Effect of Poly(etherimide) Chemical Structures on the Properties of Epoxy/Poly(etherimide) Blends and Their Carbon Fiber-Reinforced Composites

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ABSTRACT: Poly(etherimide)s (PEIs) with different chemical structures were synthesized and characterized, which were employed to toughen epoxy resins (EP/PEI) and carbon fiber-reinforced epoxy composites (CF/EP/PEI). Experimental results revealed that the introduction of the fluorinated groups and meta linkages could help to improve the melt processability of EP/PEI resins. The EP/PEI resins showed obviously improved mechanical properties including tensile strength of 89.2 MPa, elongation at break of 4.7% and flexural strength of 144.2 MPa, and

INTRODUCTION

Epoxy resins are extensively used as matrices for high performance carbon fiber-reinforced polymers composites in military and commercial aircrafts and spaceflights due to their high modulus, corrosion resistance, and low shrinkage on curing.^{1–3} However, highly crosslinked epoxy resins are inherently brittle and have poor resistance to crack propagation,⁴ which limits their use for some advanced applications for which high toughness is highly required, such as carbon fiber-reinforced polymers for aerospace.

The most common method to increase the fracture toughness of epoxy resin is to incorporate thermoplastics to epoxy resins.^{5–11} Compared with other methods such as rubber toughening,¹² thermoplastic toughening can serve as tougheners to improve fracture toughness of epoxy resins without significantly compromising other properties. The frequently used thermoplastics are poly(etherimide) (PEI),^{5,6} polyester,⁷ polysulfone^{8,9} as well as poly(etheretherketone),^{10,11} which have been reported as good tougheners for epoxy resins. Among these thermoplastics,

good thermal properties including glass transition temperature (T_g) of 211°C and initial decomposition temperature (T_d) of 366°C. Moreover, CF/EP/PEI-1 and CF/EP/PEI-4 composites showed significantly improved toughness with impact toughness of 13.8 and 15.5 J/cm², respectively. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 119: 3162– 3169, 2011

Key words: poly(etherimide); epoxy resin; toughening; composites

it has been found that poly(etherimide) showed the outstanding thermal and mechanical properties with more obvious toughening effect.^{12,13} However, many existing poly(etherimide)s usually have poor melt processability for CF/EP composites prepared by hot-melt method because of the rigid molecular backbones and the strong polymer chain to chain interactions in poly(etherimide)s.

To improve the toughness and melt processability of PEI resin, in this study, the flexible structure such as meta linkage was introduced to the polymer chain and fluorinated substituent was also introduced to increase the steric hindrance and decease polymer chain to chain interactions. A series of novel molecular weight-controlled poly(etherimide)s (PEIs) with different chemical structures were synthesized by thermal polycondensation. Effects of the PEI's chemical structures on the properties of epoxy resins were systematically investigated. The mechanical and thermal properties of the PEI-toughened CF/EP composites prepared by hot-melt method were also determined.

EXPERIMENTAL

Materials

4,4'-diaminodiphenylsulfone (DDS) was purchased from Shanghai Institute of Synthetic Resins. 1,4-bis

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(4'-amino-2'-trifluoromethylphenoxy) benzene (1,4,4-6FAPB) and 1,4-bis (4-aminophenoxy) benzene (1,4,4-APB) were synthesized in this laboratory according to a reported method.¹⁴ 1,3-bis (4-amino-phenoxy) benzene (1,3,4-APB), 3,4'-oxydianiline (3,4'-ODA) and phthalic anhydride (PA) were obtained from Beijing Beihua Fine Chemicals Co., China. 2,2-bis [4-(3,4-dicarboxyphenoxy)phenyl] propane dianhydride (BPADA) (Shanghai Institute of Synthetic Resins) was dried at 120°C for 12 h under vacuum prior to use. Commercially available N-methyl-2-pyrrolidinone (NMP) was purified by vacuum distillation over CaH₂ and stored over 4-À molecular sieves before use. Toluene, isoquinoline and other solvents were used as received. Tetraglycidyldiamino diphenylmethane epoxy resin (TGDDM) was supplied by Shanghai Institute of Synthetic Resins (epoxy value, 0.80) and diglycidyl ester of aliphatic cyclo epoxy resin (DGEAC) was provided by Tianjin Jindong chemical factory (epoxy value, 0.85). T700 (12K) carbon fiber was obtained from Toray Co., Japan. The chemical structures of the materials as mentioned above are given in Table I.

