Miscibility and Mechanical Properties of Tetrafunctional Epoxy Resin/Phenolphthalein Poly(ether ether ketone) Blends

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ABSTRACT: Phenolphthalein poly(ether ether ketone) (PEK-C) was found to be miscible with uncured tetraglycidyl 4,4′-diaminodiphenylmethane (TGDDM), which is a type of tetrafunctional epoxy resin (ER), as shown by the existence of a single glass transition temperature (T_g) within the whole composition range. The miscibility between PEK-C and TGDDM is considered to be due mainly to entropy contribution. Furthermore, blends of PEK-C and TGDDM cured with 4,4′-diaminodiphenylmethane (DDM) were studied using dynamic mechanical analysis (DMA), Fourier-transform infrared (FTIR) spectroscopy, and scanning electron microscopy (SEM). DMA studies show that the DDM-cured TGDDM/PEK-C blends have only one T_g. SEM observation also confirmed that the blends were homogeneous. FTIR studies showed that the curing reaction is incomplete due to the high viscosity of PEK-C. As the PEK-C content increased, the tensile properties of the blends decreased slightly and the fracture toughness factor also showed a slight decreasing tendency, presumably due to the reduced crosslink density of the epoxy network. SEM observation of the fracture surfaces of fracture toughness test specimens showed the brittle nature of the fracture for the pure ER and its blends with PEK-C. © 2000 John Wiley & Sons, Inc. J Appl Polym Sci 79: 598–607, 2001

Key words: epoxy resin; phenolphthalein poly(ether ether ketone); polymer blends)

INTRODUCTION

Epoxy resins (ERs) are one of the most important classes of thermosetting polymers. They are used as matrices for fiber-reinforced composite materials, adhesives, and coatings as well as other engineering materials.[1–4] However, due to their tightly crosslinked structure, these materials have some undesirable properties, such as low toughness and poor crack resistance, which constrain their many end-use applications. One of the most successful methods of improving the toughness of ERs is to incorporate a second phase of dispersed rubbery particles into the crosslinked polymer.[5–17] In these systems, toughening is considered to arise mainly from shear deformation in the matrices and the presence of rubber particles.[9,18–20] Unfortunately, the addition of rubbery materials to ERs has been shown to lower their glass transition temperatures (T_g's) and thermal and oxidative stability while enhancing their fracture toughness. However, the rubber-toughening technique was not found to be effective for tetrafunctional ERs because the high crosslink density constrains the shear-yielding deformation.

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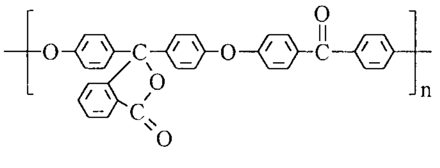
Recently, many attempts have been made to modify ERs with high-performance engineering thermoplastics.[20–35] Various types of thermoplastics, such as poly(ether sulfone) (PES),[18,21,22] polyetherimide (PEI),[20,23–25] and poly(ether ether ketone) (PEEK)[26–28] have been employed to toughen ERs. Two reviews of the developments in the area of thermoplastic-modified ER are available.[36,37] The enhanced toughness of the system has been suggested to arise from crack pinning and the rupture of the dispersed thermoplastics.[18,19] Some results indicate that a fine phase-separated structure and good interfacial adhesion between the two separated phases usually yields greater fracture toughness. Since the resulting morphology and extent of phase separation is known to affect the optical and mechanical properties of the cured blends, systematic studies of the relationship of phase behavior and mechanical properties of the thermosetting blends are of great importance.

In previous work on thermoplastic modifications of ERs,[38–46] we studied difunctional ERs, for example, diglycidyl ether of bisphenol A (DGEBA)/thermoplastic blends. Tetrafunctional ERs, which have a higher crosslink density than that of difunctional ERs, have been increasingly used in applications for advanced composite materials in the aerospace and aircraft industries.[47–50] Therefore, it is not only of academic importance but also of practical interest to study the toughening mechanism of tetrafunctional ER/thermoplastic blends. As a part of a series of studies, this work concerns an investigation on the miscibility and mechanical porperties in blends of a kind of tetrafunctional ER, that is, tetraglycidyl 4,4′-diaminophenylmethane (TGDDM) and phenolphthalein poly(ether ether ketone) (PEK-C). PEK-C is a recently developed high-performance engineering thermoplastic[51] with the following repeat unit:


PEK-C

PEK-C is an amorphous polymer with a glass transition temperature (T_g) of 228°C as measured by DSC. It was chosen as a modifier because of its relatively low price, excellent properties, and high temperature stability. In our work, PEK-C was physically blended with TGDDM and 4,4′-diaminodiphenylmethane (DDM) was used as the curing agent. The miscibility of both the uncured TGDDM/PEK-C and the DDM-cured TGDDM/PEK-C blends was studied. Furthermore, the tensile properties and fracture toughness of the DDM-cured blends are presented.

EXPERIMENTAL

Materials and Preparation of Samples

The tetrafunctional ER AG-80 with an epoxide equivalent weight 115–135, that is, TGDDM, was obtained from the Shanghai Institute of Synthetic Resins (Shanghai, China). PEK-C with a reduced viscosity $[\eta] = 1.10$ dL/g in a solution of chloroform at 25°C was supplied by the Xuzhou Chemical Factory (Xuzhou, Jiangsu, China). PEK-C was dried at 150°C for 2 h before using. The curing agent was chemically pure grade DDM and was purchased from the Shanghai Reagent Co. (Shanghai, China).

