

# Synthesis and Characterization of a Novel Silicon-Containing Polytriazole Resin

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**ABSTRACT:** 4,4'-Diazidomethylbiphenyl (DAMBP) and poly(dimethylsilylene-ethynylene)phenyleneethynylene (PDMSEPE) were thermally polymerized to form a novel silicon-containing polytriazole resin (PDMSEPE-DAMBP) by 1,3-dipolar cycloaddition. Differential scanning calorimetry, FTIR, and  $^{13}\text{C}$ -NMR were used to characterize the curing behaviors of PDMSEPE-DAMBP resins. The results indicated that the resins could cure at temperatures as low as 80°C. Dynamic mechanical analysis showed that there was a glass

transition at 302°C for the cured PDMSEPE-DAMBP resin. The carbon fiber (T700) reinforced PDMSEPE-DAMBP composites exhibited excellent mechanical properties at room temperature and high property retention at 250°C. © 2009 Wiley Periodicals, Inc. *J Appl Polym Sci* 114: 1725–1730, 2009

**Key words:** silicon-containing polytriazole resin; high-performance resin; arylacetylene resin; 1,3-dipolar cycloaddition; advanced composites

## INTRODUCTION

The Huisgen's [2+3] 1,3-dipolar cycloaddition of azides and alkynes offers a simple method achieving low temperature polymerization.<sup>1</sup> In the 1960s, this [2+3] cycloaddition reaction was utilized to produce linear poly(triazole)s via cycloaddition homopolymerization of AB-type monomers containing both alkyne and azide moieties.<sup>2</sup> Recently, some research works have focused on the synthesis of block copolymers,<sup>3</sup> graft copolymers,<sup>4</sup> dendrimers,<sup>5–9</sup> dendronized linear polymers,<sup>6</sup> star polymers,<sup>10</sup> and hyperbranched polymers<sup>11</sup> by 1,3-dipolar cycloaddition of azide and alkyne groups of monomers, polymers, and oligomers. 1,3-Dipolar cycloaddition of azides and alkynes is found to be a simple, reliable, and efficient approach to develop novel resins for low-temperature molding composites that were found in a wide of applications where accuracy of size and material cost were considered important.

Our laboratory has recently developed some novel thermosetting polytriazole (PTA) resins prepared from multipropargyl compounds and multi azide compounds by 1,3-dipolar cycloaddition.<sup>12,13</sup>

The silicon-containing arylacetylene polymers composed of  $[-\text{SiR}_2-\text{C}\equiv\text{C}-\text{Ar}-\text{C}\equiv\text{C}-]$  (R = alkyl or phenyl) units exhibit excellent thermal stabilities after curing.<sup>14–17</sup> There is high content of the ethynylene group in the backbone of the polymers. Low temperature curing can be realized by 1,3-dipolar cycloaddition between ethynyl or ethynylene groups of the silicon-containing arylacetylene polymers and azide groups of azide compounds. In this article, a novel thermosetting silicon-containing PTA resin (PDMSEPE-DAMBP resin) was prepared from poly(dimethylsilylene ethynylene)phenyleneethynylene (PDMSEPE) and 4,4'-diazidomethylbiphenyl (DAMBP). The structure and properties of PDMSEPE-DAMBP resin were studied.

## MATERIALS AND METHODS

### Materials

Tetrahydrofuran (THF) was used as purchased from Shanghai No. 1 Reagent Factory (China). The reinforcement material was carbon fiber (T700-12K) from Toray (Japan). DAMBP was synthesized in our laboratory.<sup>12,13</sup> PDMSEPE was synthesized in our lab according to literature.<sup>15,16</sup>