Synthesis of poly(etherimide)s with different chemical structures

The poly(etherimide)s were synthesized via the high-temperature polycondensation procedure using NMP as solvent and toluene as azeotropic agent. In a typical experiment, the fluorinated poly(etherimide) (PEI-1) was prepared by the following procedure. In a 1000-mL, three-necked flask equipped with mechanical stirrer, thermometer, and Dean-Stark trap topped by condenser, 1,4,4-6FAPB (47.3447 g, 0.1105 mol), BPADA (55.4494 g, 0.1065 mmol) and PA (1.1850 g, 0.0080 mol) were placed, and the mixture of NMP/toluene (300/30 mL) and 0.5 mL of isoquinoline as a catalyst were then added with stirring in nitrogen atmosphere. The resulted homogeneous solution was heated at 180°C and stirred for 12 h. In the thermal polycondensation, the azeotrope was continuously distilled and collected in the Dean-Stark trap to promote the imidization proceeded smoothly. After cooled to room temperature, the solution was poured slowly into an excess amount of ethanol with vigorous agitation. The white precipitate was collected by filtration, washed thoroughly with ethanol and water, and dried at 200°C overnight. After pulverized, 97.3 g of white solid powders (PEI-1) was obtained (vield: 97%). Other PEIs with different chemical structures, i.e., PEI-2(BPADA-1,4,4-APB-PA), PEI-3(BPADA- 1,3,4-APB-PA), and PEI-4(BPADA-3,4'-ODA-PA), were prepared by the similar procedure as described above.



TABLE I Chemical Structures of the Materials

Preparation of PEI-toughened epoxy resins

In a 500-mL three-necked flask equipped with mechanical stirrer and thermometer, 50 g of TGDDM, 50 g of DGEAC, and 15 g of PEI-1 were placed, which was then heated with mechanical stirring at 140°C to give a homogeneous viscous liquid. Then 15 g DDS and 35 g 1,4,4-6FAPB were added and stirred to give a viscous and homogeneous liquid. After cooled down to 120°C, the viscous liquid was evacuated in <10 mmHg for 10–20 min. The evacuated liquid was transferred into an open mold preheated at 120°C, which was then cured in



Scheme 1 Synthesis of the molecular weight-controlled PEIs.

an air convection oven at 130°C for 2 h, 150°C for 1 h, 180°C for 3 h and then post cured at 200°C for 2 h, successfully, to give the thermally cured PEI-toughened epoxy resins (EP/PEI-1). The curing technology is determined by the onset temperature, peak temperature, and offset temperature of the curing curves of DSC analysis. Other thermally cured EP/PEI resins with different chemical structures of PEIs, i.e., EP/PEI-2, EP/PEI-3, and EP/PEI-4, were prepared by the similar procedure as described above.

Fabrication of the PEI-toughened CF/EP composites

The liquid matrix resin was employed to impregnate carbon fiber (T700) at 100°C to prepare unidirectional CF/EP prepregs in a laboratory-made barrel mold, and then thermally cured at 130°C for 2 h, 150°C for 1 h, 180°C for 3 h and post cured at 200°C for 2 h to give the CF/EP composite laminate. The target resin content for the fiber reinforced composite is about 40%, which is determined according to the GB/T 3855-1983.

Measurements

The inherent viscosities were measured with an Ubbelohde viscometer in NMP at 35° C (0.5 g/dL). Number-average molecular weights (M_n) were obtained via gel permeation chromatography (GPC) performed with Waters 2414 (DMAc was used as an eluent). ¹H Nuclear Magnetic Resonance (¹H NMR) spectra were recorded on a Varian Unity 400 spectrometer operating at 400 MHz in CDCl₃. Fourier Transform-infrared spectroscopy (FT-IR) spectra were collected on a PerkinElmer 782 FTIR spectrophotometer. Elemental analysis (EA) was performed using a Carlo Erbra EA 1108 elemental analyzer. Complex viscosity (η^*) was measured on a TA AR2000 rheometer at a heating rate of 4° C/min. T_{g} s were determined with a TA Instruments DSC Q100 in nitrogen atmosphere at a heating rate of 10°C/min. Thermogravimetric analysis (TGA) was performed on a TA Instruments TGA Q50 at a heating rate of 20°C/min in nitrogen atmosphere. Dynamic mechanical analysis (DMA) was performed on a TA Instruments DMA Q800 at a heating rate of 5°C/min and at a frequency of 1 Hz. Scanning electron microscope (SEM) analysis was performed on a Hitachi S-4300 SEM.