Uncured TGDDM/PEK-C blends were prepared by solution casting from chloroform; the solvent was evaporated slowly at room temperature. The residual solvent was removed under a vacuum at ambient temperature for 2 weeks.

To prepare the DDM-cured TGDDM/PEK-C blends, PEK-C was first dissolved in TGDDM with continuous stirring at 140°C. The mixture obtained was degassed under a vacuum for 0.5 h and then cooled to 100°C. Then, DDM was added to the mixture as the curing agent at 100°C with continuous stirring until a homogeneous ternary mixture was obtained. The ternary mixture was cured in a Teflon mold which was highly polished. The samples were cured successively at 80°C for 2 h, 140°C for 2 h, and 180°C for 2 h and postcured at 220°C for 5 h.

Dynamic Mechanical Analysis (DMA)

DMA was performed on a multifunctional internal friction instrument with temperature scanning from ambient temperature to 270°C using a torsion pendulum mode, at a frequency of 5 Hz and a heating rate of 1.5°C/min. The specimen dimensions were $2.0 \times 0.5 \times 0.1$ cm^3.

Differential Scanning Calorimetry (DSC)

Calorimetric measurements were performed on a Perkin–Elmer DSC-2C thermal analysis apparatus in a dry nitrogen atmosphere. To remove the thermal history of the samples, a thermal pretreatment was used. All samples (about 10 mg in weight) were first heated to 250°C and held for 3 min and then quenched to −60°C. All DSC curves were recorded at a heating rate of 20°C/min. The glass transition temperatures (T_g's) were taken as the midpoint of the capacity change.

Fourier Transform Infrared (FTIR) Spectroscopy

A Bomem MB104 FTIR spectrometer was used to study the curing reaction of the ERs. Samples of cured ER were pressed into a pellet together with KBr and used for the measurements. All spectra were recorded at room temperature with 64 scans at a resolution of 2 cm^{-1} and were signal-averaged.

Morphological Observation

To investigate the phase morphology of the cured ER/PEK-C blends, the specimens were fractured under cryogenic conditions using liquid nitrogen. The fractured surfaces thus obtained were then immersed in chloroform at room temperature for 15 h. The PEK-C content was preferentially etched by the solvent while the cured epoxy network remained unaffected. The etched samples were dried to remove the solvent. The K_{IC} fracture surface was also observed. A Hitachi X-650 scanning electron microscope (SEM) was used to observe the surfaces which were coated with thin layers (200 Å) of gold.

Tensile Tests

Tensile tests were carried out on a DCS-5000 testing machine (Shimadzu Co. Ltd., Japan) at ambient temperature (25°C). Standard dumbbell specimens (ASTM D638) with a $2.5 \times 0.6 \times 0.4$-$cm^3$ neck were used. The crosshead speed was 2 mm/min and a minimum of five specimens were tested in each case to obtain the average value.

Fracture Toughness

Fracture toughness was measured using a notched three-point bending test with a crosshead speed of 1.3 mm/s according to ASTM E399 on a MTS-810 material test system. A schematic diagram of a three-point bending specimen is shown in Figure 1. Central vee-notches were machined in the bars and extended by pressing a fresh razor blade into the tip of the notch to give a crack length of 3.3 ± 0.3 mm. The thickness of the specimen was about 8 mm and a minimum of five specimens were tested in all cases.

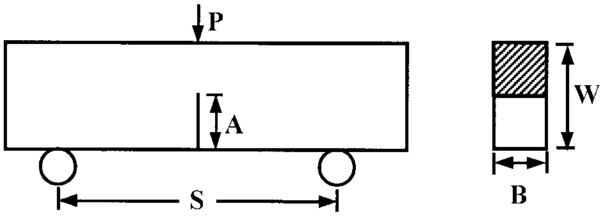


Figure 1 Schematic diagram of three-point bending specimen for measuring fracture toughness.

The critical stress intensity factor, K_{IC}, that is, fracture toughness, was calculated for all the samples using the equation

$$K_{IC} = [P_C S / B W^{3/2}] f(A/W) \tag{1}$$

where P_C is the load at crack initiation; B, the thickness of the specimen; S, the span width; W, the width of the specimen; and A, the crack length (see Fig. 1). The critical strain energy release rate G_{IC}, that is, the fracture energy, was calculated from K_{IC} as

$$G_{IC} = (1 - \gamma^2) K_{IC}^2 / E \tag{2}$$

where γ is Poisson's ratio and E is the tensile modulus.

RESULTS AND DISCUSSION

Miscibility of TGDDM/PEK-C Blends

All the TGDDM/PEK-C blends prepared as described above were transparent at ambient temperature. After additional heating to 250°C, these blends still remained clear. Therefore, there was no visual evidence of phase separation in the TGDDM/PEK-C blends.

All the blends were subjected to thermal analysis and were found to have a single T_g. Figure 2 summarizes the T_g values obtained from DSC as a function of blend composition. According to the transparency and glass transition behavior, the conclusion is that TGDDM/PEK-C blends are clearly miscible at the entire composition.

