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MECHANICAL PROPERTIES AND CURING KINETICS OF EPOXY RESINS CURED BY VARIOUS AMINO-TERMINATED POLYETHERS[*]

Li-ying Zhao, Jian-guo Guan[**], Hui-ru Ma and Zhi-gang Sun
State Key Laboratory of Advanced Technology for Materials Synthesis and Processing,
Wuhan University of Technology, Wuhan 430070, China

Abstract A high performance thermosetting epoxy resin crosslinkable at room temperature was obtained *via* directly moulding diglycidyl ether of bisphenol A (DGEBA) and flexible α,ω-bisamino(*n*-alkylene)phenyl terminated poly(ethylene glycol). The influences of the *n*-alkylene inserted in aminophenyl of flexible amino-terminated polythers (ATPE) on the mechanical properties, fractographs and curing kinetics of the ATPE-DGEBA cured products were studied. The results show that the insertion of *n*-alkylene group into the aminophenyl group of the ATPE, on one hand, can significantly increase the strain relaxation rate and decrease glass transition temperature of the ATPE-DGEBA cured products, resulting in slight decrease of the Young's modulus and tensile strength, and significant increase of the toughness and elongation of the ATPE-DGEBA cured products. On the other hand, it can remarkably enhance the reactivity of amine with epoxy, much accelerating the curing rate of the ATPE-DGEBA systems. The activation energy of DGEBA cured by BAPTPE, BAMPTPE and BAEPTPE was 53.1, 28.5 and 25.4 $kJ\cdot mol^{-1}$, respectively. The as-obtained ATPE-DGEBA cured products are homogeneous, transparent, and show excellent mechanical properties including tensile strength and toughness. Thus they are promising to have important applications in structure adhesives, casting bulk materials, functional coatings, cryogenic engineering, damping and sound absorbing materials.

Keywords: Epoxy resin; Amine-terminated polyether; Fractography; Curing kinetics; Mechanical properties.

INTRODUCTION

Cured products of epoxy resins generally have high mechanical strength, low creep and curing shrinkage, strong anticorrosion and good electrical properties, and are thus widely used in structural adhesives, composite materials, surface coatings and electrical laminates[1–4]. However, these materials also have a main shortcoming of low toughness due to their high crosslinking density and are therefore prone to fracture. During the past decades, many efforts have been made in an attempt to enhance the toughness of the epoxy resin thermosets. For example, macroscopic orientation of the liquid crystalline epoxy thermosets cured in a magnetic field can improve their tensile modulus, break strength and elongation at break[5]. However, one of the most successful methods to increase their toughness is to incorporate a second phase such as polyether-amide[6, 7], polysiloxane[8, 9], poly(ether ether ketone)[10, 11], polysulfone[12–14], epoxidized natural rubber[15], engineering thermoplastic[16–18], rigid particles[19–21], interpenetrating polymer networks (IPNs)[22, 23], branched and block copolymers[24–28] *etc*. into the epoxy resin phase. In this case, the toughness of the resultant composites depends not only on the concentration, size and distribution of the second component, but also on the interface properties

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[**] Corresponding author: Jian-guo Guan (官建国), E-mail: guanjg@whut.edu.cn

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and composition of each phase[29]. Consequently, the preparation process is somewhat complicated and difficult to repeat[30].

Although the epoxy resin toughened by flexible curing agents often exhibits the decreased tensile strength and heat stability, it still has obvious advantages when applied at low temperatures and for cryogenic engineering projects[31−33]. Moreover, epoxy resin thermosets cured by flexible curing agents can also significantly improve both fracture toughness and strength when the flexible curing agents are appropriately designed at a molecular level. For example, at moderate and low temperatures the epoxy resin thermosets based on diglycidyl ether of bisphenol A (DGEBA) and α,ω-bisaminophenyl terminated poly(ethylene glycol) (BAPTPE) exhibit excellent mechanical properties which strongly depend on poly(ethylene glycol) segment length ($\overline{M}_{\rm PE}$). At the same time, the incorporation of poly(ethylene glycol) segments with a long length brings about both a high activation energy for the curing reactions and a low concentration of reactive functional groups so that it is difficult to crosslink at room temperature[32].