The mechanical properties except impact test were performed on an Instron Universal Tester Model 3365. The tensile properties of the solution-cast films were measured according to GB/T 13022-1991. The tensile properties of the cured resins were determined according to GB/T 16421-1996. Flexural properties of



Figure 1 FT-IR spectra of the poly(etherimide) (PEI1-4).

the cured resins were measured according to GB/T 16419-1996. Flexural properties of the carbon fiber reinforced composites were measured according to ASTM D 790-00. Impact strengths of the carbon fiber reinforced composites without notch were measured with a Reger Impact Tester according to GB 1451-83. Interlaminar shear strengths (ILSS) of the carbon fiber reinforced composites were measured test according to the ASTM D 2344.

RESULTS AND DISCUSSION

Synthesis and characterization of PEIs

The poly(etherimide)s have been prepared using the one-step high temperature polycondensation procedure (Scheme 1). Figure 1 shows FT-IR spectra of PEI1-4 in which the absorptions at 1780 and 1720 cm⁻¹ were attributed to the asymmetrical and symmetrical stretching vibrations of the imide groups. The band at 1380 cm⁻¹ was due to the C—N stretching vibration and those at 1100 and 725 cm⁻¹ were due to the imide ring deformation. The strong absorption in the region of 1100–1280 cm⁻¹ was assigned to the C—O stretching. The results of FT-IR and EA (Table II) agree well with the expected structures of PEIs. The molecular weights (M_n) and inher-

TABLE III Solubility in Organic Solvents of the Poly(etherimide)s at Room Temperature

Samples	NMP	DMAc	DMF	Methylene chloride	THF
PEI-1	≥25%	$\geq 25\%$	$\geq 25\%$	≥25%	≥25%
PEI-2	≥25%	10–15%	5–10%	15–20%	5–10%
PEI-3	≥25%	$\geq 25\%$	$\geq 25\%$	20–25%	20–25%
PEI-4	≥25%	$\geq 25\%$	$\geq 25\%$	20–25%	≥25%

ent viscosities are tabulated in Table II. The numberaverage molecular weights (M_n) of PEIs determined by GPC were close, which were in the range of 19,270–21,598. The inherent viscosities were measured in the range of 0.44 to 0.54 dL/g. Table III summarizes the solubility of the poly(etherimide)s in different organic solvents. It can be seen that PEI-1 and PEI-4 showed excellent solubility.

Melt processability of PEI-toughened epoxy resins

The melt processability of the EP/PEI resins were investigated by melt rheology. Figure 2 shows the melt viscosities of the EP/15 wt % PEI resins as a function of temperature. The melt viscosities of the EP/PEI resins were dependent on both the temperature and the chemical structures. The melt viscosities at 100°C of EP/PEI-1, EP/PEI-2, EP/PEI-3, and EP/ PEI-4 resins were 23.90, 59.17, 46.59, and 33.31 Pa s, respectively. The minimum viscosities of EP/PEI-1, EP/PEI-2, EP/PEI-3, and EP/PEI-4 resins were 0.38, 1.35, 1.06, and 0.88 Pa s, respectively. It is clear that EP/PEI-1 and EP/PEI-4 resins possessed better melt processability. The isothermal melt viscosities are shown in Figure 3. At the same cure procedure, EP/ 15 wt % PEI-1 and EP/15 wt % PEI-4 showed better melt processability, while EP/15 wt % PEI-2 exhibited worst melt processability. It can be seen that the time of EP/15 wt % PEI-1 and EP/15 wt % PEI-4 achieved to 10 Pa s were 91.5 and 83.7 min, respectively. The time of EP/15 wt % PEI-2 is 64.6 min which was obvious shorter that others. This may be attributed to the introduction of fluorinated groups and meta linkages in backbone. In addition, although PEI-3 has meta linkages in backbone, it also contains more para