Several theoretical and empirical equations have been used to describe the T_g-composition of miscible polymer blends. One of these, the Gordon–Taylor equation,[52] is written as

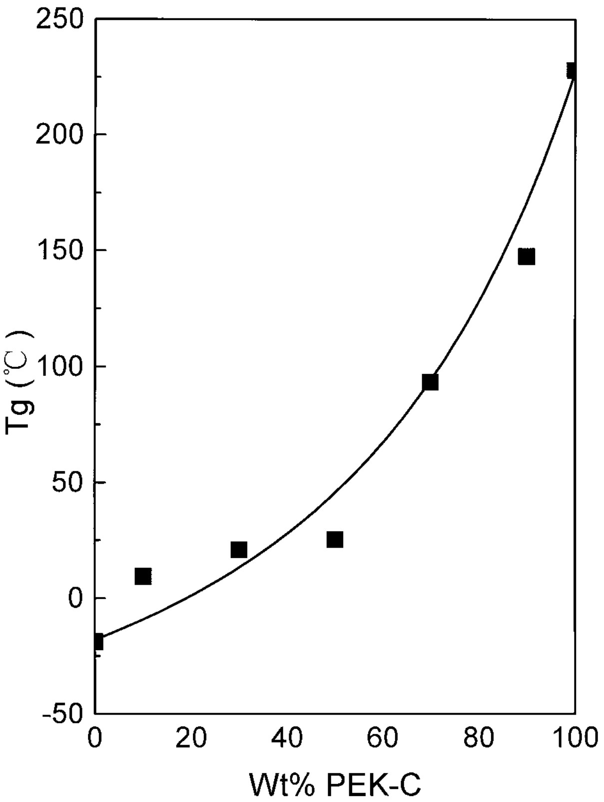


Figure 2 Composition dependence of glass transition temperature of TGDDM/PEK-C blends. The curve is as predicted by the Gordon–Taylor equation using a *k* value of 0.36.

$$T_g = (W_1T_{g_1} + kW_2T_{g_2})/(W_1 + kW_2) \quad (3)$$

where T_g is the glass transition temperature of the blend; T_{g_1} and T_{g_2}, the glass transition temperatures of components 1 and 2, respectively; W, the weight fraction, and k, a constant. The curve in Figure 2 was drawn using the Gordon–Taylor equation with a k value of 0.36, fitting the experimental data relatively well.

Prud'homme et al.[53,54] suggested that k can be taken as a semiquantitative measure of the strength of the interaction between the components of the blend. For instance, in blends of poly(ε-caprolactone) with chlorinated polyethylene, poly(vinyl chloride) (PVC), and chlorinated PVC, k increases from 0.26 to 1.0. When such an approach is used for the TGDDM/PEK-C blends, a k value of 0.36 suggests a weak interaction between TGDDM and PEK-C. The miscibility of the TGDDM/PEK-C blends should be considered to be due mainly to the entropy contribution as the molecular weight of TGDDM is rather low.

Phase Behavior of DDM-Cured TGDDM/PEK-C Blends

All the DDM-cured TGDDM/PEK-C blends studied were transparent at room temperature. With further heating to 280°C, these blends still remained clear. This observation primarily suggests that these blends were homogeneous and had a single-phase structure.

Dynamic Mechanical Analysis

Presented in Figure 3 are the dynamic mechanical spectra for the pure DDM-cured TGDDM [Fig. 3(a)] and the DDM-cured TGDDM/PEK-C blends [Fig. 3(b–e)]. Only a single glass transition peak was observed in the mechanical spectra of the specimens. Both the transparency and the presence of only a single T_g shows that the blends are homogeneous. We can also see from Figure 3 that as the PEK-C content increased, the relative width of the glass transition peak increased. The width of the glass transition can reflect the magnitude of local composition fluctuations in the polymer blends, and hence the relative homogeneity or miscibility of the system. The increasing peak width with PEK-C content can indirectly imply that the blends are miscible.

Figure 4 shows the T_g of all the DDM-cured TGDDM/PEK-C blends obtained from DMA. We found that there exist the highest T_g when the PEK-C is 5 wt %. A similar phenomenon was reported in our previous work of DDM-cured DGEBA/PEK-C blends.[46] The first increase of T_g is due to the high T_g of PEK-C (228°C by DSC) and the sequential decrease is caused by the reduced crosslink density of the epoxy network because the curing reaction is incomplete as will be seen below from FTIR analysis.

Phase Morphology

The morphology of the cured blends was investigated using scanning electron microscopy (SEM). SEM micrographs of solvent-etched fracture ends of pure DDM-cured TGDDM and 95/5and 90/10 DDM-cured ER/PEK-C are presented in Figure 5. There is no evidence that the blends are heterogeneous. The SEM observation supports the DMA result.

