

Effect of High-Performance Polymers on Crystallization and Multiple Melting Behavior of Poly(phenylene sulfide)

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ABSTRACT: The crystallization and multiple melting behavior of poly(phenylene sulfide) (PPS) and its blends with amorphous thermoplastic bisphenol A polysulfone (PSF) and phenolphthalein poly(ether ketone) (PEK-C), crystalline thermoplastic poly(ether ether ketone) (PEEK), and thermosetting bismaleimide (BMI) resin were investigated by a differential scanning calorimeter (DSC). The addition of PSF and PEK-C was found to have no influence on the crystallization temperature (T_c) and heat of crystallization (ΔH_c) of PPS. A significant increase in the value of T_c and the intensity of the T_c peak of PPS was observed and the crystallization of PPS can be accelerated in the presence of the PEEK component. An increase in the T_c of PPS can also be accelerated in the BMI/PPS blend, but was no more significant than that in the PEEK/PPS blend. The T_c of PPS in the PEEK/PPS blends is dependent on the maximum temperature of the heating scans and can be divided into three temperature regions. The addition of a second component has no influence on the formation of a multiple melting peak. The double melting peaks can also be observed when PPS and its blends are crystallized dynamically from the molten state. © 1998 John Wiley & Sons, Inc. *J Appl Polym Sci* 69: 637–644, 1998

Key words: poly(phenylene sulfide); poly(ether ether ketone); crystallization and melting behavior; polymer blends

INTRODUCTION

Poly(phenylene sulfide) (PPS) possesses high-temperature resistance combined with good mechanical properties and exceptional chemical and solvent resistance. However, crystalline PPS is rather brittle. Blending semicrystalline PPS with other polymers is an effective way of improving properties such as impact strength and toughness. Its applications potential can also be extended by blending it with other thermoplastics for cost dilution and improved toughness. A few

scientific articles dealing with blends of PPS with other polymeric resins have been published during the last 10 years.^{1–15} The mechanical and physical properties of these materials are dependent on the phase morphology, the crystalline behavior, and the miscibility of the component polymer. The addition of different polymers (amorphous and crystalline thermoplastic and thermosetting resin) has a different effect on the crystallization behavior and physical as well as mechanical properties of PPS.

The aim of this work was to study the crystallization behavior and multiple melting phenomena of PPS blends with amorphous thermoplastics with high glass transition temperatures (T_g): bisphenol A polysulfone (PSF) ($T_g = 190^\circ\text{C}$), phenolphthalein poly(ether ketone) (PEK-C) ($T_g = 225^\circ\text{C}$), and crystalline thermoplastic poly(ether ether ke-

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tone) (PEEK) ($T_m = 334^\circ\text{C}$), and thermosetting bismaleimide (BMI) resin. The effect of high-performance amorphous and crystalline thermoplastics as well as thermosetting resin on the crystallization and multimelting behavior of PPS are characterized and discussed.

EXPERIMENTAL

PPS was Ryton grade V-1 supplied by Phillips Petroleum Co. (Bartlesville, OK). PSF with a reduced viscosity of 0.48 was obtained from Shuang Chemical Plant (Shanghai, China). PEK-C with a reduced viscosity of 1.1 was received from Xuzhou Engineering Plastic (Xuzhou, China), and PEEK was obtained from Jinlin University (Changchun, China). BMI was received from the Fongguang Chemical Plant (Hubei, China). The particle size for the starting materials were 5–10 μm for PPS, 10–20 μm for BMI, and 20–40 μm for PEEK, PEK-C, and PSF.

The sample preparation for these studies was as follows: Weighted amounts of powders of different polymer blends, PEEK/PPS, PEK-C/PPS, PSF/PPS, and BMI/PPS, were dry mixed. This charge was fed to a metal die and cold-compressed at 500 kg/cm² using a 10-ton hydraulic press. Cylindrical discs (1 mm-thick, 12-cm² area) were produced. PPS and its blends were melted in a differential scanning calorimeter (DSC) cell at 320 or 350°C for 10 min and quenched in ice water. The quenched samples were annealed in the DSC. The crystallization and melting behavior of quenched and annealed samples was determined using a Perkin–Elmer DSC-7 between 50 and 320°C at a heating or cooling rate of 20°C/min in

a nitrogen atmosphere. Test samples were approximately 9–11 mg. The transition temperature and the heat of crystallization and fusion were calibrated using an indium standard.

