Crystallization and Dynamic Mechanical Properties of Polypropylene/Polystyrene Blends Modified with Maleic Anhydride and Styrene

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ABSTRACT: Polypropylene (PP)/polystyrene (PS) blends modified with reactive monomers, such as maleic anhydride (MAH) and styrene (St), and *in situ* formed PP/PS blends were prepared by melting extrusion. The crystallization and melting behavior and the dynamic mechanical properties of the PP/PS blends, including the structure of the grafted copolymer, were investigated with differential scanning calorimetry, dynamic mechanical analysis, and Fourier transform infrared. The results indicated that the addition of MAH hardly influenced the crystallization temperature of PP in the blends, but the addition of MAH and St increased the crystallization temperature of PP in its blends. The blends showed no remarkable variety for the melting temperature, but the shapes of the melting peaks were influenced by the addition of the reactive monomers. In addition, a significant increase in the storage and loss moduli of all the modified PP/PS blends was observed. © 2005 Wiley Periodicals, Inc. J Appl Polym Sci 96: 2038–2045, 2005

Key words: crystallization; mechanical properties; poly(propylene) (PP); polystyrene

INTRODUCTION

Melt-blending different polymers is a more direct and less expensive way of producing multiphase new materials. Polypropylene (PP) and polystyrene (PS) are two of the most widely used plastics in the world. In PP/PS blend systems, one is the crystalline phase, and the other is the amorphous phase. For PP/PS blends, much research has been focused on the crystallization, morphology, and mechanical properties. Xie et al.[1] studied the relationship between the morphology and macroscopic mechanical properties of PP and PS blends. Fortelny et al.[2] investigated the effects of the rate and time of mixing on the phase structure of PP/PS blends with various rheological properties of the components. Pluta et al.[3] examined the phase structure and dynamic mechanical properties of three PP/PS systems of similar compositions but various dispersions of the minor PS component. Blends containing *in situ* polymerized PS show nanoscale phase separation of PS and physical entanglements between PS and the noncrystalline phase of PP. The PS component in blends prepared by melt mixing appears to be completely phase-separated into micrometer-sized domains. When PP is the major component in a blend, its crystallization behavior is not affected by the blending with PS. However, if PP is the minor component, it is dispersed in the immiscible PS matrix, and so the nucleation mechanism changes from being predominantly heterogeneous to being predominantly homogeneous as long as the size of the dispersed PP droplets is below a critical value (ca. 1–2 μm).[4] Wenig et al.[5] determined crystallization kinetic parameters, such as spherulitic growth rates, nucleation densities, and Avrami exponents, for isotactic polypropylene (iPP) blended with atactic polystyrene (aPS). The crystallization of iPP has been found to be strongly influenced by the presence of PS. With increasing PS concentration in a blend, the nucleation densities decrease, whereas the spherulitic growth rates and the positions of the thermal peaks remain independent of the sample composition. Because of the formation of interfaces as a result of the increasing dispersion of PS, the nucleation changes from preferentially thermal to athermal. Bartczak et al.[6] studied the morphology and primary nucleation of spherulites and their growth rate in immiscible blends of iPP and aPS. The spherulitic growth rate in such blends does not depend on either the concentration of aPS in the blends or the time of mixing.

However, PP/PS blends exhibit poor mechanical properties because of the incompatibility of these two