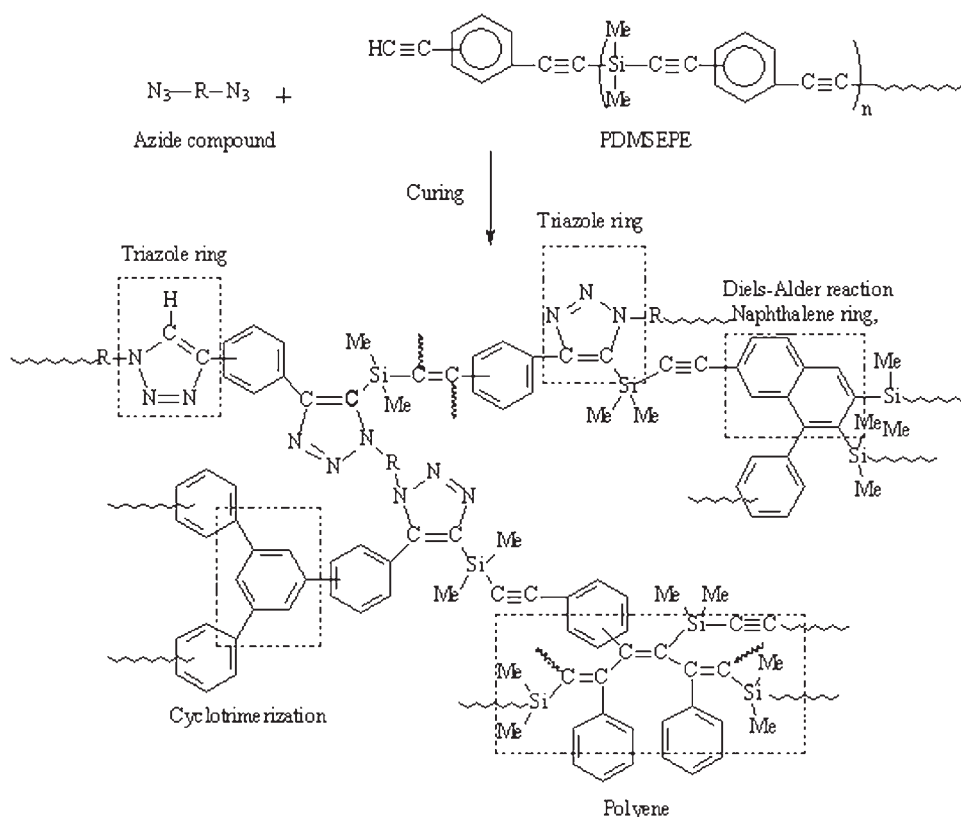
### Analysis of DAMBP

$^1\text{H}$ -NMR ( $\text{CDCl}_3$ , TMS): 4.40 (s,  $\text{CH}_2$ ), 7.41 (d, Ar-H), 7.61 (d, Ar-H);  $^{13}\text{C}$ -NMR ( $\text{CDCl}_3$ , TMS): 54.83 ( $-\text{CH}_2-\text{N}_3$ ); 128.55, 130.64, 136.55, 140.94 ( $-\text{C}_6\text{H}_4-$ ).

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**Scheme 1** Preparation and polymerization of PDMSEPE-DAMBP.

### Analysis of PDMSEPE

FTIR:  $3300\text{ cm}^{-1}$  ( $\equiv\text{C-H}$ ),  $2156\text{ cm}^{-1}$  ( $\text{C}\equiv\text{C}$ );  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ , TMS): 3.05 ( $\equiv\text{CH}$ ), 0.37 ( $\text{Si-CH}_3$ ), 7.2–7.7 ( $\text{Ar-H}$ ).  $^{29}\text{Si-NMR}$  ( $\text{CDCl}_3$ , TMS):  $-39.0$  ( $\text{C}\equiv\text{C-Si}$ );  $^{13}\text{C-NMR}$  ( $\text{CDCl}_3$ , TMS): 0.57 ( $-\text{Si-CH}_3$ ); 78.19, 82.12 ( $-\text{C}\equiv\text{CH}$ ); 92.60, 105.01 ( $-\text{C}\equiv\text{C}-$ ); 123.17, 128.53, 132.58, 135.89 ( $-\text{C}_6\text{H}_4-$ ).

### Preparation and cure of a novel silicon-containing PTA resin

PDMSEPE (7.60 g) and 4.70 g DAMBP were charged into a 100 mL three-neck flask, stirred, and heated to  $80^\circ\text{C}$ . After the mixture in the flask became homogeneous at  $80^\circ\text{C}$ , a silicon-containing PTA resin was obtained and designated as PDMSEPE-DAMBP resin. Then the melt resin was poured into a mold for the preparation of the cured sample. The resin was cured continuously in an oven under air at  $80^\circ\text{C}$  for 12 h,  $120^\circ\text{C}$  for 2 h,  $160^\circ\text{C}$  for 2 h,  $180^\circ\text{C}$  for 2 h,  $210^\circ\text{C}$  for 2 h, and  $250^\circ\text{C}$  for 2 h. The polymerization of the PDMSEPE-DAMBP resin and speculative structures are shown in Scheme 1.