RESULTS AND DISCUSSION

Crystallization and Melting Behavior of PPS in Quenched Blends

Table I shows the DSC results of PPS and its blends melted for 10 min at 320 and 350°C, respectively, and quenched in ice water. The addition of thermoplastic PSF, PEK-C, PEEK, and the thermosetting resin BMI has no significant effect on the glass transition temperature (T_g), temperature (T_{cc}) and heat (ΔH_{cc}) of cold crystallization, melting point (T_m), and heat of fusion (ΔH_m) of PPS. A small change in the T_{cc} and T_m of PPS and its blends was attributed to the retardation of the cold-crystallization of solid PPS in the presence of a second component, which leads to the small decrease in the T_m of PPS. It must be pointed out that the effect of various polymers on the crystallization behavior of PPS is different in the DSC cooling scanning process (Fig. 1) from the molten state after the samples were heated to 320°C. It can be seen from Table I and Figure 1 that amorphous thermoplastic PSF and PEK-C with a high T_g have no influence on the crystallization temperature (T_c) and heat of crystallization (ΔH_c) of PPS. A significant increase in the value of the T_c of PPS was observed in the presence of the PEEK component. The shape of the T_c peak of PPS in the PEEK/PPS = 20/80 blend became narrower and the intensity of the T_c peak was increased.

Table I Crystallization and Melting Parameters of PPS in Quenched Blends

	T_{melt} (°C)	T_g (°C)	T_{cc}^0 (°C)	T_{cc} (°C)	ΔH_{cc} (J/g)	T_m^0 (°C)	T_m (°C)	ΔH_m (J/g)	T_c^0 (°C)	T_c (°C)	ΔH_c (J/g)	ΔT (°C)
PPS	320	86.2	118.2	123.5	29.78	269.3	284.0	43.19	226.0	214.4	46.91	58.0
PSF/PPS = 20/80		84.1	119.2	125.6	27.70	267.8	282.9	38.74	227.9	214.5	43.63	55.0
PEK-C/PPS = 20/80		85.2	121.8	127.3	28.70	268.1	284.1	43.94	226.8	213.5	44.90	57.3
PEEK/PPS = 20/80		86.9	113.3	121.6	20.29	267.0	282.2	43.49	244.0	238.6	45.93	38.2
BMI/PPS = 20/80		86.3	121.9	127.9	26.45	264.6	283.1	42.31	232.1	221.5	39.30	51.0
PPS	350	82.7	119.3	124.6	32.03	269.5	285.0	41.92	229.9	209.8	47.10	55.1
PSF/PPS = 20/80		86.2	120.1	126.4	31.25	267.5	283.8	40.44	230.0	206.3	44.73	60.8
PEK-C/PPS = 20/80		79.2	117.3	125.7	29.60	267.2	283.0	42.68	232.2	211.6	43.88	50.8
PEEK/PPS = 20/80		84.1	119.5	126.0	25.80	266.0	283.3	39.61	246.8	242.3	51.23	36.5
BMI/PPS = 20/80		89.4	118.6	124.7	26.44	265.2	283.1	42.93	236.1	222.6	40.50	47.8

Melting time: 10 min; quenched in ice water.

