known for their good mechanical strengths and wear and tear resistances. and low temperature elasticity.[15,16] That is, thermoplastic polyurethanes are excellent candidates for polymer blending.[17,18]

To decrease the brittleness and increase the impact and tear resistances of sPS, the technique of polymer alloys is one effective method. However, if the two polymers are immiscible, the mechanical properties of the resulting blends would not to satisfying because of poor compatibility resulting from a low interphase adhesive force. A compatibilizer is recognized as an interphase active reagent and has been distributed in the interphase of two-phase blends, where the adhesive force is enhanced significantly.[19–23] A block or graft copolymer would be the first choice as a compatibilizer.

A variety of block copolymers of styrene and other monomers have been synthesized by living stable free-radical polymerization, including polystyrene (PS)-*block*-polycaprolactone,[24] PS-*block*-poly(styrene sulfonic acid),[25] PS-*block*-poly(*p*-bromostyrene).[26] Yoshida and Fujii[27] used a 4-methoxy derivative of 2,2,6,6-tetramethyl 1-1-piperidingyloxy (TEMPO) to polymerize methyl styrene in accordance with the living mechanism.

The syntheses of graft polymers with uniform grafts via the macromer technique have been reported in a

TABLE I
Materials Used in the Experiment

Material	Supplier	Grade	Format
sPS	Shanghai Research Institute of Petrochemical Engineering	Industry grade	$M_n = 17 \times 10^4$, $T_m = 273°C$
Itaconic acid	Xinhua Active Material Research Institute	Analysis pure	$T_m = 162–168°C$
Dibutyl maleate	Shanghai Chemistry Reagent Co. Ltd, China Physic Group	Analysis pure	bp = 280°C; ρ = 0.993–0.997 g/mL
Dicumyl peroxide	Shanghai Chemistry Reagent Co. Ltd. China Physic Group	Analysis pure	$T_m = 39–41°C$

M_n, number-average molecular weight; ρ = density.

lot of articles concerning the graft polymerization of double-bond-terminated macromers, including macromers of styrene, alkyl (meth)acrylate, and ethylene oxide.[28–34] Schulz and Milkovich[35] reported the synthesis of PS macromers through the termination of living PS anions with methacryloyl chloride and their copolymerization with butyl acrylate and ethyl acrylate. Rempp[36] and Masson et al.[37] reported the synthesis of polyoxyethylene macromers either with alkali alcoholate as the initiator and methacryloyl chloride as the terminating agent or with unsaturated alkali alcoholate as the initiator and alkyl chloride as the terminating agent. Ito and coworkers[38,39] obtained polystearyl methacrylate macromer through free-radical polymerization with thioglycollic acid as the chain-transfer agent followed by a reaction with glycidyl methacrylate. Bornaldi et al.[40] used thioglycol as the chain-transfer agent in the preparation of macromers of dodecyl methacrylate, followed by a reaction with methacryloyl chloride.

To broaden the application range of sPS, it is important to study the blending of sPS with other polymers, especially polar engineering plastics, such as polyadimide 66 (PA66). However, sPS is not compatible with PA66. The preparation of a compatibilizer for sPS/PA66 blends was the primary subject of this study.

So far, not many academic studies have been published on no-melting graft copolymers of sPS, although there were some similar graft copolymers synthesized via solution graft copolymerization.[41.42] Melting graft copolymers, such as grafted syndiotactic polystyrene (g-sPS), were desirable compatibilizers for sPS/PA66 blends, in which the prepared g-sPS was compatible with sPS on the basis of their similar molecular structure and in which the side chain of g-sPS with polar poly(dibutyl maleate-*block*-itaconic acid), especially the polar carboxylic group RCOOH, was expected to be miscible with polar polymers as PA66 because of specific interactions between them. By this method, the interphase adhesive force between sPS and PA66 was expected to increase, and the purpose of strengthening interphase adhesion for the sPS/PA66 blends was obtained. A report on the study of the sPS/PA66 blends will be published in a later work.