polymers. The compatibility and mechanical properties of incompatible PP/PS blends normally improve with the addition of block copolymer compatibilizers. Horak et al.[7] investigated effects of three different linear SB block copolymers on the morphology and select mechanical properties of HIPS/PP blends. Smit and Radonjic[8] studied the supermolecular structure of iPP/aPS blends compatibilized with SBS. The nucleation, crystal growth, and phase morphology are affected by the addition of SBS. Navratilova and Fortelny[9] studied the compatibilization effect of SBS and the effect of the rheological properties of PP on the morphology of PP/PS blends. Fortelny and Michalkova[10,11] studied the development of the phase structure in PP/PS blends compatibilized with SBS and the effects of the SBS compatibilizer, time of mixing, sequence of the chamber filling, and mixing temperature on the development of the phase structure in PP/PS blends. An admixture of SBS did not lead to a substantial increase in the phase structure uniformity, but it reduced the average size of dispersed particles. The uniformity of the phase structure increased with increasing temperature in the melt during mixing. Radonjic and Smit[12] investigated the compatibilizing effect of SBS on the morphology and mechanical properties of immiscible PP/PS blends. Hlavata et al.[13] studied the structure of PS/PP blends compatibilized by SB and SBS. The triblock influenced the formation of an interfacial layer in the blends in a much more pronounced way than the diblock. Macaubas and Demarquette[14] studied the morphology and response to small-amplitude oscillatory shear of PP/PS blends compatibilized with SBS or SEBS copolymers. The morphological, viscosity, and interfacial tension results showed that SEBS is a better compatibilizer for PP/PS blends than SBS. Santana and Campomanes[15,16] studied the compatibilizing effect of SEBS on the morphology and mechanical properties of virgin and recycled PP/HIPS blends. SEBS reduced the diameter of dispersed HIPS particles and improved the adhesion between the dispersed phase and the matrix. Radonjic[17] investigated the compatibilizing effects of SBS, SEP, and two types of SEBS on the morphology and mechanical properties of PP/PS blends. The compatibilizing efficiency of the block copolymer was strongly dependent on the chemical structure of the rubber block, the molecular weight of the block copolymer molecule, and its concentration. Raghu et al.[18] studied the mechanical, thermal, rheological, and morphological properties of PP/PS blends compatibilized with SIS, SBS, and SBR. The SIS- and SBS-compatibilized blends showed significantly improved impact strength and ductility in comparison with the SBR-compatibilized blends. You and Jia[19] investigated the effects of an SEP diblock copolymer on the morphology and mechanical properties of PP/PS blends. Halimatudahliana and Nasir[20] studied the mechanical

properties and morphologies of PS/PP blends compatibilized by SEBS, Surlyn, EVA, and sodium salt hydrate of 4-styrenesulfonic acid (4ssa,ssh). The incorporation of SEBS into the PS/PP blends resulted in a finer degree of dispersion of particles. EVA showed higher plastic deformation, whereas Surlyn showed finer phase domains. However, with the presence of 4ssa,ssh in the PS/PP blend, the interface did not show clear changes in comparison with the uncompatibilized blend. The blends containing SEBS and EVA showed a positive effect on the ductility of the blends. In the presence of Surlyn, the strength of the blends increased, whereas 4ssa,ssh showed a negative effect. Hlavata and coworkers[21–23] studied the compatibilizing effect of a series of SB block copolymers in PS/PP blends. The SBS and SBSBS copolymers were better compatibilizers than the SB, SBSBSBS, and BSBSBSB copolymers. The ability of a block copolymer to participate in the formation of an interfacial layer (and, therefore, in the compatibilization process) has been shown to depend primarily on the interactions of the block copolymer with the blend components and to be closely associated with the molar mass of the styrene (St) blocks. The compatibilizing effect of isotactic polystyrene (iPS)-b-iPP was examined by Xu and Lin[24] in iPS–iPP blends. Chen et al.[25] evaluated the effects of a polymeric alloy (SP-A) containing syndiotactic polystyrene (sPS), atactic polypropylene (aPP), and approximately 66 wt % sPS-b-aPP diblock copolymer as a compatibilizer for sPS and iPP blends.