This paper investigates the curing kinetics, mechanical properties and the fractographs of the thermosetting products of DGEBA cured by a series of flexible polyethers with different terminated amine groups. The results indicate that with increasing the chain length of the alkylene group inserted in the aminophenyl group of the amine-terminated polyethers (ATPE), the curing reactivity of DGEBA and ATPE can dramatically increase and the cured products exhibit improved mechanical properties at moderate and lower temperatures. The n-alkylene group inserted in the aminophenyl group can significantly improve the reactivity of the terminated amine and remarkably reduce the strain relaxation rates of the ATPE-DGEBA crosslinked networks. Consequently, a novel high performance thermosetting epoxy resin with a fast curing reactivity and excellent mechanical properties is successfully prepared based on DGEBA and α,ω-bisamino(n-alkylene)phenyl terminated poly(ethylene glycol).

EXPERIMENTAL

Materials

The epoxy resin studied here was liquid diglycidyl ether of bisphenol A (DGEBA) with an epoxide equivalent weight of 210−240 purchased from Yueyang Baling Pertrochemical Technology Ltd. The various amino terminated polyethers (ATPE) including α,ω-bisaminophenyl terminated poly(ethylene glycol) (BAPTPE), α,ω-bisaminomethylenephenyl terminated poly(ethylene glycol) (BAMPTPE) and α,ω-bisaminoethylenephenyl terminated poly(ethylene glycol) (BAEPTPE) were synthesized according to reference [34]. The chemical structures of ATPE are shown in Fig. 1.

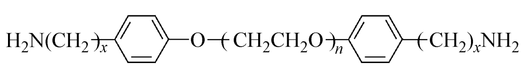

Fig. 1 Chemical structures of ATPE with various x of 0 (BAPTPE), 1 (BAMPTPE) and 2 (BAEPTPE)
The average of n is 8.68.

Preparation of Samples

The flexible ATPE was first mixed with DGEBA homogeneously according to stoichiometric ratio of epoxy and amino groups. Then the mixture was degassed with a vacuum pump to eliminate air bubbles. The bubble free mixtures were then poured into a preheated tetrafluoroethylene mold in an oven. The epoxy resin adhesives were cured at 100°C for 24 h to ensure a complete curing reaction.

Reactivity

The viscosity-time curves of the curing samples were examined by an advanced rheometric expansion system (ARES-RFS, TA) using parallel plates at isothermal curing temperature (T). Radius of the plates was 15 mm. The gap between the plates was 1.0 mm. The gelation time ($t_{\rm gel}$) is defined as the time which corresponds to the maximum value of viscosity change with time. The apparent activation energy ($E_{\rm a}$) for the curing reaction can be calculated from the slop of a $\ln t_{\rm gel}$ versus $1/T$ plot[35]:

$$\ln t_{\mathrm{gel}} = A + \frac{E_{\mathrm{a}}}{RT} \tag{1}$$

where A is a constant, R is the molar gas constant.

Tensile Test

The testing samples were prepared according to GB/T528-1998. The tensile properties were measured on a CMT-5105 SANS mechanical tester under a 5 kN load cell with a crosshead speed of 10 mm/min at 25°C and 0°C. The tensile toughness is measured by calculating the area under the stress-strain curve. The quoted data is the average value of 5 samples.

Impact Properties

The charpy test was carried out according to GB/T528-1998 using an impact tester (Chengde, Model XCJ-40). The impact test was carried out at 25°C and 0°C. The quoted data is the average value of 5 samples.

Dynamic Mechanical Thermal Analysis (DMTA)

The shear storage modulus (G') and shear loss modulus (G'') of the fully cured samples (50 mm × 10 mm × 1 mm) were measured using ARES-RFS in air atmosphere, with a heating rate of 1.0 K/min and a frequency of 1 Hz. The heating temperature ranges from −40°C to 80°C. The glass transition temperature (T_g) was assigned as the value corresponding to the maximum G''.

Morphological Observation

The fracture surfaces of the impact samples were examined by scanning electron microscopy (SEM, Hitachi S-4800). The fracture surfaces were sputtered with a thin layer of platinum to improve the conductivity.