In this study, we attempted to synthesize a novel graft copolymer composed of sPS, itaconic acid, and dibutyl maleate. Our investigations mainly focused on the identification of the resulting g-sPS and the effects of the addition of the total addition of monomers composed of itaconic acid and dibutyl maleate with various proportions on the σ_I, tensile strength (σ_T), and heat properties of the resulting g-sPS. Combined with the microstructural analysis of the fracture, the corresponding mechanisms are discussed.

EXPERIMENTAL

Materials

The materials used in this work are shown in Table I. Therefore, the sPS we used was an industry-scale product supplied by Shanghai Research Institute of Petrochemical Engineering (Shanghai, People's Republic of China).

Melting graft copolymerization of sPS

A mixture with a composition as shown in Table II was inserted into a small-type injector (Jilin University Science and Education Equipment Factory, People's Republic of China), heated to 280°C, and then maintained at 280°C for 25 min. Finally, the reacted mixture was injected into a small standard model with 50 mm $\times$ 5 mm $\times$ 4 mm in size. After it was cooled, the sample was taken off the model and used for property measurements and scanning electron microscopy (SEM).

TABLE II
Composition of Samples

Material	Composition (phr)
sPS	100
Itaconic acid + dibutyl maleate	0–18
DCP	1

Measurement of graft ratio

The graft ratio of the g-sPS was calculated from acid–base titration. The purification sample (0.2–0.3 g) was in circumfluence for 4 h and was dissolved completely in 100 mL of dimethyl benzene. The solution was cooled to about 60°C, and 10 mL of standardized methanolic NaOH (0.05N) solution was added, and then, the solution was reheated at circumfluence temperature for 2 h to fully hydrolyze the ester groups of dibutyl maleate. After the solution was cooled to about 60°C, the graft ratio was determined by titration with a standardized isopropylnolic HCl (0.05N) solution with phenolphthalein ethynol as an indicator. The graft ratio (Gra$_1$) with a unit in a grafted molar amount of carboxylic and ester groups (including itaconic acid and dibutyl maleate) per unit sPS segment was easily calculated as follows:

$$\text{Gra}_1 = \frac{(10N_1 - VN_2) \times 10^{-3}}{2w} \times 100\%$$

$$= \frac{(10 - V) \times 5 \times 10^{-5}}{2w/M} \times 100\% \quad (1)$$

where V is the consumed volume (mL) of isopropylnolic HCl solution, w is the mass (g) of purification g-sPS, $N1$ is the normality of NaOH solution, $N2$ is the normality of HCl solution, and M the molecular weight of the sPS unit segment (104).

Similarly, the graft ratio of itaconic acid (Gra$_2$) was determined by titration with a standardized methanolic NaOH (0.05N) solution before hydrolysis. The graft ratio of dibutyl maleate was Gra$_1$ − Gra$_2$.

IR spectroscopy

IR spectra of the obtained products were recorded on Avatar E. S. P. spectrometers (Nicolet, Ltd.) in the range 4000–400 cm^{-1}.

Measurement of mechanical properties

σ_I of the resulting g-sPS was measured by a charpy σ_I measurement set produced by VEB Verkstoffprufmaschinen Leipzig Co. (Germany) with unnotched specimens. The size of each specimen was 50 mm × 5 mm × 4 mm. σ_I was calculated as follows:

$$\sigma_T = \frac{P}{ab} \quad (2)$$

where P is the maximum impact force at the final failure of the samples and a and b are the width and thickness of the used specimen, respectively.

σ_T was measured with a Hounsfield 10K-S mechanical testing machine produced by England Test Equip-

ment, Ltd. Tensile velocity was controlled at 0.5 mm/min. The size of the testing specimens was the same as mentioned previously. σ_T was given by eq. (3):

$$\sigma_T = \frac{P_1}{ab} \quad (3)$$

where P_I is the maximum tensile load.

At least five specimens were used for tests of σ_I and σ_T.

SEM

The sonic fracture surfaces of the specimens cooled in liquid nitrogen were observed with a S-2700 scanning electron microscope (Hitachi, Japan), which was coated previously by sputtering with gold metal to achieve a conducting and protecting layer.