The compatibility and mechanical properties of incompatible PP/PS blends also improve with the addition of graft copolymer compatibilizers. Kim et al.[26] investigated the compatibilizing effect of polyethylene (PE)-g-polystyrene on the morphology and thermal properties of PP/PS blends. Adewole et al.[27] determined the efficiency of PP-g-PS with respect to SEBS as a compatibilizer for PP/PS blends. Both PP-g-PS and SEBS enhanced the compatibilization of the blends, but the graft copolymer was more effective. The presence of another component can have a significant influence on the melting and crystallization temperatures, crystallite growth rate, and degree of crystallization. D'Orazio et al.[28,29] studied the influence of the crystallization conditions on the morphology and thermal behavior of PP/PS blends compatibilized with a graft copolymer of unsaturated propylene and styrene (uPP-g-PS). The addition of the uPP-g-PS copolymer induced a drastic change in the iPP spherulitic texture and the inner structure of the spherulitic fibrils. A linear reduction of the equilibrium melting temperature and the folding surface free energy values with increasing uPP-g-PS content was observed. The crystalline lamellar thickness and interlamellar amorphous layer thickness were higher than those observed in plain iPP. Danella and Manrich[30] studied the efficiency of compatibilization of three different

copolymers, PP-g-MAH, PE/PS, and PP-g-PS, on PP/PS blends. Pionteck et al.[31] studied the efficiency of different mechanisms for the reactive compatibilization of PP/PS blends compatibilized by terminal functionalized PP and terminal functionalized PS.

The compatibility and mechanical properties of incompatible PP/PS blends can also be improved with irradiation, reactive compatibilization, and inorganic particles. Albano and coworkers[32,33] studied the effect of γ irradiation on the mechanical, morphological, and thermal behavior of PS/PP blends with and without a compatibilizer (block SBS). Xie and Zheng[34] studied the one-step reactive compatibilization of PP/PS alloys with a twin-screw extruder in the presence of dicumyl peroxide (DCP). Diaz et al.[35] assessed the application of the Friedel–Crafts alkylation reaction to the compatibilization of PP/PS blends. Zhang et al.[36] reported changes in the phase morphology and properties of immiscible PP/PS blends compatibilized with SiO_2 nanoparticles. Inorganic clay was investigated as a compatibilizer for immiscible PP/PS blends.[37] The addition of barium sulfate ($BaSO_4$) to the PP/PS blend resulted in a decrease in the domain size of the minor polymer phase.[38,39] $BaSO_4$ alone did not have a nucleation effect on PP; however, in combination with PP-g-MAH, a clear nucleation effect was observed.

In this article, we present the results of a study on the influence of reactive monomers, maleic anhydride (MAH) and St, on the crystallization and melting behaviors and dynamic mechanical properties of PP/PS blends.

EXPERIMENTAL

Materials

The polymers used in this study were CTS-700 PP (Yinzhu Polypropylene Limited Corp., Guangzhou, China) and PG-33 PS (Qimei Chemical Industry, Taiwan, China). St (Xingang Chemical Limited Co., Guangzhou, China) was analytical-reagent-grade. MAH (Chemical Reagent Institute, Tianjin, China) and DCP (Chemical Reagent Central Factory, Shanghai, China) were chemically pure.

Preparation of the blends

An acetone solution containing the reactive monomers and 0.1 wt % DCP (with respect to the blends) was prepared, and then it was added to PP/PS blends or pure PP. The mixtures were melt-blended in an HL-200 kneader at 185–190°C and 50 rpm for 15 min.

Dynamic mechanical behavior characterization

The dynamic mechanical behavior of the blends was determined in the single-cantilever oscillating mode with a strain amplitude of 20 μm at a frequency of 10 Hz. A DMA2980 dynamic mechanical thermal analyzer (TA instrument, Inc., United States) was used. The testing temperature range was −90 to 100°C in an atmosphere of N_2. The sample size was 35 mm × 3.65 mm × 9.80 mm wide. Before the measurements were begun, the samples were precooled at −90°C for 3 min. The heating rate was 5°C/min, and the measurement interval was 5 s.