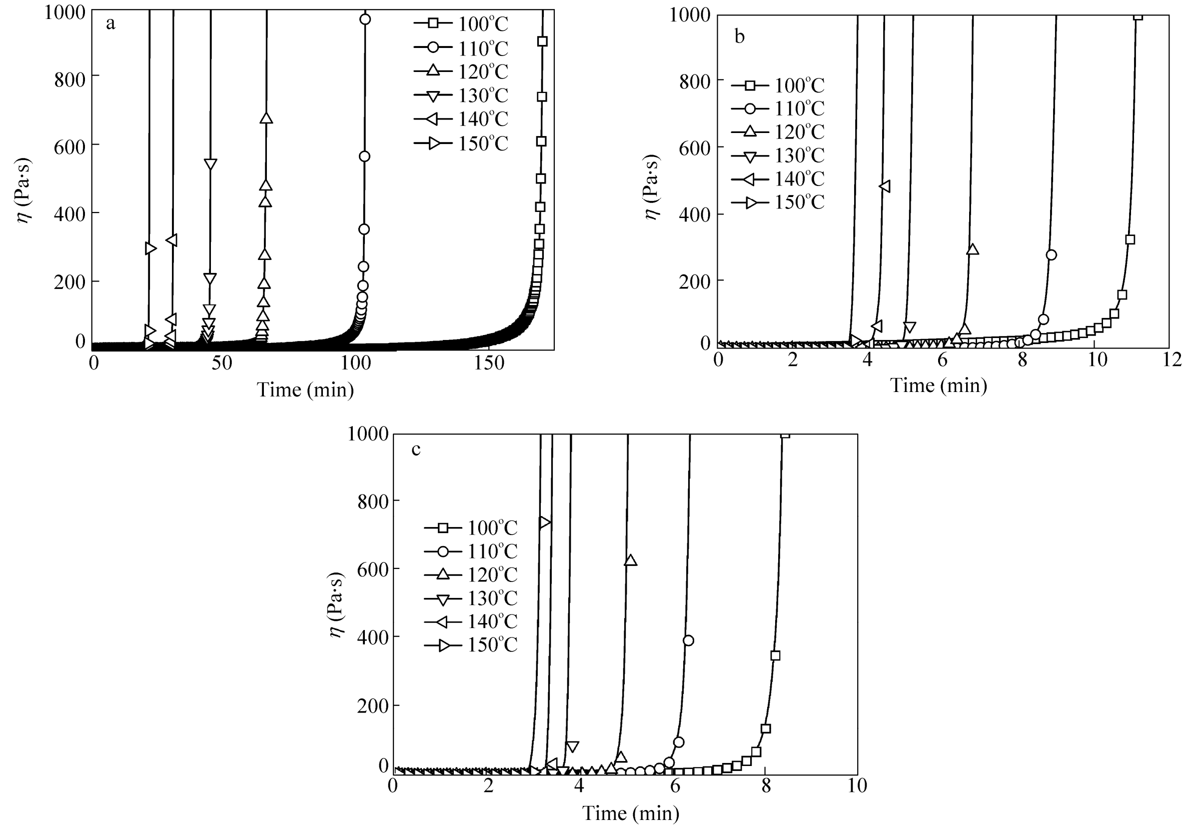

Fig. 2 Viscosity versus curing time curves for the thermosetting systems of DGEBA cured by (a) BAPTPE, (b) BAMPTPE and (c) BAEPTPE

RESULTS AND DISCUSSION

Curing Reactivity of the ATPE-DGEBA Systems

Figure 2 shows viscosity (η) versus curing time (t) curves for the ATPE-DGEBA curing systems at different isothermal cured temperatures. Obviously, with the extension of curing time, the η of the ATPE-DGEBA curing system increases at first slowly, then significantly after the gelation reaction occurs. The gelation time (t_{gel}) decreases with increasing the cured temperature (T). The comparison of (a), (b) and (c) also indicates that t_{gel} has a strong dependence on chemical structure of the ATPE. The t_{gel} of the ATPE-DGEBA curing system is dramatically shorten with increasing the chain length of the n-alkylene group inserted in the aminophenyl group of the ATPE (L_{alkyl}). The t_{gel} at 100°C for the thermosetting system of DGEBA cured by BAPTPE (in this case, $x = 0$) is more than 150 min while that by BAEPTPE ($x = 2$) is less than 10 min.