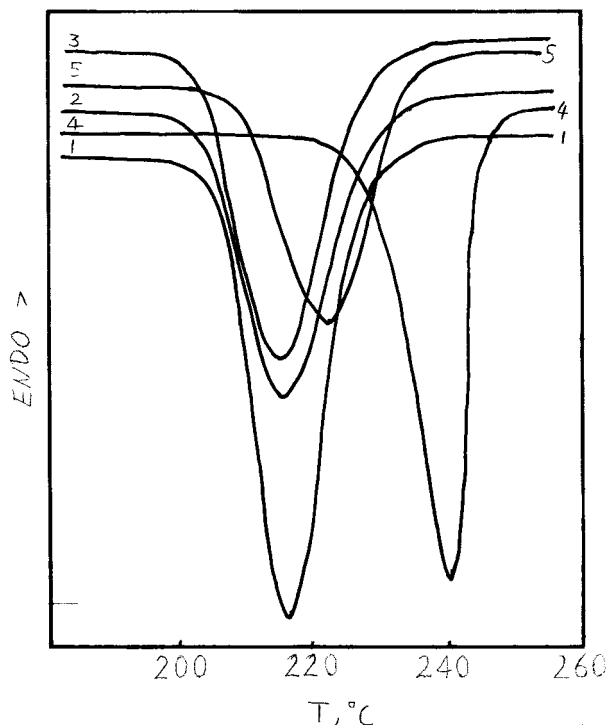


Figure 1 DSC cooling scans of PPS and its blends: (a) PPS; (b) PSF/PPS = 20/80; (c) PEK-C/PPS = 20/80; (d) PEEK/PPS = 20/80; (e) BMI/PPS = 20/80.

The T_c of PPS in the PEEK/PPS = 20/80 blend is much higher than that of neat PPS at the same experimental conditions. The T_c of PPS in the

PEEK/PPS = 20/80 blend was increased from 214.4°C for neat PPS to 238.6°C. The degree of supercooling ($\Delta T = T_m - T_c^0$) was also decreased from 58 to 38.2°C. These results indicate that the crystallization of PPS can be accelerated in the presence of the crystalline PEEK component.

Table I shows that when the melting temperature was increased to 350°C in the preparation of the quenched samples, the T_c of the neat PPS decreased compared to that in the melting temperature at 320°C. However, an increase in T_c and a decrease in the degree of supercooling of PPS in the PEEK/PPS = 20/80 blend are more significant. The T_c of PPS in the PEEK/PPS blend was increased from 209.8 for neat PPS to 242.3°C. The degree of supercooling was decreased from 55.1 to 36.5°C. In the BMI/PPS blend, the cure reaction of BMI took place and a dispersed rigid particle of thermosetting BMI resin in the PPS matrix can be formed in the preparation of quenched BMI/PPS blend samples. When the quenched sample was prepared at the temperatures of 320 and 350°C, an increase in the T_c of PPS can be observed in the BMI/PPS = 20/80 blend. However, the increase in the T_c of PPS in the BMI/PPS blend is no more significant than that in the PEEK/PPS blend. The increase in the T_c of PPS in the PEEK/PPS and BMI/PPS blends was attributed to the acceleration of nucleation in the presence of the parti-

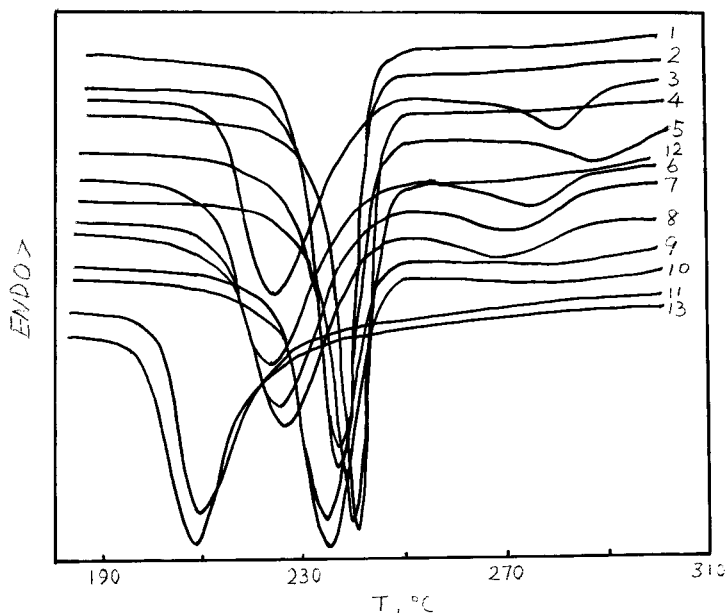


Figure 2 Effect of maximum temperature of heating scans (T_{max}) and number of scans on the DSC cooling scans of PEEK/PPS = 20/80 blend. Number of cooling scans, T_{max} (°C): one, 320; two, 320; three, 360; four, 320; five, 340; six, 360; seven, 350; eight, 345; nine, 330; ten, 335; eleven, 380; twelve, 320; thirteen, 400.