Crystallization and melting behavior characterization

The crystallization and melting behavior of the samples was determined with differential scanning calorimetry (DSC) with a PerkinElmer DSC-7 in a nitrogen atmosphere. The samples were preheated quickly from room temperature to 220°C, held at that temperature for 3 min to eliminate the thermal history, cooled from 220 to 50°C at a rate of 10°C/min, and finally reheated again to 220°C at the same rate. The crystallization and melting parameters were recorded from the cooling and reheating scans. The degree of crystallization was calculated with the general standard of $\Delta H_f = 207.15$ J/g [40]

Grafted copolymer characterization

Each sample was compressed into thin film by melting and then was extracted with ethyl acetate at 80°C for 4 h to extract the free PS; it was finally dried in a vacuum oven at 70°C for 12 h. The existence of the grafted copolymer was characterized by Fourier transform infrared (FTIR) spectroscopy (Equinox 55, Bruker, Germany).

RESULTS AND DISCUSSION

Characterization of the grafted copolymer

The characterization of the grafted copolymer was investigated with FTIR spectroscopy. Typical IR spectra of the PP/PS blends modified by MAH and St/MAH are shown in Figure 1. As shown in Figure 1(a), no absorption peak at 1780 cm^{-1} was observed for the PP/PS blends modified by MAH (the C=O stretching vibration). This result indicated no existence of the maleic anhydride grafted polypropylene copolymer (MA-g-PP) in the blends. It suggested that MAH mainly formed the maleic anhydride grafted polystyrene copolymer (MA-g-PS) with PS. Then, *in situ* formed MA-g-PS was extracted by ethyl acetate. However, as shown in Figure 1(b), there was a significant absorption peak at 1780 cm^{-1} for the PP/PS blends modified with the St/MAH comonomer, and this confirmed the existence of MA-g-PP. Figure 1(b) shows that the height of the peak at 1780 cm^{-1} increased with
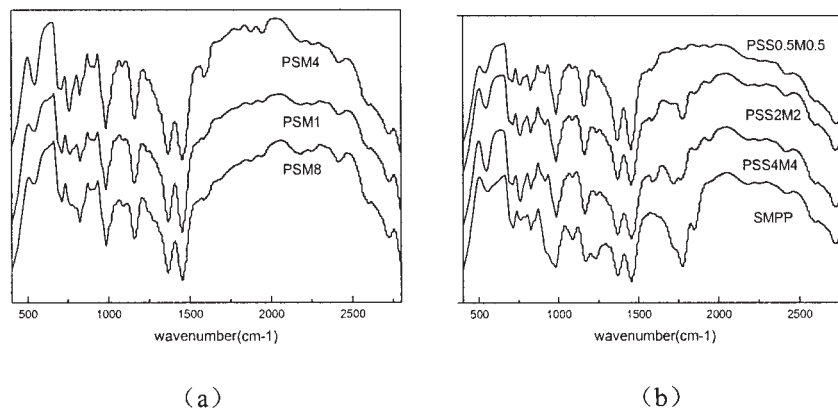
Figure 1 FTIR spectra of PP/PS blends modified by (a) MAH and (b) St/MAH after extraction by ethyl acetate at 80°C for 4 h.

an increasing content of the comonomer. For the *in situ* formed PP/PS blend (PP modified by the comonomer, i.e., SMPP), the peak at 1780 cm^{-1} was more significant, and the height of the peak was higher, than those of the blends modified by the comonomer.

Crystallization and melting behavior of the PP/PS blends modified by MAH

DSC crystallization curves of the PP and PP/PS blends modified by different MAH contents are shown in Figure 2; the crystallization and melting data are presented in Table I. For pure PP, the peak temperature of crystallization (T_c^p) and the temperature at the onset of crystallization (T_c^{on}) were 110.1 and 114.6°C, respectively. The addition of PS (20 wt %) to PP did not influence remarkably T_c^p and T_c^{on} of PP. This result indicated that no heterogeneous nucleation of PS existed for PP crystallization. T_c^p of PP in the PP/PS blends modified by MAH did not vary significantly either. With an increasing concentration of MAH, T_c^p and the peak temperature of melting (T_m^p) of PP remained invariable. Perhaps there was competition of MAH grafting with PP or PS when MAH was added to the PP/PS blends. The

FTIR results confirmed that MAH was inclined to graft with PS. As a result, there was no MA-g-PP in the blends, or its concentration was low because of the low grafting rate between MAH and PP.[41] The main grafting product was MA-g-PS, which hardly changed the crystallization and melting behavior of PP.