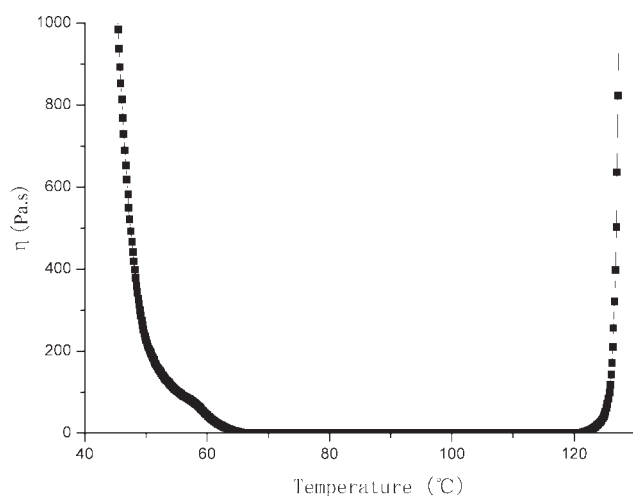
### Preparation of composites

A unidirectional prepreg was prepared from carbon fiber (T700) and solution containing a 40 wt % of

PDMSEPE-DAMBP in THF by using a drum-winding technique. The fibers were passed through the solution of the resin, impregnated, and then wound on the mandrel. The prepreg was dried on the mandrel to strip off the most of solvent. After the removal of the prepreg from the drum, the resultant prepreg had good tack and drape. The prepreg was laid unidirectionally, plied stack, and further dried in a vacuum oven. The prepreg stack was pressed and cured at  $80^\circ\text{C}$  for 12 h under the pressure of 3 MPa and then postcured at  $120^\circ\text{C}$  for 2 h,  $160^\circ\text{C}$  for 2 h,  $180^\circ\text{C}$  for 2 h,  $210^\circ\text{C}$  for 2 h, and  $250^\circ\text{C}$  for 2 h successively under the pressure of 3 MPa or free pressure. The composite with a thickness of ca. 2.0 mm was obtained. The composite sample for the flexural test was made with the dimension of  $80 \times 15 \times 2\text{ mm}^3$ .

### Characterization

Carbon magnetic resonance ( $^{13}\text{C-NMR}$ ),  $^1\text{H-NMR}$ , and  $^{29}\text{Si-NMR}$  spectra were obtained in deuteriochloroform solvent with aid of the Bruker Avance 500 (500 MHz) spectrometer and tetramethylsilane (TMS) was used as an internal standard. The solid-state cross polarization/magic-angle spinning (CP/MAS)  $^{13}\text{C-NMR}$  spectra were recorded on the Bruker DSX-300 at room temperature. The chemical shifts



**Figure 1** Viscosity–temperature curve of PDMSEPE-DAMBP resin.

were calculated with respect to TMS. Differential scanning calorimeter (DSC) analyses were performed on a Universal V2.3 TA Instruments 2910 modulated system at the heating rate of 10°C/min. Dynamic mechanical analysis (DMA) was carried out on a Rheogel-E4000 analyzer operating in the tension mode from 30 to 400°C at the frequency of 11 Hz with the programmed heating rate of 3°C/min. FTIR spectra were obtained on the Nicolet 550 spectrometer and solid samples were pressed into pellets with KBr. Rheological behavior of the resin was traced on a RheoStress RS600 Rheometer at the heating rate of 2.0°C/min and the shear rate of 0.01 s<sup>-1</sup>. Thermogravimetric analysis (TGA) was conducted on a Mettler TGA/SDTA 851 analyzer under nitrogen at the heating rate of 10°C/min. The flexural properties of composite were measured with a Shimadzu AG-50kNE universal tester and the crosshead speed of the flexural test was 2 mm/min according to China Standard GB 3356. The sample dimension was 80 × 15 × 2 mm. The five samples were chosen and tested.