Table II Temperatures and Heat of Crystallization of PPS in PEEK/PPS = 20/80 Blend in Cyclic Heating–Cooling Experiment

No. Cooling Scans	T_{\max} (°C)	T_c^0 (°C)	T_c (°C)	ΔH_c (J/g)
1	320	243.3	237.3	48.49
2	320	243.0	237.4	46.80
3	360	240.3	224.5	45.80
4	320	245.4	240.8	45.81
5	340	244.5	237.4	46.17
6	360	239.8	223.8	43.76
7	350	239.8	225.7	44.93
8	345	240.6	226.9	44.19
9	330	244.2	235.4	44.56
10	335	244.4	235.8	44.31
11	380	224.4	210.6	43.94
12	320	245.8	240.5	43.66
13	400	220.1	208.0	44.94

Melting time: 3 min.

cles of crystalline PEEK and cured BMI resin. Crystalline PEEK and cured BMI resin can act as a nucleating agent of PPS, so that the onset

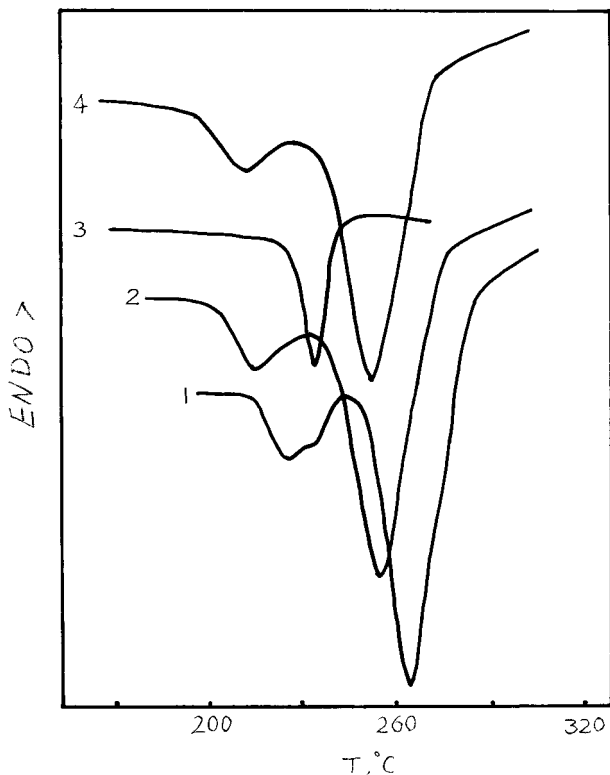


Figure 3 Effect of maximum temperature of heating scans (T_{\max}) and number of scans on the DSC cooling scans of PEEK/PPS = 80/20 blend. Number of cooling scans, T_{\max} (°C): one, 360; two, 400; three, 320; four, 360.

of the crystallization temperature (T_c^0) and T_c was shifted to high temperature.

Acceleration of Crystallization of PPS in PEEK/PPS Blends

The above results indicated that the addition of PEEK can accelerate the crystallization of PPS and shift the T_c of PPS to high temperature. The degree of this action depends upon the maximum temperature of the heating samples in the DSC scans. The multiple-scanning DSC results of the PEEK/PPS = 20/80 blend is shown in Figure 2 and Table II. It can be seen from Figure 2 and Table II that the T_c of PPS in the PEEK/PPS blend is dependent on the maximum temperature of the heating scans (T_{\max}). The T_c of PPS can be divided into three temperature regions based on the effect of the maximum temperature of the heating scans on the T_c of PPS in the PEEK/PPS blend: (1) When T_{\max} is below 340°C (lower than the normal melting point, T_m , of PEEK), the increase in the T_c of PPS is more significant in the presence of the PEEK component. The tempera-