Differential scanning calorimetry (DSC)

Thermal analysis was made with a LABSYS TG-TDA-DSC (France) under an argon atmosphere. For samples containing sPS, the following procedure was used: samples were heated at 350°C for 5 min to eliminate the influence of thermal history and the effect of thermal treatment on the crystalline structure of sPS; they were then cooled to 70°C and reheated to 350°C, all at a rate of 20°C/min.

RESULTS AND DISCUSSION

Purification of g-sPS

Besides graft copolymerization, there were byproducts, such as poly(dibutyl maleate-*block*-itaconic acid), and homopolymers, such as poly(dibutyl maleate) and poly(itaconic acid), during the graft reaction of sPS, dibutyl maleate, and itaconic acid. Because the total addition of monomers was small (<18 phr), the molecular weight of these byproducts was not great. To identify whether the graft structure of sPS was produced or not, the resulting g-sPS was identified by IR spectroscopy. However, the produced byproducts needed to be eliminated before IR spectroscopy. The purifying process of g-sPS is shown in Figure 1. The resulting samples were first dissolved in tetrahydrofuran and then precipitated into methanol to remove the free dibutyl maleate and itaconic acid. The precipitate was completely dried and dissolved in ethyl acetate. The solution was stirred for 24 h to completely dissolve poly(dibutyl maleate-*block*-itaconic acid) and the homopolymers poly(dibutyl maleate) and poly(itaconic acid). The pure g-sPS was filtered and washed three times with ethyl acetate and then put into an oven, dried, and milled into powder. The small powder of g-sPS was added into a no-water potassium
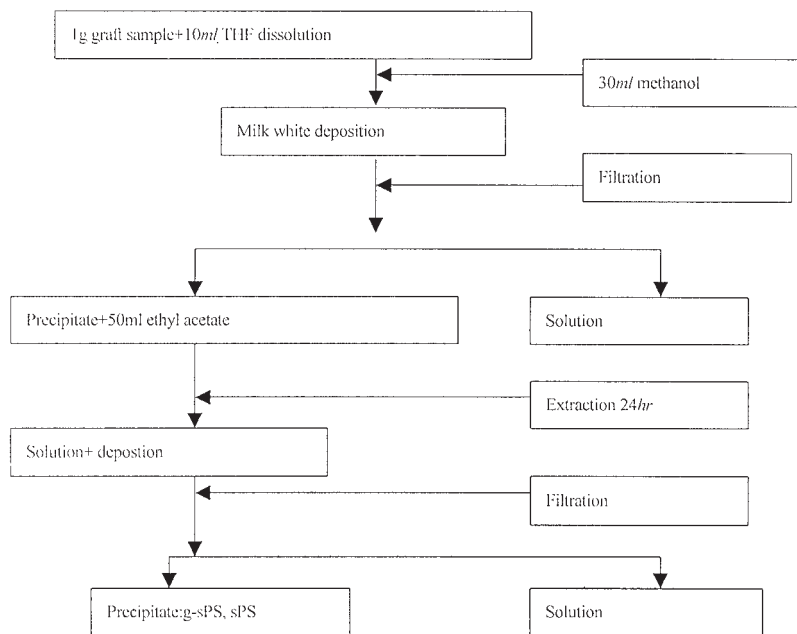
Figure 1 Solvent extraction of g-sPS.

bromide powder, pressed into pieces, and used for IR spectroscopy.

Structural identification of g-sPS

Three IR spectra corresponded with pure sPS, g-sPS1, and g-sPS2, as shown in Figure 2. The structure of g-sPS was identified by the characteristic absorbing peaks of each IR spectrum.

Figure 2(a) shows the high wave-number zone of IR spectra corresponding to sPS, g-sPS1, and g-sPS2. The characteristic absorbing peaks of sPS were found at 3061 and 3033 cm^{-1}, which were stretching vibrations of the =CH group, The framework vibration absorbing peaks appeared near 1601, 1493, and 1452 cm^{-1}.