TABLE II Molecular Weights, Inherent Viscosities, and Chemical Compositions of the Poly(etherimide)s

		Inherent	N	[%	C	.%	Н	%
Samples	M_n (g/mol)	viscosity (dL/g)	Calcd.	Found	Calcd.	Found	Calcd.	Found
PEI-1	23825	0.520	3.15	3.19	67.09	66.58	3.10	3.39
PEI-2	25405	0.582	3.68	3.71	75.64	74.98	4.13	4.24
PEI-3	21175	0.544	3.98	4.13	75.64	74.72	4.23	4.25
PEI-4	20983	0.440	4.16	4.20	75.31	74.56	4.11	4.24



Figure 2 Melt viscosities of EP/PEI as a function of temperature.

structures obtained from 1,3,4-APB, which may affect the melt processibility of EP/PEI-3 resins. The good melt processability of EP/PEI resin is an advantage for the production of CF/EP prepregs by hot-melt method which requires low viscosity of resin matrix to impregnate carbon fibers well.

Mechanical and thermal properties of PEIs

Table IV summarizes the mechanical and thermal properties of the PEIs including tensile properties, T_{g} , T_{d} , and temperature of maximum rate of degradation (T_{max}). As can be seen, PEI-1 and PEI-4 showed better mechanical properties with tensile strengths of 90.4 and 94.1 MPa, elongations at break of 5.6 and 5.3% and tensile moduli of 2.7 and 3.0 GPa, respectively. It can be also seen that PEI-2 showed better thermal properties with T_g of 211°C, T_d of 536°C, and T_{max} of 556°C, respectively. This might be because of the para structures of PEI-2 drove from 1,4,4-APB with all-para linkages.



Figure 3 Isothermal viscosities of EP/PEI at 130°C.

TABLE IV Mechanical and Thermal Properties of the Poly(etherimide)s

		2				
		PEI film		DSC	T	GA
Samples	Tensile strength (MPa)	Elongation at break (%)	Tensile modulus (GPa)	T _g (°C)	<i>T</i> _d (°C)	T _{max} (°C)
PEI-1 PEI-2 PEI-3 PEI-4	$\begin{array}{r} 90.4 \pm 5.2 \\ 84.0 \pm 1.9 \\ 83.5 \pm 3.2 \\ 94.1 \pm 5.4 \end{array}$	$\begin{array}{l} 5.6 \pm 0.3 \\ 5.8 \pm 0.5 \\ 5.1 \pm 0.3 \\ 5.1 \pm 0.9 \end{array}$	$\begin{array}{c} 2.7 \pm 0.2 \\ 2.4 \pm 0.1 \\ 2.5 \pm 0.1 \\ 3.0 \pm 0.2 \end{array}$	199 211 193 200	526 536 541 540	550 556 559 557

 T_d , the initial decomposed temperature; *T*max, temperature of maximum rate of degradation.

Mechanical properties of the toughened epoxy resins

Table V summarizes the tensile and flexural properties of the cured EP/PEI resins. It can be seen that the mechanical properties of the EP/PEIs were improved obviously. The tensile strength and elongation at break of EP/PEI could reach to 89.2 MPa and 4.7%, respectively. The flexural strength of EP/ PEI was improved to 144.8 MPa for 15 wt % PEI-1 loading, compared with 130.7 MPa of the untoughened resin. Meanwhile, no obvious deterioration in tensile and flexural moduli was observed. In comparison, the mechanical properties of the toughened epoxy resins by other tougheners such as liquid rubber,¹⁵ hyperbranched polymer,¹⁶ and even other thermoplastics^{17,18} were usually to some extent compromised.

Thermal properties of the toughened epoxy resins

Figure 4 depicts the storage moduli (E') and tan delta versus temperature for the thermally cured EP/PEI resins with different chemical structures of PEIs. It can be seen that two relaxations can be observed, indicating that phase separation in the thermal cured resins has occurred, which is required for the thermoplastic-toughened resins to achieve good toughness improvement. The relaxations between 192 and 200°C were assigned as the T_{os} of the PEI-rich phases including PEI-1, PEI-3, and PEI-4 and the relaxation at 218°C was the T_{α} of the PEI-2 rich phase, while the relaxations between 205 and 210°C correspond to the T_{gs} of the epoxy-rich phases. The T_g s obtained from the peaks of the epoxy-rich phase are listed in Table VI, which did not change significantly with the different chemical structures of PEIs.