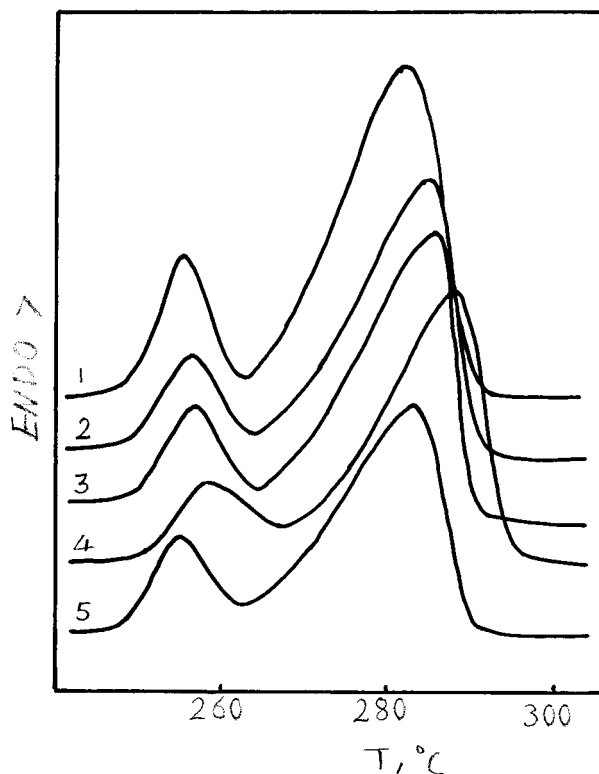


Figure 4 DSC heating scans of PPS and its blends annealed at 240°C for 1 h: (1) PPS; (2) PSF/PPS = 20/80; (3) PEK-C/PPS = 20/80; (4) PEEK/PPS = 20/80; (5) BMI/PPS = 20/80.

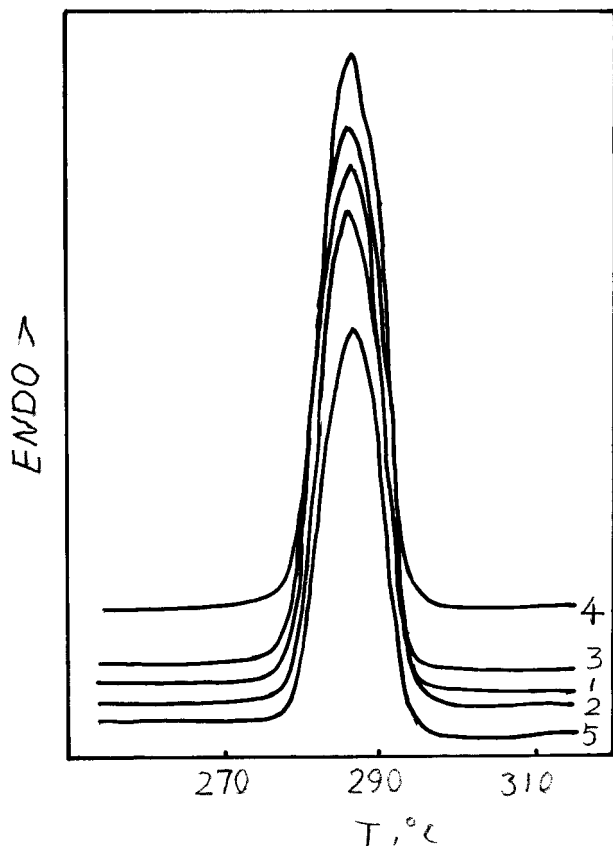


Figure 5 DSC heating scans of PPS and its blends annealed at 270°C for 1 h: (1) PPS; (2) PSF/PPS = 20/80; (3) PEK-C/PPS = 20/80; (4) PEEK/PPS = 20/80; (5) BMI/PPS = 20/80.

ture of the T_c of PPS in its blends is above 235°C and a small increase is seen as the number of heating scans increases. (2) When T_{\max} is between 345 and 360°C (higher than the T_m of PEEK and lower than the equilibrium melting point of PEEK, $T_m^0 = 380^\circ\text{C}$; ref. 16), the T_c of PPS in its blends is about 225°C. (3) When T_{\max} is equal to or higher than the T_m^0 of PEEK, the T_c of PPS is below 210°C and an increase in the T_c of PPS by the addition of PEEK was not observed. It is suggested that the crystallization behavior of PPS in the presence of the PEEK component depends upon the condensed state of the PEEK component.