Figure 3 shows lnt_{gel} versus $1/T$ curves for the systems of DGEBA cured with different ATPE. It is clear that all the 3 curves between lnt_{gel} and $1/T$ have good linear relationships, and the linear correlation coefficients (R) all are larger than 0.98 (Table 1). The t_{gel} for the thermosetting systems based on epoxy resin cured by ATPE at various cured temperature can be easily obtained by extrapolating the regression lines. As shown in Table 1, t_{gel} for epoxy resin cured by BAPTPE, BAMPTPE and BAEPTPE at room temperature (298 K) are 11913 min, 105 min and 60 min, respectively. This suggests that inserting alkylene groups into the aminophenyl groups in the ATPE can obtain an epoxy resin thermosetting system which is crosslinkable at room temperature.

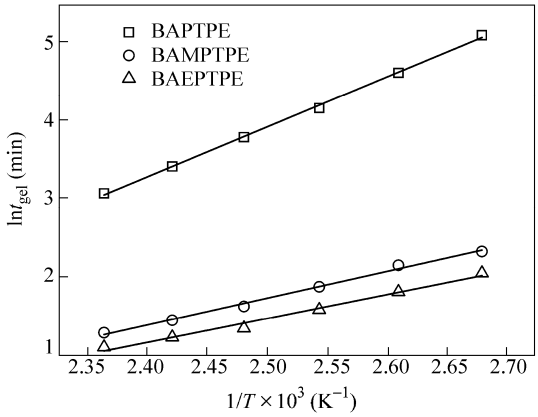

Fig. 3 lnt_{gel} versus $1/T$ curves for the systems of DGEBA cured with different ATPE

The symbols are the experimental date and the solid lines are the regression curves.

Table 1. Kinetic parameters for the ATPE-DGEBA thermosets

ATPE	E_a (kJ·mol^{-1})	t_{gel} at 298 K (min)	R
BAPTPE	53.1	11913	0.9987
BAMPTPE	28.5	105	0.9928
BAEPTPE	25.4	60	0.9844

The slope of the regression line for the ATPE-DGEBA curing system decreases with increasing L_{alkyl}. By applying the equation (1), the activation energy (E_a) can be calculated from the slopes. The E_a of DGEBA cured by BAPTPE, BAMPTPE and BAEPTPE are 53.1, 28.5 and 25.4 kJ·mol^{-1}, respectively. The fact that E_a decreases following the sequence of BAPTPE, BAMPTPE and BAEPTPE can be explained in terms of the chemical structure change of ATPE. On one hand, the lone pair electron orbit of nitrogen overlaps with the π system of the aromatic ring in BAPTPE whose amino groups directly attached aromatic rings, and the lone pair electrons form double bonds with the aromatic rings *via* resonance, possibly resulting in the formation of three zwitterionic resonance structures (Fig. 4)[36]. Thus the amines in BAPTPE are less active to accept protons. On the other hand, the alkylamine in BAMPTPE or BAEPTPE is a stronger base as its linked aromatic ring has an electron-donating effect on it, resulting in a stabilizing effect to accept proton. Thus, BAPTPE is a weaker base than BAMPTPE or BAEPTPE. As a consequence, BAPTPE is more difficultly reacted with DGEBA than BAMPTPE or BAEPTPE, and the former has higher activation energy than the latter.

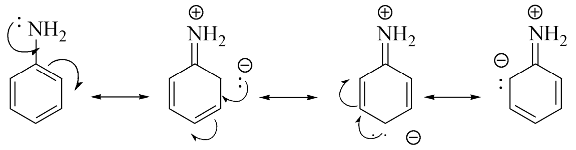

Fig. 4 Resonance interactions between nitrogen's lone pair and the aromatic ring[36]

Mechanical Properties of ATPE-DGEBA

Figure 5 represents the stress versus strain curves of the ATPE-DGEBA cured products. The detailed data of the mechanical properties are summarized in Table 2. Clearly, the BAPTPE-DGEBA cured product shows a typical strain-stress behavior of vitreous polymer at both 25°C and 0°C. It is noted that the BAMPTPE-DGEBA and BAEPTPE-DGEBA cured products at 25°C exhibit yield stress in the strain-stress curves, followed by extensive elongation at almost constant stress. The elongation at break of the BAMPTPE-DGEBA and BAEPTPE-DGEBA cured products could reach 170%. This can be reasonably explained in terms of the glass transition temperature (T_g). As shown in Fig. 7(b), the cured products of BAMPTPE-DGEBA and BAEPTPE-DGEBA both show a T_g of about 22°C, near room temperature. This suggests that they are both in the high-elastic state at room temperature. In this case, the crosslinked networks are facile to adjust the conformation *via* the transformation from a curled molecular chain into a stretched one by σ-σ bond rotation when they are subjected to an external force. As a result, they show a large elongation at break and strong impact toughness.