Besides these same absorbing vibration peaks in the previous three IR spectra, there were remarkable differences as in wave-number positions: the strength of the absorbing peaks near 2848 and 2922 cm^{-1} were remarkably reinforced, which resulted from the increased amount of methylene (—CH$_2$—) and methyl (—CH$_3$) because the side chain contained a dibutyl structure: the stretching absorbing vibration of methylene (—CH$_2$—) near 2848 cm^{-1} and that of methyl (—CH$_3$) near 2922 cm^{-1}. Therefore, the absorbing strength of the two corresponding vibration peaks' position was increased.

Moreover, because the carboxylic group in itaconic acid was inducted onto a side chain, the characteristic absorbing peaks of the carboxylic group RCOOH emerged within 2500–3500 cm^{-1}. Because the mass absorbing peaks was in concurrence within this zone, which resulted in a high peak foundation in width and
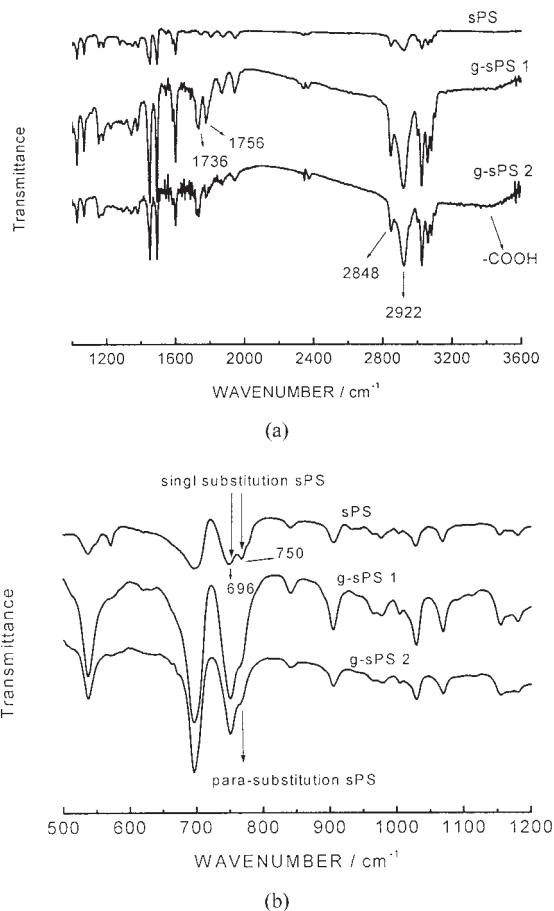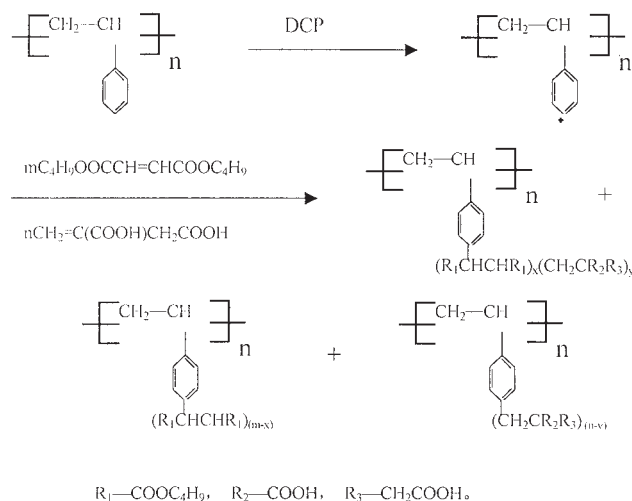

Figure 2 IR spectra of sPS and g-sPS at (a) high and (b) low wave-number zones: g-sPS1 with the addition of 6.0 phr (3A : 1B) monomer; g-SPS2 with the addition of 6.0 phr (1A : 1B) monomer (A = dibutyl maleate; B = itaconic acid).