When the blend was heated to a temperature below the T_m of PEEK, the crystalline PEEK component in the blend was not melted and was annealed to increase the crystalline perfection. The crystalline annealed PEEK can act as a nucleating agent and accelerate the crystallization of the PPS component. Although the number of scans is increased to 12, and the branching and crosslinking of PPS have taken place, the acceleration of

the crystallization of PPS in the presence of the PEEK component was not suppressed. The T_c in the 12th scan is higher than that of the first scan. When the blend was heated to a temperature between the T_m and T_m^0 of PEEK, the crystalline PEEK component was partly or completely melted. The newly formed crystalline PEEK in the cooling process and a small amount of unmelted crystalline PEEK with a higher T_m can act as nucleating agents of PPS and also accelerate the crystallization of PPS. However, the action of the newly formed crystalline PEEK is lower than that of the annealed crystalline PEEK. When the blend was heated to a temperature above the T_m^0 of PEEK, the thermocrosslinking of the PEEK component also took place and the T_c peak of PEEK was also shifted to the lower temperature as the number of scans increased. Only a low perfection of the crystallization of PEEK was achieved and the action of acceleration of the crystallization of PPS was suppressed.

The effect of the maximum temperature of the heating scans on the crystallization behavior of PPS in the PEEK/PPS = 80/20 blend was also observed (Fig. 3). In this blend, the PPS component was dispersed in the PEEK matrix. The higher T_c peak was attributed to the crystallization of PEEK. It can be seen from Figure 3 that

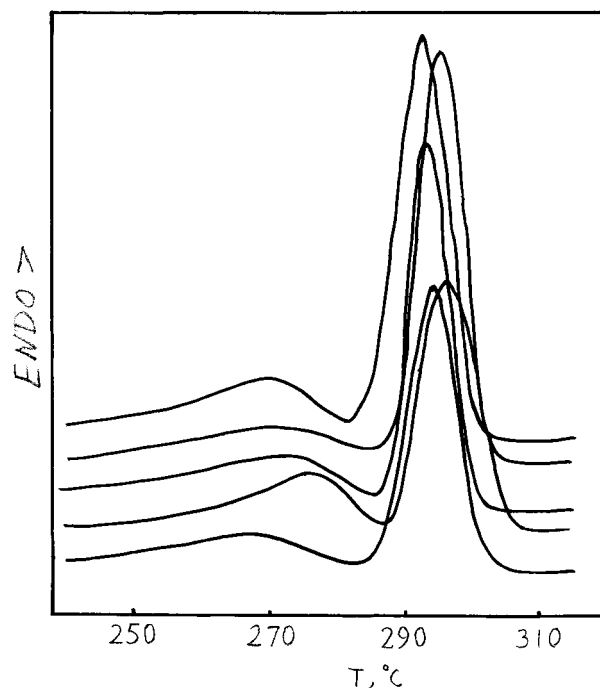


Figure 6 DSC heating scans of PPS and its blends annealed at 280°C for 4 h: (1) PPS; (2) PSF/PPS = 20/80; (3) PEK-C/PPS = 20/80; (4) PEEK/PPS = 20/80; (5) BMI/PPS = 20/80.

although the number of scans is increased the T_c of the PPS in the third scans ($T_{\max} = 320^\circ\text{C}$) is higher than that in the first ($T_{\max} = 360^\circ\text{C}$) and second scans ($T_{\max} = 400^\circ\text{C}$). Also, the shape of the T_c peak became narrower and the intensity of the T_c peak increased.

Effect of Second Component on Multiple Melting Behavior of PPS

There have been many extensive studies on the multiple melting behavior of PPS,^{17–22} PEEK,^{23–29} poly(ethylene terephthalate) (PET),^{30,31} poly(butylene terephthalate) (PBT),^{32,33} and other semicrystalline polymers. Generally, these high-melting semicrystalline thermoplastics crystallized isothermally from the molten state or amorphous state exhibit two endothermic peaks in a DSC thermogram. Two mechanisms have been proposed to explain the multiple melting phenomena in these high-melting semicrystalline thermoplastics.^{20,23,25,34–41} However, the effect of the second component on the multimelting behavior has been little reported.^{21,22} In our earlier work,²² the effect of the melting time and annealing time at a temperature closer to the normal melting point of PPS on the multiple melting behavior of neat PPS and the

PPS component in the PEEK/PPS blends prepared by powder mixing was studied. It was found that the double endothermic peak of PPS annealed at 275°C for less than 3 h is different from that annealed for 12 h.