Moreover, Figure 5 shows the TGA curves for the thermally cured EP/PEI matrix resins with different chemical structures of PEIs and the results are also tabulated in Table VI. It can be seen that both T_{ds}

Mechanical Properties of the Thermally Cured EP/PEI Resins						
Samples	Tensile strength (MPa)	Elongation at break (%)	Tensile modulus (GPa)	Flexural strength (MPa)	Flexural modulus (GPa)	
EP	62.0 ± 6.1	3.2 ± 0.5	2.6 ± 0.2	130.7 ± 10.1	3.7 ± 0.1	
EP/15 wt% PEI-1	80.9 ± 3.2	3.5 ± 0.2	$2.6~\pm~0.2$	130.9 ± 4.6	3.6 ± 0.3	
EP/20 wt% PEI-1	88.8 ± 6.2	4.7 ± 0.5	2.6 ± 0.3	140.9 ± 8.2	3.7 ± 0.2	
EP/15 wt% PEI-2	87.3 ± 5.3	4.3 ± 0.4	2.7 ± 0.1	144.8 ± 7.9	3.4 ± 0.1	
EP/20 wt% PEI-2	88.8 ± 6.5	4.7 ± 0.5	2.6 ± 0.1	141.0 ± 16.5	3.7 ± 0.1	
EP/15 wt% PEI-3	77.0 ± 5.2	3.9 ± 0.4	2.6 ± 0.2	136.1 ± 12.4	3.6 ± 0.4	
EP/20 wt% PEI-3	81.6 ± 3.8	4.2 ± 0.3	2.6 ± 0.1	133.2 ± 10.7	3.4 ± 0.2	
EP/15 wt% PEI-4	89.2 ± 2.5	4.4 ± 0.4	2.7 ± 0.2	130.9 ± 12.9	3.5 ± 0.1	
EP/20 wt% PEI-4	86.6 ± 6.1	4.4 ± 0.6	2.7 ± 0.2	144.2 ± 15.0	3.6 ± 0.2	

 TABLE V

 Mechanical Properties of the Thermally Cured EP/PEI Resins

and T_{max} s did not show obvious changes with the changes in chemical structures of PEIs, demonstrating that the PEI-toughened epoxy resins retain the essential thermal stability. And the PEI toughened epoxy resins have better thermal stabilities above 350°C because the PEI resins have better thermal stabilities compared with the neat epoxy resin.

SEM analysis of the toughened epoxy resins

Figure 6 shows the SEM micrographs of the solventetched fractured surfaces of the thermally cured EP/ PEI resins with different chemical structures of PEIs. It can be seen that all of the PEI-toughened resins exhibited dual-phase morphology. In addition, cocontinuous morphology, in which both the epoxyrich phase and the PEI-rich phase formed the continuous phase, formed in the all thermally cured EP/ PEI resins. It has been widely reported that thermoplastic-toughened epoxy resins with cocontinuous phase morphology usually show the better mechanical properties.^{19–21} In this work, the thermally cured EP/PEI resins with cocontinuous also showed outstanding mechanical properties. This is in good



Figure 4 Storage moduli (*E'*) and tan delta versus temperature for the thermally cured EP/PEI matrix resins with different chemical structures of PEIs.

agreement with the reports. The dominant toughening mechanism of the PEI-toughened epoxy matrix resins with cocontinuous morphology might be attributed to the ductile drawing of the thermoplastic-rich phase.²²