Scheme 1

TABLE III
Graft Ratio (%) of g-sPS with Various Monomer Proportions

A + B (phr)	A : B		
	1 : 3	1 : 1	3 : 1
3.28	0.44 (0.30)	0.43 (0.19)	0.41 (0.10)
5.39	1.50 (0.93)	1.73 (0.81)	1.74 (0.42)
7.16	3.86 (2.89)	3.75 (1.64)	3.78 (0.91)
10.37	4.65 (3.38)	4.82 (2.10)	4.90 (1.18)
13.41	5.35 (3.96)	5.44 (2.48)	4.82 (1.21)
15.24	5.82 (4.55)	5.71 (2.44)	5.75 (1.64)

A, dibutyl maleate; B, itaconic acid. A + B is relative to 100 phr sPS. The unit of graft ratio was A + B mol/100 mol of styrene units. The data in parentheses denoted the graft ratio of B mol/100 mol of styrene units.

strength within the zone. Compared to *g*-sPS1, *g*-sPS2 contained a greater carboxylic content, which made the strength of the absorbing peaks within the corresponding absorbing zone in *g*-sPS2 higher.

The greatest difference emerged at the following positions: near 1756 and 1736 cm^{-1}. There were very strong absorbing vibration peaks in the spectra of both *g*-sPS1 and *g*-sPS2. The absorbing peak near 1756 cm^{-1} was the characteristic absorbing peak of the carboxylic group (C═O) in carboxylic acid (RCOOH) of a side-chain graft structure and that at 1736 cm was that of a carboxylic group (C═O) in a carboxylic ester (R$_1$COOR$_2$) of a side-chain graft structure. However, there were not strong absorbing vibration peaks in the spectrum of pure sPS. All this proved that the graft copolymerization of sPS by monomers composed of dibutyl maleate and itaconic acid was carried out on the side chain.

Figure 2(b) show the spectra of pure sPS, *g*-sPS1, and *g*-sPS2 at a low wave-number zone. This zone is the so-called fingerprint zone of PS and is very useful for the identification of the substitution position of PS. In the IR spectrum of pure sPS, there were two strong absorbing peaks emerging near 696 and 750 cm^{-1}, respectively, which indicated a single-substitution structure of PS and implied that the five bordering on hydrogen atoms in the benzene ring possessed two kinds of different absorbing peaks. However, the two peaks tended to be one peak in the spectra of *g*-sPS1 and *g*-sPS2. That is, a substituent reaction was carried out on the benzene ring of sPS. Combined with a view of the location obstruction effect, we deduced that the graft reaction was carried out at the para position in the benzene ring of sPS (Scheme 1).

As shown by the previous analysis, during the graft copolymerization of sPS, the produced active center should have been on the para position of the benzene ring of sPS and induced monomers to take place in the graft copolymerization, which formed the side-chain graft copolymers.

The graft ratio of *g*-sPS with various monomer proportions is listed in Table III. Table III shows that the graft ratio increased with increasing total addition of the monomers dibutyl maleate and itaconic acid. For each total addition with different proportions, the difference in the graft ratio was small. For example, when the total addition of monomer was 10.37 phr, the graft ratios were 4.65, 4.82, and 4.90%, respectively. Because of the hydrolysis period with ethynolic NaOH solution and because the hydrolysis products of both the ester group in dibutyl maleate or the carboxyl group in itaconic acid was RCOONa, the consumed hydrochloric acid volume was almost the same at pH 7 when the total addition of monomers was the same.

Moreover, the graft ratio of itaconic acid displayed a similar change. The measured graft ratio of itaconic acid was less than its theoretic value, which resulted from its less competitive ratio.