In our previous work,[27,46] we studied difunctional ER/PEK-C blends cured by DDM. If the blends were prepared at a higher initial curing temperature of 150°C, they were heterogeneous[27]; however, if the initial curing tempera-

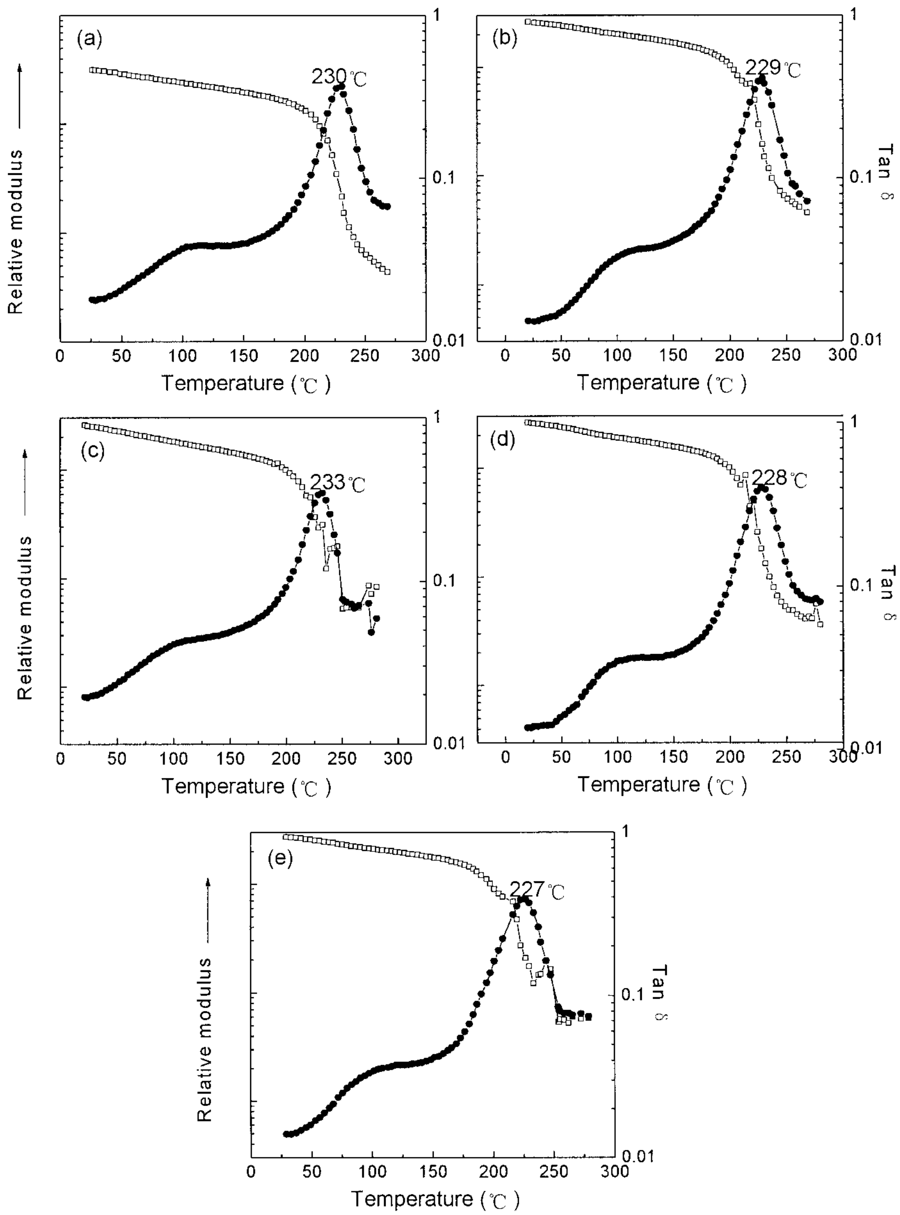


Figure 3 Dynamic mechanical spectra of (a) 100/0, (b) 97.5/2/5, (c) 95/5, (d) 92.5/7.5, and (e) 90/10 DDM-cured TGDDM/PEK-C blends.

ture was 80°C, which is the same as in this article, no phase separation was observed,[46] which is in accord with this article's result. The curing condition can greatly influence the final phase structure. The phase behavior and morphology of a thermosetting polymer blend is determined by both thermodynamic and kinetic factors. The cure process of these kinds of blends involves several stages.[55] Because one component of this blend is crosslinkable, a semi-interpenetrating network (semi-IPN) or interpenetrating network (IPN) can form. Because of the semi-IPN or IPN system's interpenetrating structure, the initial immiscible component may have no chance to show phase separation, that is, the system is compatible. It needs to be pointed out that when the interaction between the two components of the blends is strong, the enthalpy effect must be included. Sometimes, the strong hydrogen-banding interaction between the components favors miscibility. On the other hand, increase in the molecular weight causes a decrease in the configurational entropy of mixing as the curing reaction proceeds, which may cause phase separation in initially