In this article, the effect of the annealing temperature on the melting characteristics of the cold-crystallized specimens was investigated for PPS and its blends. The DSC results for PPS and its blends annealed for 1 h at 240 and 270°C , respectively, and at 280°C for 4 h are shown in Figures 4–6 and Table III. In general, the observed melting characteristics of the PPS and its blends were comparable. Examination of Figure 4 reveals a characteristic double melting behavior for the PPS and its blends annealed at 240°C for 1 h. The addition of a second component does not influence the formation of a double melting peak. The temperatures of the lower (T_{m1}) and upper (T_{m2}) melting peaks are higher than the annealing temperature (T_a). The low-temperature melting peak is observed at about 10°C above the annealing temperature. A small increase in the temperature of the supermelting peak (T_{m2}) was observed, especially in the presence of the PEEK component. The addition of BMI decreases the heat of fusion (ΔH_m) of PPS. When the PPS

Table III Melting Parameters of PPS in Its Blends Annealed at Different Conditions

	T_a ($^\circ\text{C}$)	t_a (h)	T_{m0} ($^\circ\text{C}$)	T_{m1} ($^\circ\text{C}$)	T_{m2} ($^\circ\text{C}$)	ΔH_m (J/g)	ΔT_m ($^\circ\text{C}$)
PPS	240	1	249.0	255.3	281.5	48.48	51.4
PSF/PPS = 20/80	240	1	249.9	256.6	284.8	47.21	52.5
PEK-C/PPS = 20/80	240	1	250.2	256.6	286.0	47.90	49.8
PEEK/PPS = 20/80	240	1	251.8	258.5	288.1	49.50	53.9
BMI/PPS = 20/80	240	1	249.9	255.2	283.0	42.80	47.3
PPS	270	1	279.8		286.5	51.99	
PSF/PPS = 20/80	270	1	279.1		286.5	48.88	
PEK-C/PPS = 20/80	270	1	278.1		286.9	49.81	
PEEK/PPS = 20/80	270	1	279.6		286.3	49.28	
BMI/PPS = 20/80	270	1	279.0		286.9	39.06	
PPS	275	1	284.0		290.8	45.99	24.6
PSF/PPS = 20/80	275	1	281.2		289.0	46.19	21.1
PEK-C/PPS = 20/80	275	1	282.8		289.2	45.15	19.7
PEEK/PPS = 20/80	275	1	284.0		291.3	44.89	23.9
BMI/PPS = 20/80	275	1	284.4		291.2	38.29	22.5
PPS	280	4	285.3	270.2	292.9	48.97	
PSF/PPS = 20/80	280	4	288.7	270.0	294.8	44.23	
PEK-C/PPS = 20/80	280	4	287.5	272.0	292.6	44.93	
PEEK/PPS = 20/80	280	4	289.7	273.9	295.2	40.48	
BMI/PPS = 20/80	280	4	286.4	267.6	294.3	37.82	

ΔT_m , width of the melting peak.

blends were annealed at 270 and 275°C for 1 h, only one melting peak and a small increase in the melting temperature with increasing T_a can be observed. The double melting peak was also observed for the PPS blends annealed at 280°C for 4 h. However, the T_{m1} is lower than the T_a and the normal melting point, which is attributed to the melting of crystalline PPS with a lower T_m formed during the cooling process from the annealing temperature. When T_a is near the normal melting point, the T_m of PPS, only a small amount of crystalline PPS with a lower T_m was melted and recrystallized during the cooling process from the annealing temperature. The T_{m2} is due to the melting of crystalline PPS formed during the annealing process. Many authors have shown that the observed high-temperature melting peak is independent of T_a for the cold-crystallized samples⁴² and suggested that it should result from the melt, recrystallization, and subsequent remelt of the crystalline species with a lower melting point. However, it can be seen from Table III that the temperature of the supermelting peak of cold-crystallized PPS and its blends also displays an increase with increasing T_a as T_a is close to the T_m of PPS. It must be attributed to the presence of two distinct crystalline morphology populations.¹⁸ The double melting peak can also be observed when PPS and its blends are crystallized dynamically from the molten state at 320°C at a cooling rate of 20°C/min (Fig. 7). However, the intensity of the low-temperature melting peak is not significant.