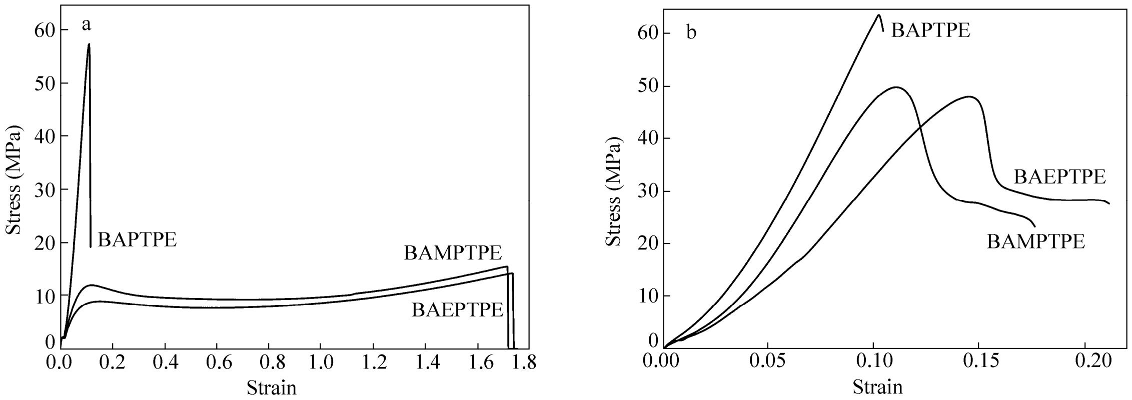

Fig. 5 Stress-strain curves for the ATPE-DGEBA cured products at (a) 25°C and (b) 0°C

Table. 2 Mechanical properties of the ATPE-DGEBA cured products

ATPE	Tensile strength (MPa)		Young's modulus (MPa)		Elongation at break (%)		Tensile toughness (kJ·m^{-2})		Impact strength (kJ·m^{-2})	
	25°C	0°C	25°C	0°C	25°C	0°C	25°C	0°C	25°C	0°C
BAPTPE	57.55	64.36	408	471	11.68	10.42	2956	2868	29.05	18.93
BAMPTPE	15.59	50.02	163	413	171.69	17.61	18485	4575	> 98	62.25
BAEPTPE	14.32	48.14	131	307	173.76	21.13	16182	5356	> 98	68.81

It can be seen from Table 2 that when L_{alkyl} increases for the ATPE-DGEBA epoxy resin thermosets, the tensile strength and Young's modulus decrease while the tensile toughness and impact strength significantly increase at both 25°C and 0°C. Generally, the decrease of the tensile strength and Young's modulus mainly comes from either the increasing volume fraction of the chain segments with low cohesive energy density or the increase of the distance between crosslinking points[32]. Figure 6 obviously shows that the insertion of methylene or ethylene group into the aminophenyl groups in the ATPE has only a slight effect on the volume fraction of methylene (from $x = 0$ of 36.11% to $x = 1$ of 37.78% and $x = 2$ of 39.36%) and the crosslinking density for the ATPE-DGEBA cured products. This suggests that the increasing volume fraction of methylene and the reducing crosslinking density can not explain well the remarkable depression of the tensile strength and Young's modulus at 25°C by the insertion of methylene or ethylene group into the aminophenyl groups in the ATPE for the ATPE-DGEBA cured products. This is quite different from that case of the increasing flexible poly(ethylene glycol) segment length[32]. However, in comparison of the BAPTPE-DGEBA cured products, the BAMPTPE-DGEBA and BAEPTPE-DGEBA cured products offer a σ-σ bond (methylene or ethylene) rather than a p-π bond (directly) to connect the epoxy resin hard segment with the rigid phenyl group. It is known that σ-σ bonds easily change the conformation *via* rotation while p-π bonds do not. As a result, the BAMPTPE-DGEBA and BAEPTPE-DGEBA cured products significantly increase the segment mobility, and thus reduce tensile strength and Young's modulus at 25°C. For the BAMPTPE-DGEBA and BAEPTPE-DGEBA, the tensile strength and Young's modulus at 25°C are much lower than those at 0°C, implying that the method to insert *n*-alkylene group into the aminophenyl groups in the ATPE is not fit to prepare high-temperature resins. This is also confirmed by T_g. As shown in Fig. 7(b), T_g for the ATPE-DGEBA thermosets obviously reduces when *n*-alkylene group is inserted into the aminophenyl groups in the ATPE.