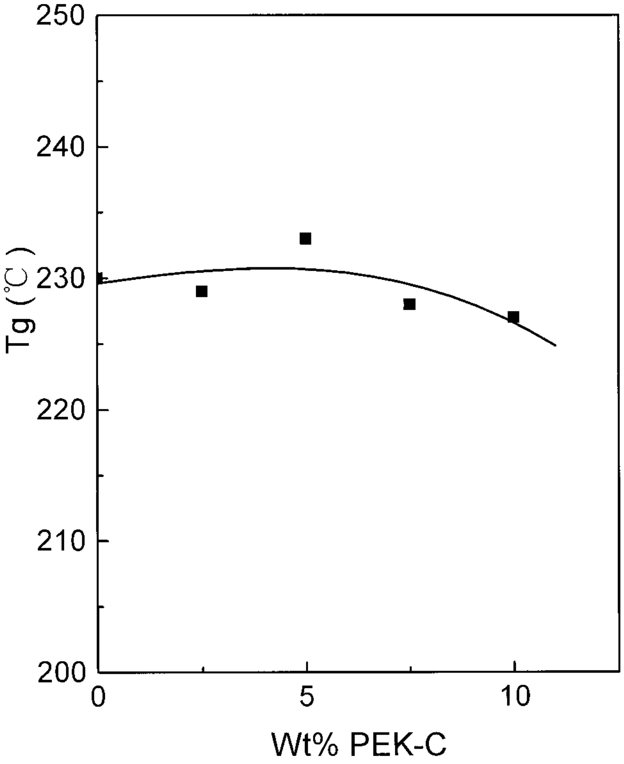


Figure 4 T_g of pure DDM-cured TGDDM and all the DDM-cured TGDDM/PEK-C blends obtained from the DMA data as a function of PEK-C content.

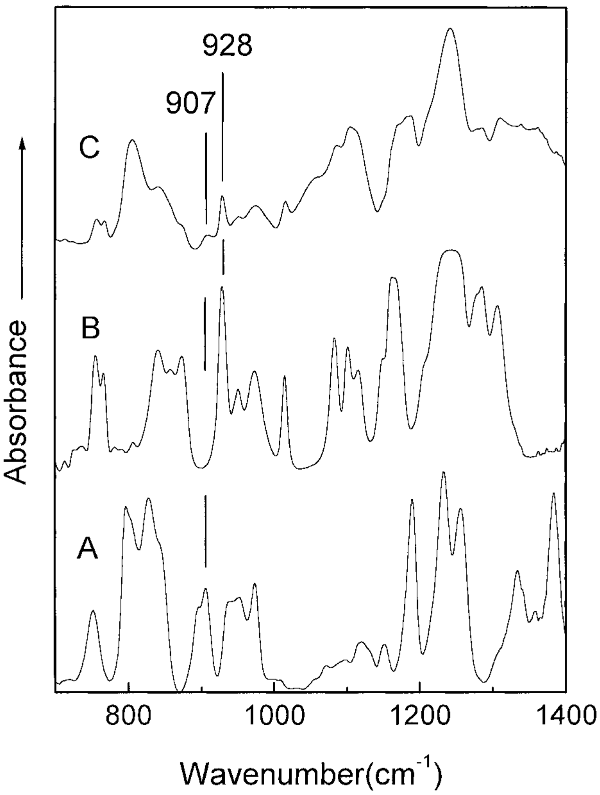


Figure 6 FTIR spectra in the 700–1400 cm^{-1} region of (a) TGDDM, (b) PEK-C, and (c) 90/10 DDM-cured TGDDM/PEK-C blends.

miscible blends. The phase behavior is influenced by a kinetic factor as well. When the curing temperature increases, the molecular and segmental motion increases and the rate of phase separation increases. Meanwhile, the increased temperature causes an increase in the curing rate and the time to the gelation point is reduced. Therefore, gela-

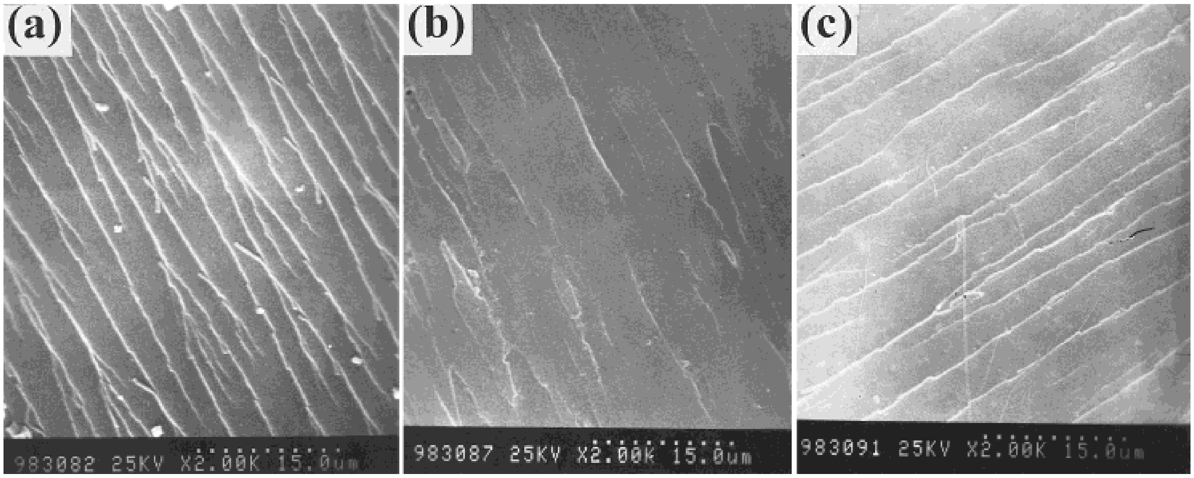


Figure 5 SEM micrographs of fracture surfaces of (a) pure DDM-cured TGDDM and (b) 95/5 and (c) 90/10 DDM-cured TGDDM/PEK-C blends etched with chloroform for 15 h.