Mechanical properties of *g*-sPS

To look for the best composition of monomers composed of dibutyl maleate and itaconic acid, three proportions of dibutyl maleate to itaconic acid were cho-

TABLE IV
σ_I of g-sPS

A + B (phr)	A : B		
	1 : 3	1 : 1	3 : 1
3.28	2.90	2.52	3.36
5.39	2.94	2.73	3.44
7.16	3.7	2.86	4.41
10.37	2.65	2.52	3.87
13.41	2.35	2.44	2.71
15.24	2.22	2.12	2.22

A, dibutyl maleate; B, itaconic acid. A + B is relative to 100 phr sPS. σ_I of the pure sPS sample was 2.60 kJ/m^2.
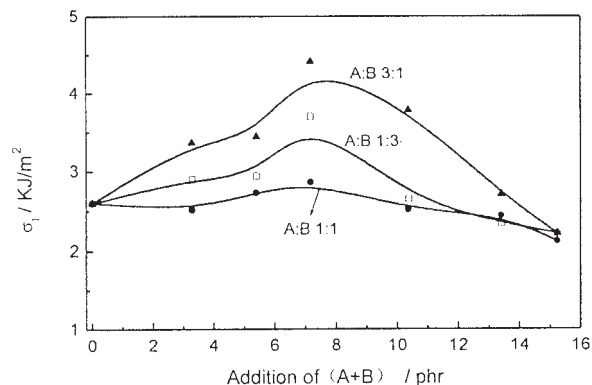
Figure 3 σ_I as a function of the addition of monomer with various proportions (A, dibutyl maleate; B, itaconic acid).

Figure 4 σ_T versus the addition of monomer at various compositions (A, dibutyl maleate; B, itaconic acid).

sen: 1 : 3, 1 : 1, and 3 : 1 (weight proportion). The total addition of dibutyl maleate and itaconic acid was changed in the different graft copolymerizations. However, other conditions were the same: the melting graft temperature was 280°C, the reaction time was 25 min, and there was a 1-phr addition of dicumyl peroxide (DCP). The σ_I of the resulting g-sPS are listed in Table IV, and the corresponding relationships between σ_I and the total addition of monomers is shown in Figure 3.

Table IV and Figure 3 show that σ_I first increased and then decreased with increasing monomer amount. The maximum σ_I value was concurrence at the point of 7.16 phr addition of monomers for the three proportion monomers. Therefore, when the proportion of dibutyl maleate to itaconic acid was 3 : 1 and the total addition of monomers was 7.16 phr, σ_I was the greatest.

σ_T of the resulting g-sPS with various monomer proportions are listed in Table V, and the relationships between σ_T and the addition of monomer are shown in Figure 4. σ_T increased with increasing addition of monomers, which resulted from increased flexible side-chain g-sPS.
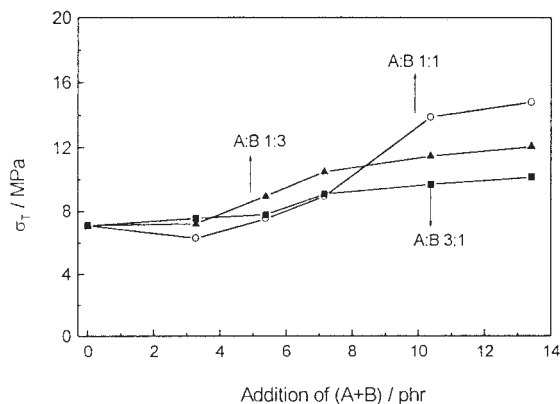
Heat properties and microstructure

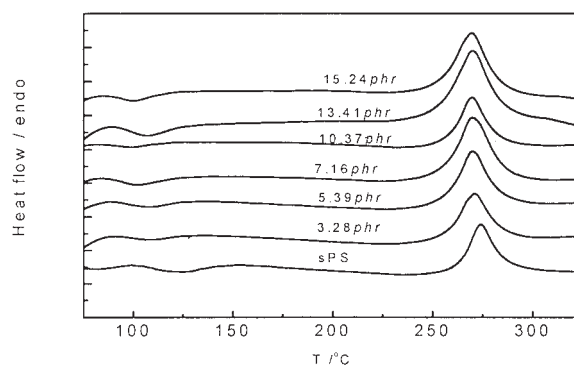g-sPS was used as a compatibilizer for the sPS/PA66 blends, which was expected to enhance the σ_I of the

sPS/PA66 blends. Therefore, we hoped that the resulting g-sPS would still have a high σ_I and σ_T. From the previous comprehensive analysis, we know that the proportion between dibutyl maleate and itaconic acid was best at 3 : 1.