As shown in Figure 2(b) and Table I, pure PP showed a shoulder peak at a high temperature in the DSC melting curve. Although T_m^p and the temperature at the onset of melting (T_m^{on}) of PS20 were lower than those of pure PP, the intensity of the shoulder peak at a high temperature of PS20 was higher than that of pure PP. The addition of MAH to the PP/PS blends had little effect on T_m^p and T_m^{on} of PP, but the intensity of the shoulder peak at a high temperature of the blends decreased gradually with an increasing concentration of MAH. This indicated that PP in the blends modified by MAH formed different perfect degrees of crystallization during cooling.[42]

Crystallization and melting behavior of the PP/PS blends modified by St and MAH

DSC crystallization curves of the PP/PS blends modified with different concentrations of St and MAH are
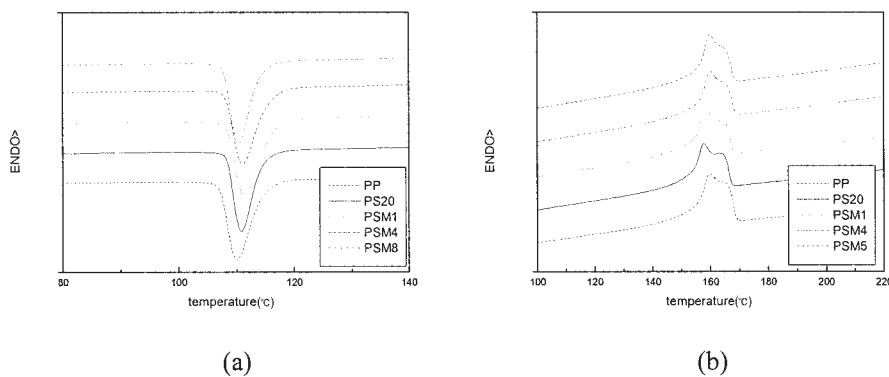

Figure 2 DSC (a) crystallization and (b) melting curves of PP/PS blends modified by MAH.

<div style="text-align:center">

TABLE I
DSC Results of PP/PS Blends Modified by MAH

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Sample	PP (wt %)	PS (wt %)	MAH (phr)	T_c^p (°C)	T_c^{on} (°C)	X_c (%)	T_m^p (°C)		T_m^{on} (°C)	X_m (%)
PP	100	0	0	110.1	114.6	44.6	159.6	164.8	154.2	45.3
PS20	80	20	0	110.8	114.5	43.9	157.7	163.9	153.0	45.7
PSM1	80	20	1	111.4	116.4	44.7	158.4	163.8	154.0	45.4
PSM4	80	20	4	111.1	115.6	43.4	160.0	163.9	155.4	45.8
PSM8	80	20	8	110.4	114.1	43.5	159.6	163.9	155.3	44.5

phr: relative to 100 blends. X_c, degree of crystallization from the DSC crystallization; X_m, degree of crystallization from the melting peak.

shown in Figure 3; the crystallization and melting data are presented in Table II. As shown in Figure 3 and Table II, the addition of the comonomer to the PP/PS blends had a significant effect on the crystallization and melting behavior of PP. Some studies have confirmed that comonomers St and MAH react readily to form a styrene/maleic anhydride copolymer (SMA) with the help of an initiator agent. It is much easier for SMA than MAH to graft with PP; therefore, the degree of grafting of MAH increases, and more MA-g-PP is formed *in situ*.[41,43,44] The addition of St and MAH had a significant effect on the crystallization behavior of PP in the blends because of the formation of MA-g-PP and the heterogeneous nucleation of MA-g-PP on PP.[45,46] When the comonomer concentration in the PP/PS blends was lower, the variability of the crystallization behavior of PP was low. It could be suggested that the *in situ* formation of MA-g-PP was so low that its effect on the heterogeneous nucleation of PP was lower. When the concentration of the comonomer was high (4 phr), high T_c^p and T_c^{on} values of PP in the blend were observed. It could be suggested that with a high concentration of the comonomer, PP reacted with the comonomer and formed much more MA-g-PP, which induced more heterogeneous nucleation and increased the crystallization temperature of PP. However, when the concentration of the comonomer was excessively high (8 phr), T_c^p of PP decreased. This could be attributed to excessive PP grafted with