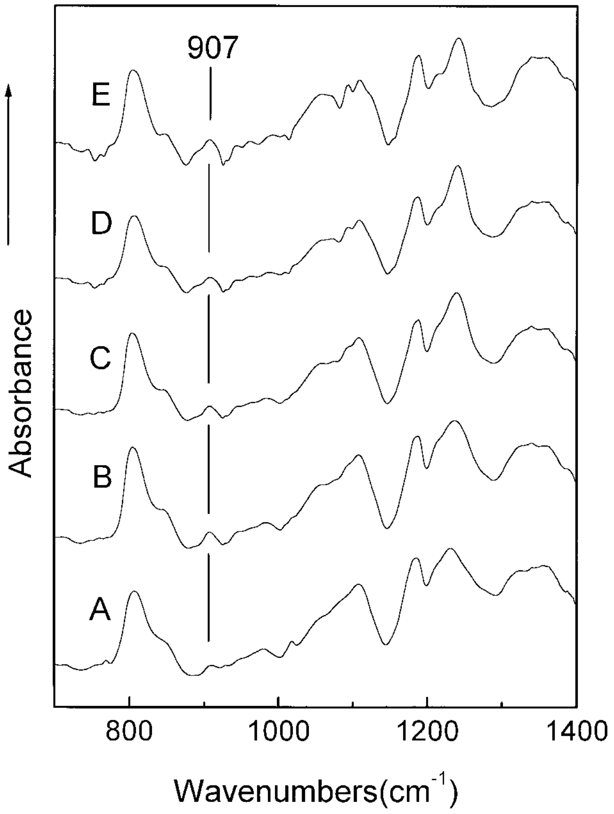


Figure 7 FTIR spectra in the 700–1400 cm^{-1} region of the DDM-cured TGDDM/PEK-C blends. ER/PEK-C: (a) 100/0,; (b) 97.5/2/5; (c) 95/5; (d) 92.5/7.5; (e) 90/10.

tion and phase separation are two competitive kinetic processes. If gelation is accomplished first, there will be no phase separation; otherwise, the blend will be heterogeneous. Our work with ER/PEK-C blends showed that the elevation of the initial curing temperature is beneficial to phase separation, that is, the kinetic factor of phase separation is dominant in this system.

Fourier Transform Infrared (FTIR) Analysis

The T_g's obtained from the DMA results suggest that as the PEK-C content increases the T_g of the blend first increased and then decreased. To explain this phenomenon, FTIR spectroscopy was used to observe whether the curing reaction was complete or not.

The spectra of pure TGDDM, pure PEK-C, and DDM-cured TGDDM/PEK-C blends (PEK-C10 wt %) are shown in the range of 700–1400 cm^{-1} in Figure 6. There are a 907 cm^{-1} peak, which suggests the unreacted oxirane ring[56] and a 928 cm^{-1} peak, which suggests the C—O—C unit of the side chain of PEK-C. To separate the influence of the PEK-C's 928 cm^{-1} peak, we used a spectral subtraction technique based on the method described by Banerjee and Friese.[57,58] to subtract the PEK-C component from the blends. Figure 7 shows the pure DDM-cured TGDDM and the subtracted spectra of the DDM-cured TGDDM/PEK-C blends. Little unreacted oxirane remains in the pure DDM-cured TGDDM. As the PEK-C content increases, there is a significant increase in the 907 cm^{-1} peak strength, and this suggested that the curing reaction is becoming more and more incomplete. As we have mentioned, PEK-C was dissolved in TGDDM to form a homogeneous solution. Due to PEK-C's high viscosity ($[\eta] = 1.10$ dL/g), the addition of PEK-C increased the viscosity of the system remarkably. Therefore, the segment motion became more and more difficult with increasing PEK-C content. Thus, the curing reaction became difficult to complete. The greater the PEK-C content, the more viscous the system and the more incomplete the curing reaction.

There are two factors influencing the T_g of the DDM-cured TGDDM/PEK-C blends. One is the high T_g of PEK-C, which leads to increase of the T_g of the blends; the other is the decrease of the

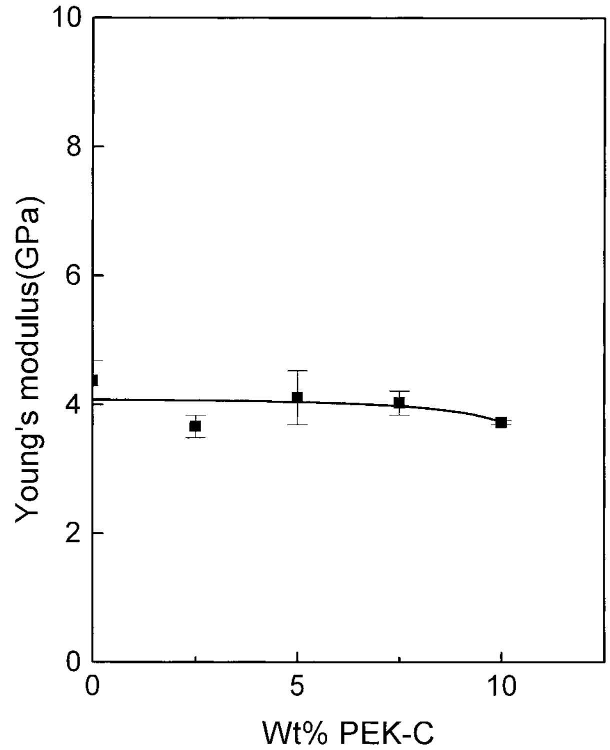


Figure 8 Tensile modulus as a function of PEK-C content for the DDM-cured TGDDM/PEK-C blends.