CONCLUSIONS

1. Various polymers have different effects on the crystallization behavior of polyphenylene sulfide (PPS). The addition of amorphous thermoplastics bisphenol A polysulfone (PSF) and phenolphthalein poly(ether ketone) (PEK-C) has no influence on the crystallization temperature (T_c) and heat of crystallization (ΔH_c) of PPS. A significant increase in the T_c value and in the intensity of the T_c peak of PPS and a decrease in the degree of supercooling of PPS were observed in the presence of the poly(ether ether ketone) (PEEK) component. An increase in the T_c of PPS was also observed in the bismaleimide (BMI)/PPS blend. However, the shift of the T_c to high temperature is no more significant than that in the PEEK/PPS blend. The increase in the T_c of PPS was attributed to the acceleration of nucleation in the presence of the

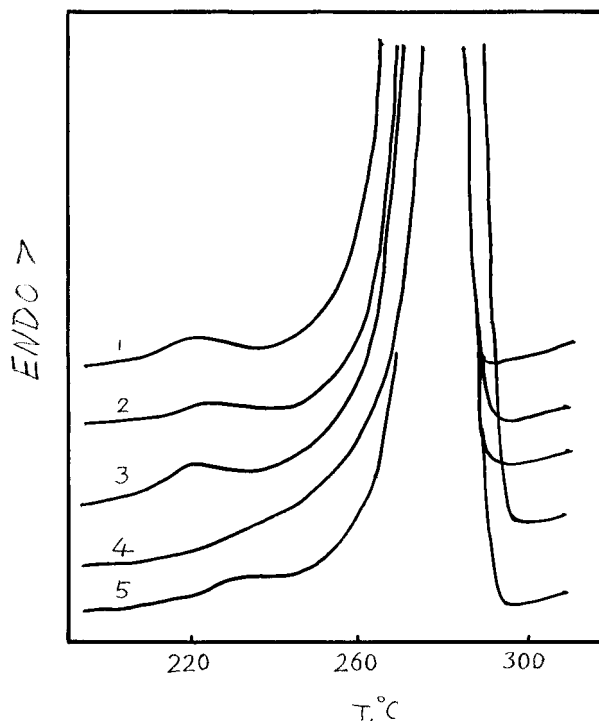


Figure 7 DSC heating scans of PPS and its blends crystallized dynamically at a cooling rate of 20°C/min: (1) PPS; (2) PSF/PPS = 20/80; (3) PEK-C/PPS = 20/80; (4) PEEK/PPS = 20/80; (5) BMI/PPS = 20/80.

particles of crystalline PEEK and cured BMI resin.

2. The T_c of PPS in the PEEK/PPS blends is dependent on the maximum temperature of the heating scans (T_{max}) in the DSC experiments and can be divided into three temperature regions: (1) When T_{max} is below 340°C, the increase in the T_c of PPS is more significant. The T_c is above 235°C and slightly increases as the number of heating scans increases. (2) When T_{max} is between 340 and 360°C, the T_c of the PPS in its blends is about 225°C. (3) When T_{max} is equal to or higher than the equilibrium melting point of PPS, the T_c of PPS is below 210°C and the action of the increase in the T_c of PPS was not observed. It is suggested that the crystallization behavior of PPS in the presence of the PEEK component depends upon the condensed state of the PEEK component.
3. The addition of a second component has no influence on the formation of a multiple melting peak. The observed melting characteristics of PPS and its blends were comparable in the experimental conditions of this article. The multiple melting behavior can also be ob-

served when PPS and its blends are crystallized dynamically from the molten state, but the intensity of the low-temperature melting peak is not significant.

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