The carbon fiber-reinforced EP/PEI composites

Since EP/PEI-1 and EP/PEI-4 resins possess better melt processability, they were chosen as the matrix to manufacture carbon fiber reinforced EP/PEI composites by hot-melt method. Table VII depicts the mechanical properties and T_{gs} of CF/EP/PEI composites prepared by hot-melt method. T_{g} s were obtained from the peaks of tan delta. It can be seen that the impact strength of the CF/EP/PEI composites were improved obviously compared with the untoughened composite. For instance, the impact strengthof the CF/EP/PEI-1 and CF/EP/PEI-4 composites were determined at 13.6 and 15.5 J/cm², respectively, which were 37 and 57% higher than that of the un-toughened composite (9.9 J/cm^2) . Meanwhile, the flexural properties and T_{g} s of the composites were kept well by the incorporation of PEIs as toughener. For instance, the flexural strength and modulus of CF/EP/PEI-1 composite were 1.6 GPa and 93.6 GPa, respectively, compared with those of the un-toughened composite (1.5 GPa and

TABLE VI Thermal Properties of the Thermally Cured EP/PEI Resins

	DMA	TGA		
Blends	T_g (°C)/Tan delta	T_d (°C)	T_{\max} (°C)	
EP	209	360	411	
EP/15 wt % PEI-1	208	366	411	
EP/15 wt % PEI-2	209	361	418	
EP/15 wt % PEI-3	211	362	413	
EP/15 wt % PEI-4	210	359	413	

 T_d , the initial decomposed temperature; T_{max} , temperature of maximum rate of degradation.



Figure 5 TGA curves of the thermally cured EP/PEI matrix resins with different chemical structures of PEIs.

95.3 GPa). The T_g of CF/EP/PEI-1 composite was 203°C, compared with that of the untoughened composite (205°C). Clearly, the toughness of carbon

fiber-reinforced composite has been improved by the incorporation of poly(etherimide)s with no deterioration of mechanical strength and T_g .

SEM photographs of the CF/EP composites fractured surfaces after impact tests are shown in Figure 7. Dual phase morphology can be seen at the fractured surface of the CF/EP/PEI-4 composite [Figure 7(b)]. The fractured surface of the un-toughened CF/EP composite is flatness [Figure 7(a)], probably resulting in crack propagating smoothly on the matrix surface in the composite; in comparison, the CF/EP/PEI-4 composite shows toughener particles on the fractured surface [Figure 7(b)], which can confine and restrain the crack propagating where more energy will be required to continue the cracking.^{21,23}

CONCLUSIONS

Poly(etherimide)s with different chemical structures have been synthesized, which were used as the tougheners of the epoxy resins and carbon fiber-reinforced



Figure 6 SEM micrographs of the THF-etched fractured surfaces of the EP/PEI cured resins. (a) 15 wt % PEI-1; (b) 15 wt % PEI-2; (c) 15 wt % PEI-3; (d) 15 wt % PEI-4.

TABLE VII						
Mechanical and Thermal Properties of the Carbon Fiber-Reinforced						
Epoxy Composite Laminates						

		-			
T700 CF composites	Flexural strength (GPa)	Flexural modulus (GPa)	ILSS (MPa)	Impact strength (J/cm ²)	T _g /DMA (°C)
CF/EP CF/EP/15 wt % PEI-1 CF/EP/15 wt % PEI-4	$\begin{array}{l} 1.5 \pm 0.1 \\ 1.6 \pm 0.2 \\ 1.5 \pm 0.1 \end{array}$	95.3 ± 8.3 93.6 ± 3.5 93.6 ± 2.5	97.9 ± 2.6 90.2 ± 3.8 93.2 ± 1.8	9.9 ± 2.3 13.6 ± 4.0 15.5 ± 2.5	205 203 207



Figure 7 Comparison on the impact fractured surfaces of the un-toughened and PEI-toughened carbon fiber-reinforced composites. (a) Un-toughened CF/EP composite; (b) CF/EP composite toughened by 15 wt % PEI-4.

epoxy composites. The introduction of fluorinated groups and meta linkages could help to improve the melt processability of PEI-toughened epoxy resins. PEI-toughened epoxy resins showed obviously improved mechanical strength with tensile strength of 89.2 MPa, elongation at break of 4.7%, flexural strength of 144.8 MPa, respectively. PEI-toughened carbon fiber-reinforced composites showed good mechanical strength and toughness with flexural strength of 1.6 GPa, impact strength of 15.5 J/cm², respectively. Both thermal properties and mechanical moduli of PEI-toughened epoxy resins and PEI-toughened carbon fiber-reinforced composites were kept well.

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