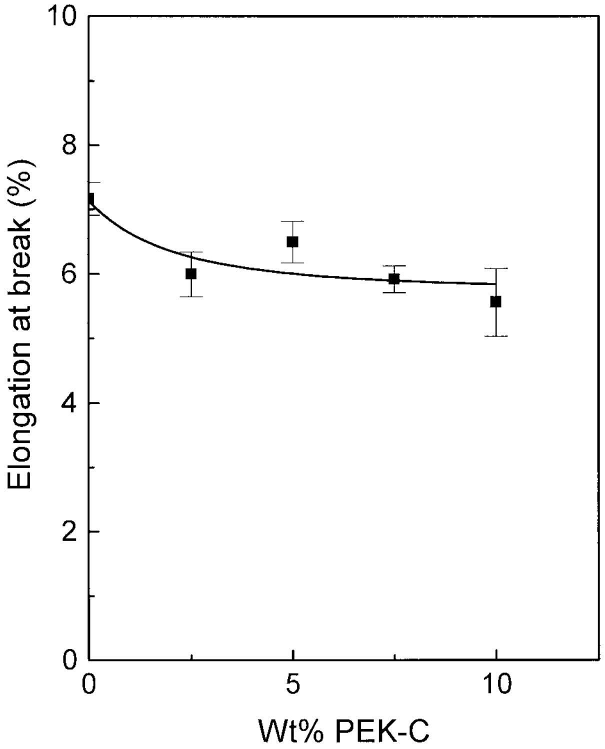


Figure 9 Elongation at break as a function of PEK-C content for the DDM-cured TGDDM/PEK-C blends.

crosslink density which is influenced by the curing reaction degree and results in a decrease of the T_g of the blends. The competition of the two factors results in the first increase and the sequential decrease of the T_g of the blends system.

Mechanical Properties of DDM-cured TGDDM/PEK-C Blends

Tensile Properties

No yields were observed on the stress–strain curves of the pure DDM-cured ER and all the blends, showing that DDM-cured TGDDM and its blends with PEK-C were basically brittle materials at room temperature. The changes of the tensile modulus, stress, and elongation at break are plotted in Figures 8–10, respectively, as functions of the blend composition. It can be seen that the tensile modulus is little changed while the tensile stress at break and elongation at break all show basically a slight decrease with increase of the PEK-C content. The addition of PEK-C slightly reduced the tensile properties of the cured ER.

Fracture Toughness

The plot of K_{IC} and G_{IC} versus PEK-C content in the cured blends is presented in Figure 11. As

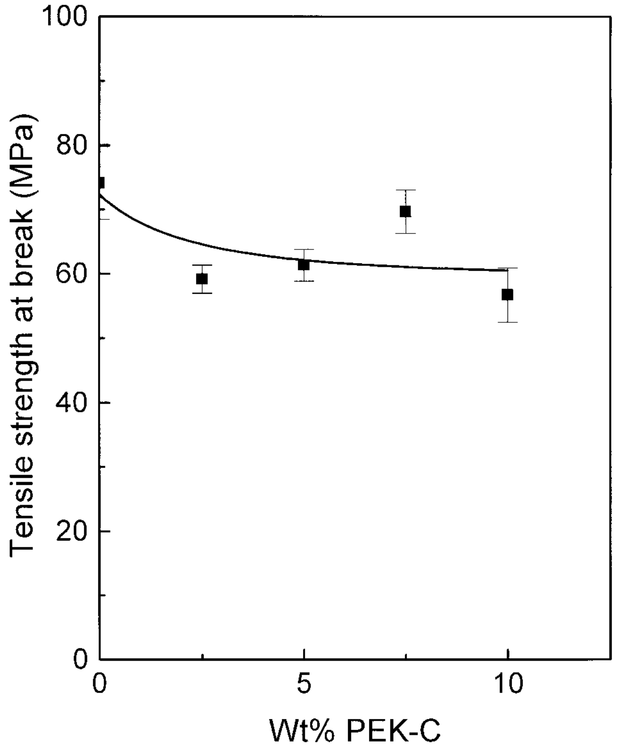


Figure 10 Tensile strength at break as a function of PEK-C content for the DDM-cured TGDDM/PEK-C blends.