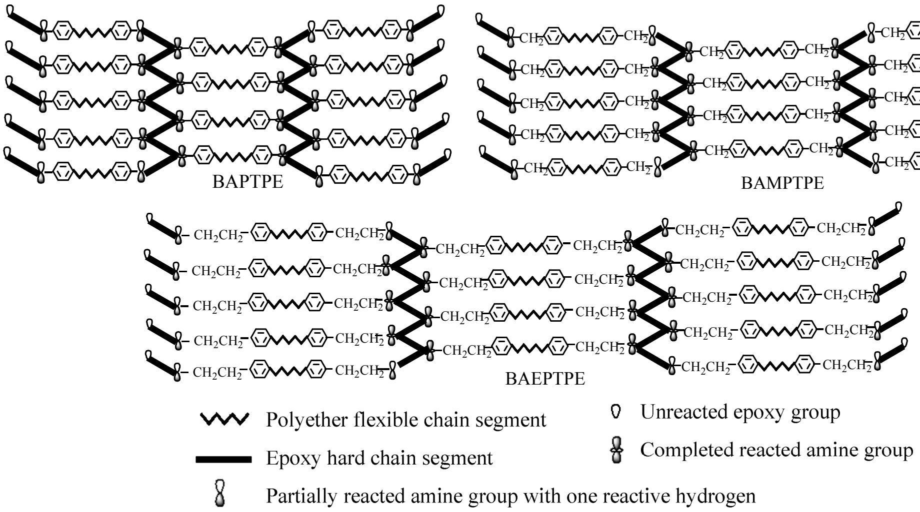

Fig. 6 Schematic network structures of the ATPE-DGEBA cured products

On the other hand, the BAMPTPE-DGEBA and BAEPTPE-DGEBA cured products show impact strength of more than 98 $kJ \cdot m^{-2}$ and can not be broken by the impact tester at 25°C. It is interesting to observe that compared with the BAPTPE-DGEBA cured products, the tensile toughness of the BAMPTPE-DGEBA and BAEPTPE-DGEBA cured products at 0°C only increases by 49% and 75%, respectively. However, the values of impact strength are 62.25 and 68.81 $kJ \cdot m^{-2}$, respectively. They are three times more than that of the BAPTPE-DGEBA (18.93 $kJ \cdot m^{-2}$). This indicates that the BAMPTPE-DGEBA and BAEPTPE-DGEBA cured products not only have excellent tensile toughness but also have strong impact strength. Due to the insertion of *n*-alkylene groups into aminophenyl groups in ATPE, the mobility of the epoxy resin hard segments in the ATPE-DGEBA cured products is almost independent on the rigid phenyl groups as they are connected by σ-σ bonds, which can freely rotate around the bond axis to change the conformation. Consequently, the chain segment length significantly increases and the relaxation time of the segment mobility decreases. Once being subjected to the external force, the crosslinked networks can response quickly *via* σ-σ bond rotation and thus rearrangement of molecular conformation, leading to the enhanced tensile toughness and impact resistance. In contrast, the epoxy resin hard segments and the rigid phenyl groups in the ATPE-DGEBA cured products are connected together by p-π bonds to form units, which exhibit slow relaxation rates. As a result, the response of the BAPTPE-DGEBA cured products to external forces shows a strong dependence on the strain rates. Thus the BAPTPE-DGEBA cured products have large tensile toughness but relatively low impact strength. The elongation at break, tensile toughness and impact strength of the ATPE-DGEBA thermosets at 0°C all are enhanced by far only at the expense of a little depression of the tensile strength and Young's modulus with the insertion of *n*-alkylene group into the aminophenyl groups in the ATPE, suggesting that it is an excellent approach to prepare high performance thermosetting resins working at moderate and low temperatures.