MAH, which demolished the regularity of the PP chain and resulted in lower T_c^p. As for pure PP modified by St and MAH (SMPP), it exhibited a higher value of T_c^p with respect to that of PS20 because of the heterogeneous nucleation of *in situ* formed MA-g-PP.

As shown in Figure 3(b) and Table II, the blend with a high T_c^p value had a high T_m^p value. However, the modified PP/PS blend exhibited different peak shapes of melting. In comparison with PS20, the intensity of the shoulder peak at a high temperature of PSS0.5M0.5 became more indiscernible. With increasing concentrations of St and MAH, a single peak was observed, and its peak temperature was between the two peak temperatures of PS20. The other difference in the shape of the melting peak caused by the effect of the comonomer on the melting behavior was the presence of a small melting peak at approximately 150°C for the modified PP/PS blends, but it remains to be confirmed by experimentation whether the small melting peak is related to the formation of β crystals. Multiple melting peaks were observed for SMPP, and the reason must be investigated in the future.

Dynamic mechanical properties of the modified PP/PS blends

The curves of the storage modulus (E'), loss modulus (E''), and tan δ of PP/PS blends modified with MAH with respect to the temperature are shown in Figure 4.
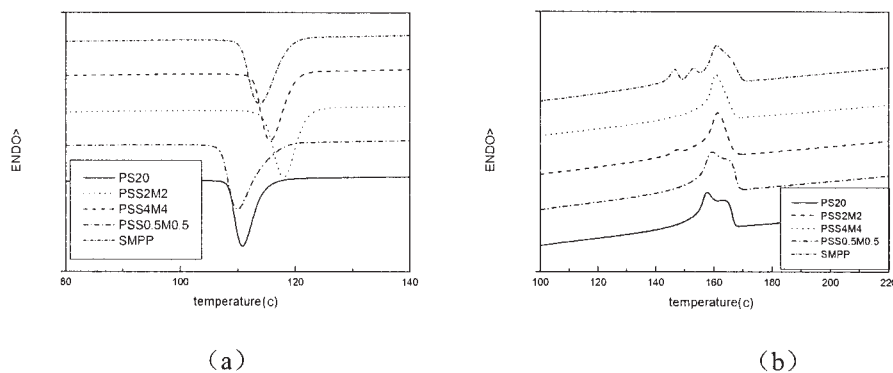

(a)

(b)

Figure 3 DSC (a) crystallization and (b) melting curves of PP and PP/PS blends modified by St and MAH.

TABLE II
DSC Results of PP and PP/PS Blends Modified by St and MAH

Sample	PP (wt %)	PS (wt %)	St (phr)	MAH (phr)	T_c^p (°C)	T_c^{on} (°C)	X_c (%)	T_m^p (°C)		T_m^{on} (°C)	X_m (%)
PS20	80	20			110.8	114.5	43.9	157.7	163.9	153.0	45.7
PSS0.5M0.5	80	20	0.5	0.5	110.0	115.3	53.3	159.5	164.6	153.8	54.5
PSS2M2	80	20	2	2	118.0	121.9	43.2	161.4		156.4	46.8
PSS4M4	80	20	4	4	115.6	119.8	42.7	160.8		156.4	44.6
SMPP	100		35.7	7.14	113.8	118.8	54.4	161.0		156.2	59.6

X_c, degree of crystallization from the DSC crystallization; X_m, degree of crystallization from the melting peak.