Therefore, the proportion between dibutyl maleate and itaconic acid was chosen as 3 : 1, and the other graft conditions were the same. That is, the addition of the DCP initiator was done at 1 phr, the melting graft temperature was 280°C, and only the total addition of monomers was changed. The DSC of the resulting g-sPS with different additions of monomer is shown in Figure 5, and the corresponding heat properties are listed in Table VI.

In Table VI, the X_c values of sPS and g-sPS are given, as calculated by the following equation:

$$X_c = \frac{\Delta H_f / \Delta H_f^0}{w} \times 100\%$$

where ΔH_f is the melting enthalpy of the polymer, ΔH_f^0 is the melting enthalpy of 100% crystalline sPS ($\Delta H_f^0 = 53 \text{ J/g}$),[7] and w is the weight fraction of sPS in the blend.

TABLE V
σ_T (kg/m²) of g-sPS with Various Monomer Proportions

A + B (phr)	A : B		
	1 : 3	1 : 1	3 : 1
3.28	7.21	6.30	7.55
5.39	8.95	7.52	7.78
7.16	10.48	8.94	9.09
10.37	11.47	13.88	9.69
13.41	12.06	14.80	10.16

A, dibutyl maleate; B, itaconic acid. A + B is relative to 100 phr sPS.

Figure 5 DSC thermograms of sPS and g-sPS (dibutyl maleate–itaconic acid = 3 : 1).

TABLE VI
Heat Properties of *g*-sPS with Various
Monomer Additions

Addition (phr)	T_g (°C)	T_m (°C)	Enthaly (J/g)	X_c (%)
0	129.03	271.37	21.23	40.1
3.28	117.32	270.98	20.05	37.8
5.39	116.62	269.75	19.29	36.4
7.16	115.08	269.76	19.21	36.2
10.37	111.77	269.75	19.89	37.5
13.41	108.59	269.95	22.75	42.9
15.24	106.18	269.57	22.84	43.1

The results indicated that the glass-transition temperature (T_g) and T_m decreased with increasing addition of monomer. However, the enthalpy and the corresponding X_c of *g*-sPS first decreased and then increased. When the total addition of monomers was 7.16 phr, the X_c of *g*-sPS was at a minimum and about 36.2%. At this time, the T_g was 115.08°C, and the T_m was 269.76°C.

Because the monomers with flexible groups were induced onto the side chain of sPS, T_g and T_m were first inclined to decrease with increasing addition of monomer, which caused the normalized X_c to be broken and made the enthalpy of *g*-PS decrease. However, there were lots of polar groups such as the ester group R_1COOR_2 and the carboxylic group RCOOH in the graft side chain, which enhanced the interaction force between the macromolecules. Therefore, the enthalpy and X_c of *g*-sPS increased with further increasing addition of monomer.

During the period of graft copolymerization, the total addition of monomers even reached 15.24 phr; the crystal peak was still a single peak, which implied that the microstructure of *g*-sPS was a single phase and there was not emergence in the two phase.

The previous analysis indicates that the orderly normalization structure of sPS was not wrecked heavily, despite the side chain produced by graft copolymerization, and the resulting *g*-sPS still possessed good X_c and heat properties.

To further study the inner structure of the resulting *g*-sPS, we observed SEM photographs of *g*-sPS with various monomer additions; these are shown in Figure 6. Compared with the SEM photograph of pure sPS, the morphology of the *g*-sPS was almost not changed, which indicated that the microstructure of *g*-sPS was a single phase. In other words, the resulting *g*-sPS was not an emergent two-phase structure.