Dynamic Mechanical Thermal Analysis

In order to better understand the structure and properties of the ATPE-DGEBA cured products, the dynamic mechanical properties were measured. Figure 7 shows that with increasing temperature (T) the storage modulus (G') for the 3 kinds of ATPE-DGEBA cured products first decreases gradually, then significantly, and last slowly. And the loss modulus (G'') shows a peak, corresponding to T_g. Clearly, only a single T_g exists in all cured epoxy systems, indicating a homogeneous phase structure. As shown in Fig. 7(b), when the group of methylene or ethylene is inserted into the aminophenyl groups in the ATPE, T_g for the ATPE-DGEBA cured products decreases from 38°C to 22°C and 21°C. This further confirms that the insertion of *n*-alkylene group into the aminophenyl groups of the ATPE indeed increases the strain relaxation rate and can adjust T_g of the ATPE-DGEBA cured products. Although the reduction of T_g is a disadvantage when the ATPE-DGEBA systems are applied at high-temperature, they still have potential applications in cryogenic engineering because of their excellent mechanical performance at low temperature.

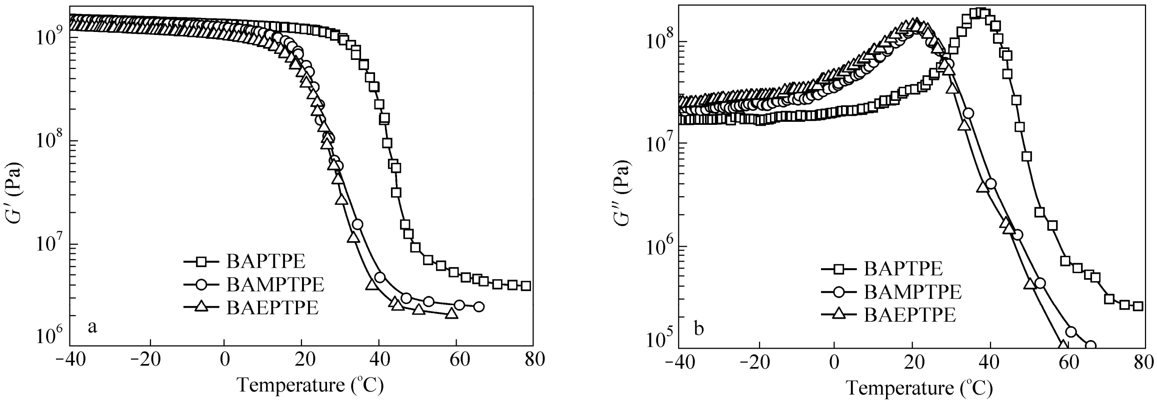

Fig. 7 Dynamic mechanical thermal analysis curves for the ATPE-DGEBA cured products

It can also be seen from Fig. 7 that the BAPTPE-DGEBA cured product shows the highest G' and the lowest G'' among the 3 kinds of ATPE-DGEBA cured products in the transition region, suggesting the worst ability to absorb vibration energy. The maximal modulus loss factor ($\tan\delta_{max}$) for the BAMPTPE-DGEBA and BAEPTPE-DGEBA cured products can be easily calculated to be more than 1.0, hinting that the products have good damping characteristics and are promising to apply in the field of anti-vibration and sound absorbing. This can be well explained by the insertion of the *n*-alkylene group into the aminophenyl group, which significantly improves the mobility of the epoxy resin hard segments and rigid phenyl segments in the ATPE-DGEBA cured products.

Impact Fracture Surface Morphology

Representative SEM micrographs of fractured surfaces of ATPE-DGEBA cured products after Charpy impact testing are shown in Fig. 8. At 25°C, the fracture surface of the BAPTPE-DGEBA cured product is relatively rough. Figure 8(a) clearly indicates the tortuous cracks and ridges in the fracture surface. The rough fracture surface suggests the deflection of crack path, *i.e.* the crack deviation from its original plane, increasing the area of the crack. Hence, the energy required for the propagation of the cracks in the fracture surfaces increases. Figure 8(b) shows that the fracture surface of the BAPTPE-DGEBA cured product at 0°C is relatively smooth, showing brittle fracture surface mode. Nonetheless, as shown in Figs. 8(c) and 8(d), ridges appear in the fracture surfaces of the BAMPTPE-DGEBA and BAEPTPE-DGEBA cured products, showing typical characteristics of toughness fracture[31]. This further confirms that the insertion of *n*-alkylene in the aminophenyl groups of the ATPE can significantly increase the fracture surface area and improve the impact strength of the ATPE-DGEBA cured products.