As shown in Figure 4, the addition of MAH to the 80/20 PP/PS blend led to a significant increase in E' and E'' over the whole temperature range, especially for the system containing 4 phr MAH. This result indicated that the incorporation of MAH increased the modulus of the PP/PS blends. However, as the concentration of MAH further increased, E' and E'' of the modified blends decreased. For the MAH-modified PP/PS blends, the temperatures of the E'' and tan δ peaks of PP shifted to a low-temperature region with an increasing concentration of MAH. This indicated that the modified blends had lower glass-transition temperatures (T_g's). The PP segmental chains of the modified blends could move more easily. The addition of 1 phr MAH had only a small effect on the intensity of the tan δ values of the blends. For 4 and 8 phr MAH, the tan δ values were considerably higher than those of the unmodified blends at a temperature below T_g of PP, but at a temperature above T_g of PP, the effect was not much pronounced. This change in the tan δ values with the temperature indicated that the interfacial interaction could be weakened at a higher temperature.

The curves of E', E'', and tan δ of the PP/PS blends modified with comonomers MAH and St with respect to the temperature are shown in Figure 5. The addition of the comonomer to 80/20 PP/PS blends led to a significant increase in E' and E'' over the whole temperature range too. For *in situ* formed PP/PS blends (SMPP), the tan δ values were remarkably higher than those of the unmodified blends at a temperature below T_g of PP. This was attributed to the fact that the relatively small molecular weight of *in situ* formed PS improved the activity of the segmental chains of the blends. For the blends modified with the comonomers
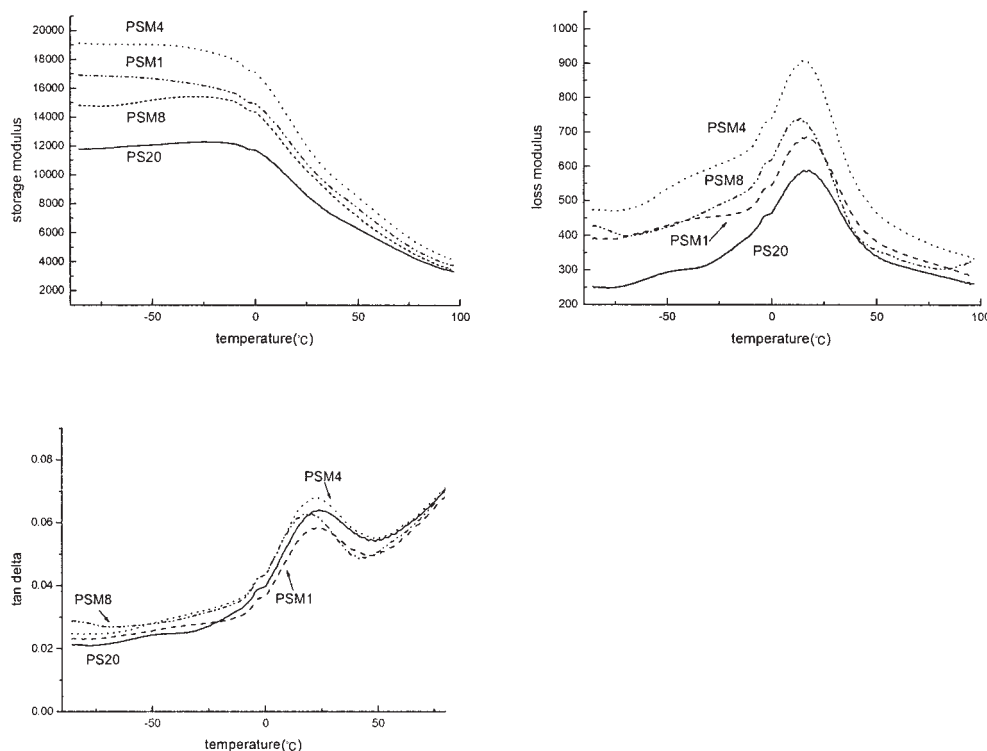