6a sPS

6b 5.39 *phr*

6c 7.16 *phr*

6d 10.37 *phr*
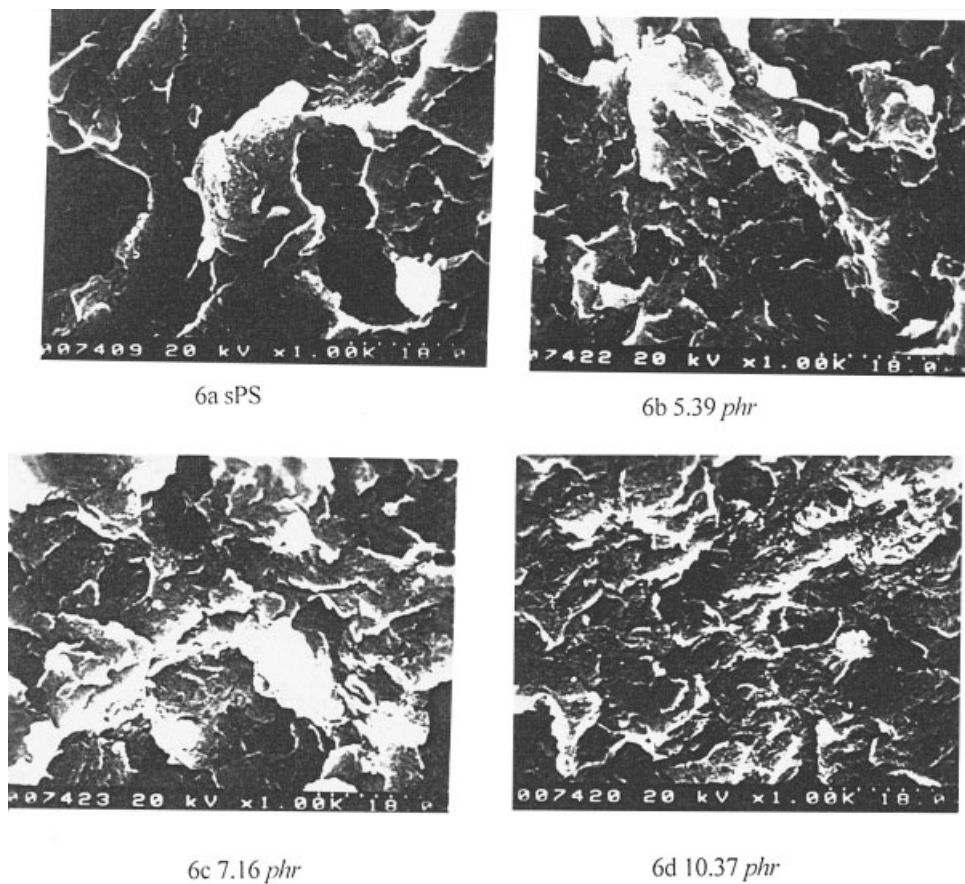
Figure 6   SEM photographs of *g*-sPS with various monomer additions.

CONCLUSIONS

In this study, g-sPS as a compatibilizer for sPS/PA66 blends was synthesized by the melting graft copolymerization of sPS with DCP as an initiator. The structure characteristic and properties of the resulting g-sPS were discussed in detail. The conclusions we obtained were as follows:

1. The characteristic absorbing peaks in the IR spectra did not prove that sPS participated in the graft copolymerization with dibutyl maleate and itaconic acid. The side chain with a polar ester group (R_1COOR_2) and a carboxylic group (RCOOH) was grafted onto the para position of the benzene ring of sPS.

2. The σ_I of the resulting g-sPS first increased and then decreased with the increased addition of monomers, and the maximum value of σ_I emerged at a 7.16-phr addition of monomers for three kinds of monomer proportions at 1 : 3, 1 : 1, and 3 : 1. Therefore, the greatest σ_I obtained was a composition with a 3 : 1 monomer proportion between dibutyl maleate and itaconic acid.

3. For the monomer proportions 1 : 1 and 3 : 1, σ_T increased with increasing addition of monomers. However, σ_T decreased with increasing addition of monomers when the addition of monomers was greater than 13.41 phr for that with a proportion of 1 : 3.

4. X_c of the resulting g-PS first decreased and then increased with increasing addition of monomers, and the g-sPS with the addition of 7.16 phr monomers corresponded to the minimum X_c. The SEM analysis exhibited that the resulting g-sPS had a single-phase structure.

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