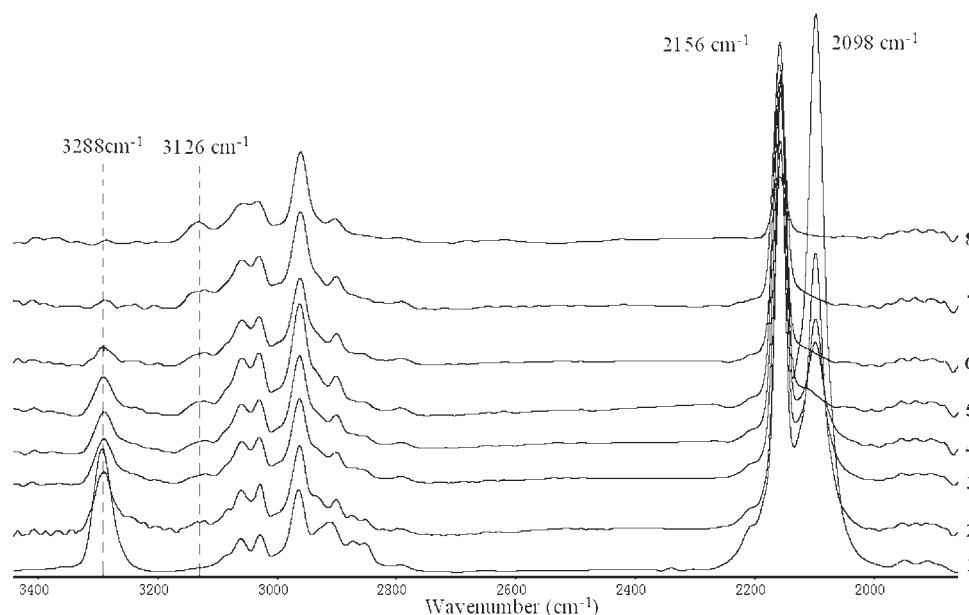
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## RESULTS AND DISCUSSION

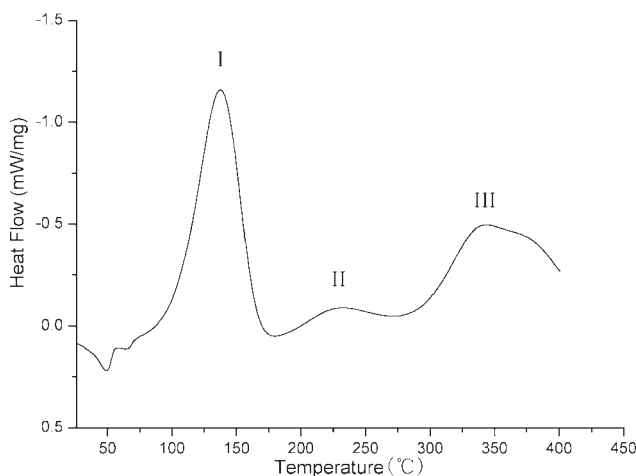
### Thermal behaviors of PDMSEPE-DAMBP resin

The viscosity response of PDMSEPE-DAMBP resin to the temperature ramping at heating rate of 2°C/min was shown in Figure 1. The viscosity of PDMSEPE-DAMBP resin decreased dramatically with the increase in the temperature from 50 to 65°C. At temperatures above 65°C, the viscosity of the resin was below 1.0 Pa.s and maintained the low viscosity up to 120°C, which was expected for the resin transfer molding process. Thereafter resin viscosity increased rapidly at temperatures above 120°C due to the further polymerization of PDMSEPE-DAMBP resin.

FTIR was employed to determine the curing reactions of PDMSEPE-DAMBP. The FTIR spectra of the resin at different curing stages were shown in Figure 2. The characteristic peak at 3288 cm<sup>-1</sup> was the absorption of C—H stretching vibrations (—Ph—C≡C—H) and the peak at 2098 and 2156 cm<sup>-1</sup> were



**Figure 2** FTIR spectra of PDMSEPE-DAMBP resin at different curing stages. (1) At the beginning; (2) 80°C/3 h; (3) 80°C/12 h; (4) 80°C/12 h + 120°C/2 h; (5) 80°C/12 h + 120°C/2 h + 160°C/2 h; (6) 80°C/12 h + 120°C/2 h + 160°C/2 h + 180°C/2 h; (7) 80°C/12 h + 120°C/2 h + 160°C/2 h + 180°C/2 h + 210°C/2 h; (8) 80°C/12 h + 120°C/2 h + 160°C/2 h + 180°C/2 h + 210°C/2 h + 250°C/2 h.