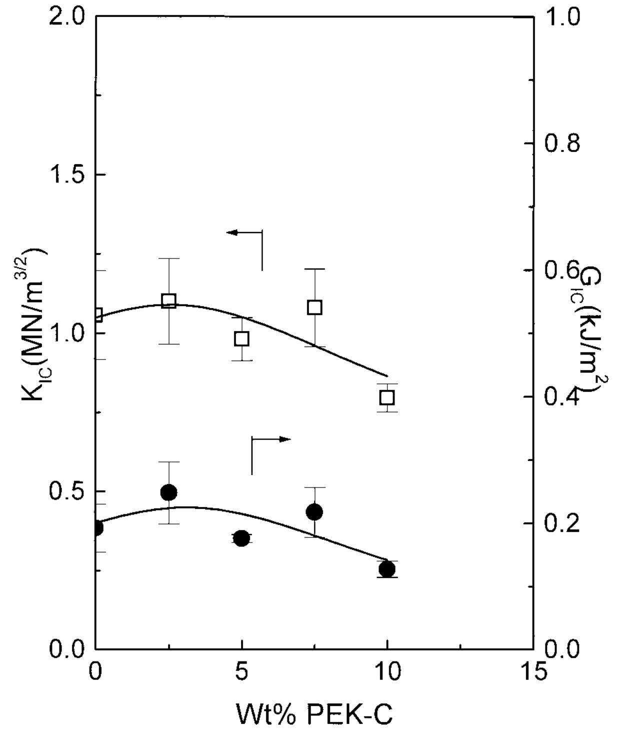


Figure 11 K_{IC} and G_{IC} as functions of PEK-C content for the DDM-cured TGDDM/PEK-C blends.

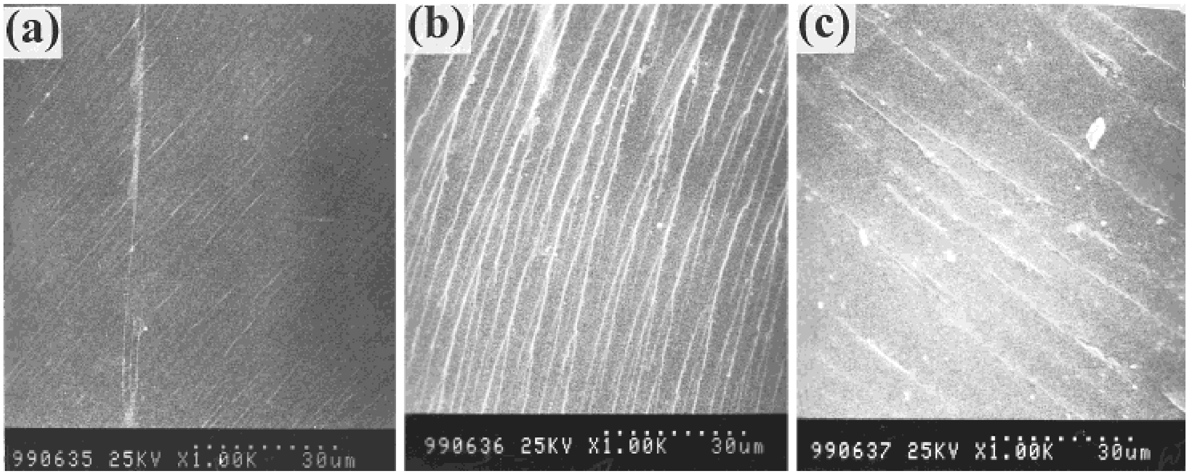


Figure 12 SEM micrographs of K_{IC} fracture surface of (a) pure DDM-cured TGDDM and (b) 95/5 and (c) 90/10 DDM-cured TGDDM/PEK-C blends.

observed, the fracture toughness is not changed greatly and has a tendency to decrease when the PEK-C content increases. Figure 12 shows the SEM micrograph of the K_{IC} fracture surface of the pure DDM-cured TGDDM and 95/5,90/10 DDM-cured TGDDM/PEK-C blends. The cracks spread freely and regularly and are oriented in the direction of loading, suggesting typical characteristics of brittle fracture.

Both the tensile properties and the fracture toughness test results suggest that PEK-C has no toughening effect on the DDM-cured TGDDM network. The lack of toughening in homogeneous blends was also reported by Chen et al. in the ER/polyetherimide system[59] and Jayle et al. in the ER/polycarbonate system.[60] In our previous work[46] of DDM-cured DGEBA/PEK-C blends, we studied difunctional ER and obtained the same result. On the other hand, we also used several relatively brittle polymers such as poly(styrene-*co*-acrylonitrile) (SAN) and poly(methyl methacrylate-*co*-styrene-*co*-acrylonitrile) (MSA) to study the relation between phase morphology and toughening,[61,62] which gave relatively good toughening results because of the phase separation structure. Due to these results, we should say that the phase separation structure is important in ER-toughening blend systems. In this work, the decreased tendency of fracture toughness as well as the slight decrease of tensile properties is ascribed to the reduced crosslink density of the epoxy network caused by the noncompletion of the curing reaction that has been mentioned.

CONCLUSIONS

The results presented here show that TGDDM/PEK-C blends are completely miscible over the entire composition. The miscibility between TGDDM and PEK-C is attributed mainly to entropy contribution. DMA and SEM studies revealed that the all the DDM-cured TGDDM/PEK-C blends undergo no phase separation and display a single-phase morphology. Tensile properties of the DDM-cured TGDDM/PEK-C blends were slightly reduced compared to those of the pure ER. Fracture toughness is slightly reduced and has a decreasing tendency with increase of the PEK-C content due to the noncompletion of the curing reaction as shown by FTIR analysis. Morphological investigation of the K_{IC} fracture surface of the blends shows typical characteristics of brittle fracture.

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