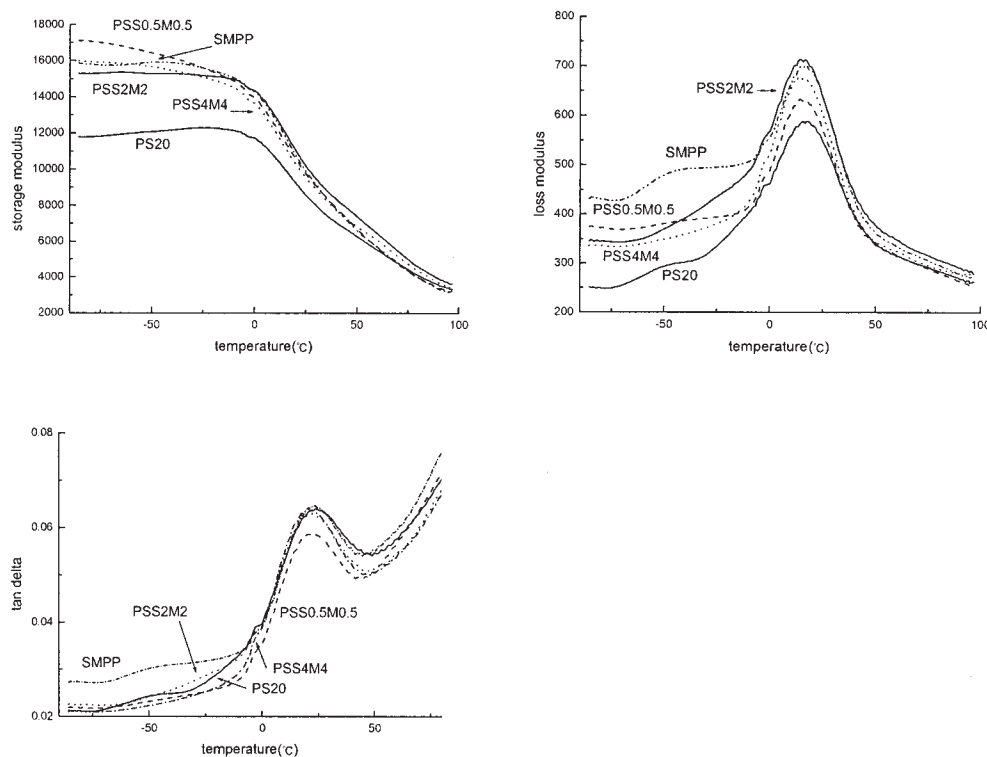**Figure 4** Dependence of E', E'', and tan δ on the temperature of 80/20 PP/PS blends modified by MAH.

Figure 5 Dependence of E', E'', and tan δ on the temperature of 80/20 PP/PS blends modified by MAH and St.

MAH and St, the tan δ peak of PP was slightly shifted to a lower temperature region, but the variability of the tan δ values showed different trends corresponding to different concentrations. The tan δ values of PSS2M2 were slightly higher than those of PS20 at a temperature below T_g of PP. However, those of PSS0.5M0.5 and PSS4M4 were lower than those of PS20.

CONCLUSIONS

The FTIR results indicated that MA-*g*-PS was formed in PP/PS blends modified by MAH. However, the formation of MA-*g*-PP was observed in PP/PS blends modified by MAH and St comonomers. In PP modified by the comonomer, the formation of MA-*g*-PP was more significant.

The addition of PS to PP hardly changed the crystallization and melting behavior of PP, just like the addition of MAH to PP/PS blends. However, MAH could significantly increase E' and E'' of the blends. Moreover, the blends modified with MAH shifted T_g of PP to a lower temperature region. It was suggested by IR results that MA-*g*-PS was the main grafting product in the blends.

The addition of the comonomers MAH and St increased T_c^p of PP and made PP form a single peak of melting instead of a shoulder peak of melting. This could be attributed to the heterogeneous nucleation of

MA-*g*-PP, which was *in situ* formed in melt mixing. The comonomer could increase the intensities of E' and E'' of the blends too, and this was accompanied by a small decrease in T_g.

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