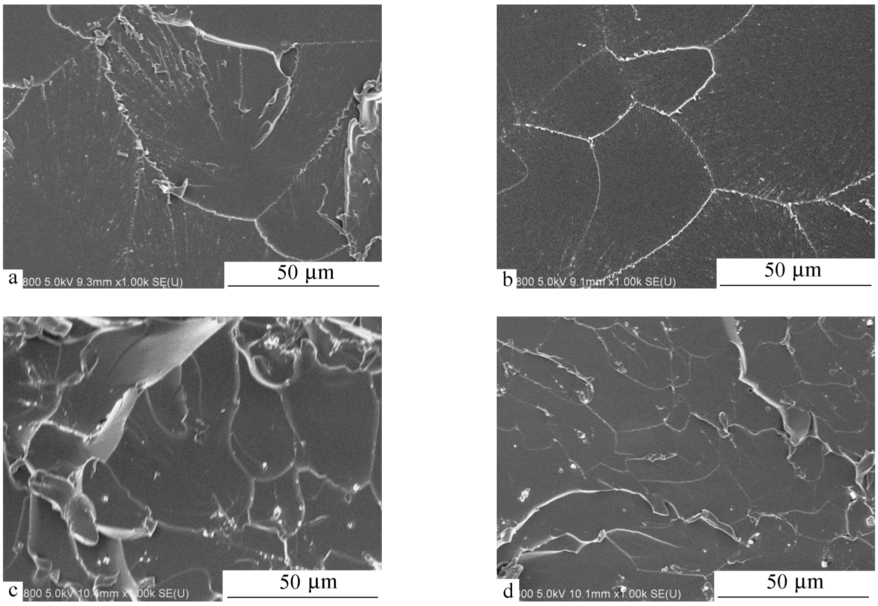

Fig. 8 SEM images of the fractured surfaces of ATPE-DGEBA cured products
a) BAPTPE at 25°C; b) BAPTPE at 0°C; c) BAMPTPE at 0°C; d) BAEPTPE at 0°C

Further, the morphology also demonstrates that the ATPE-DGEBA cured products are homogeneous without signs of phase separation, suggesting that homogeneous crosslinked network structures are formed in the curing process of the ATPE-DGEBA thermosets. These results are in accordance with dynamic mechanical analysis as discussed above.

CONCLUSIONS

In this paper, thermosetting epoxy resins with a fast curing reaction rate at room temperature and high toughness are obtained by reacting flexible α,ω-bisamino(*n*-alkylene)phenyl terminated poly(ethylene glycol) with DGEBA. The curing kinetics including t_{gel}, E_a of the ATPE-DGEBA curing systems and the tensile properties, dynamic mechanical properties, and fracture surface morphologies of the cured products were investigated as functions of the chain length of the *n*-alkylene group inserted in the aminophenyl groups in the ATPE. The results show that t_{gel} for the ATPE-DGEBA thermosetting systems is remarkably shortened due to the *n*-alkylene group inserted in the aminophenyl groups of the ATPE, which makes its linked aromatic ring have an electron-donating effect on the active amine and enhance the reactivity of amine with epoxy groups. The E_a of DGEBA cured by BAPTPE, BAMPTPE and BAEPTPE is 53.1, 28.5 and 25.4 $\mathrm{kJ\cdot mol^{-1}}$, respectively. The insertion of *n*-alkylene group into the aminophenyl group of the ATPE can significantly increase the strain relaxation rate and adjust T_g of the ATPE-DGEBA cured products, resulting in slight decrease of the Young's modulus and tensile strength, and significant increase of the toughness and elongation of the ATPE-DGEBA cured products. The as-obtained ATPE-DGEBA cured products have important potential applications in structure adhesives, casting bulk materials, electronic or magnetic coatings, cryogenic engineering, damping and sound absorbing materials as they are transparent and have excellent mechanical properties including tensile strength and toughness, and can be simply fabricated using non-toxic, non-volatile precursors.

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