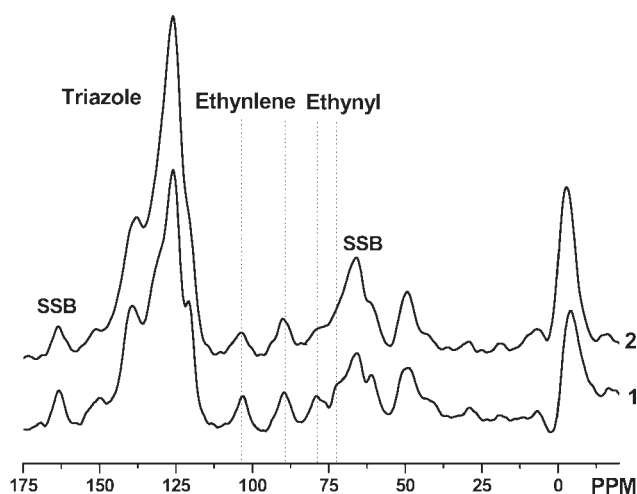
**Figure 3** DSC curve of PDMSEPE-DAMBP resin.

attributed to the absorption of azides ( $-\text{N}=\text{N}=\text{N}$ ) and stretching vibrations of ethynylene ( $-\text{Ph}-\text{C}\equiv\text{C}-\text{Si}-$ ), respectively. The intensity of three absorption peaks at 3288, 2098, and 2156  $\text{cm}^{-1}$  decreased gradually during the curing process of PDMSEPE-DAMBP resin, while a new absorption peak at 3126  $\text{cm}^{-1}$ , which was attributed to C—H of triazole ring formed during the polymerization, occurred. After the resin was held at 80°C for 12 h and then postcured at 120°C for 2 h and 160°C for 2 h, the absorption peaks at 2098  $\text{cm}^{-1}$  corresponding to azides ( $-\text{N}=\text{N}=\text{N}$ ) disappeared. However, the peaks at 3288 and 2156  $\text{cm}^{-1}$  due to residual ethynyl and ethynylene ( $-\text{Ph}-\text{C}\equiv\text{C}-\text{H}$  and  $-\text{Ph}-\text{C}\equiv\text{C}-\text{Si}-$ ) groups were still observed. This indicated that the 1,3-dipolar cycloaddition reaction of ethynyl and ethynylene with azide was complete at this stage. After the resin has cured at 80°C for 12 h and then postcured at 120°C for 2 h, 160°C for 2 h, 180°C for 2 h and 210°C for 2 h, the peak at 3288  $\text{cm}^{-1}$  due to C—H stretching vibrations ( $-\text{Si}-\text{Ph}-\text{C}\equiv\text{C}-\text{H}$ ) almost disappeared. This demonstrated that thermal reaction of the ethynyl groups approached to be finished. In addition, after the resin cured through the curing procedure at 80°C/12 h + 120°C/2 h + 160°C/2 h + 180°C/2 h + 210°C/2 h + 250°C/2 h, the peak at 2156  $\text{cm}^{-1}$  still presented. This meant that ethynylene groups existed at this stage.

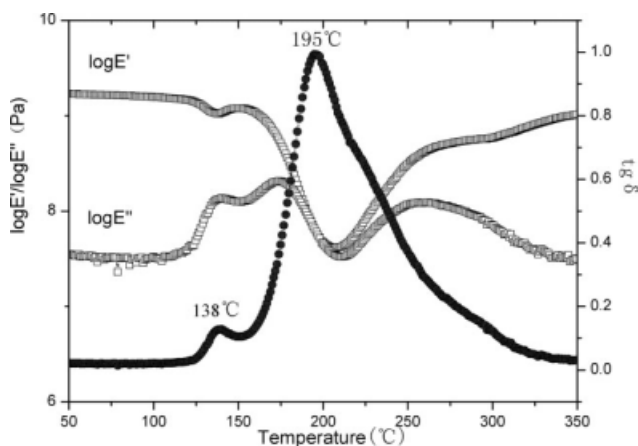
DSC technique was used to trace the thermal behaviors of PDMSEPE-DAMBP resin and the DSC curve for the resin was given in Figure 3. The figure showed three exothermic peaks. The amount of exotherm of the first exothermic peak (I) with onset at 75°C and top at 138°C was 597.4 J/g. The following wide exothermic peak (II) occurred in the range from 175 to 275°C. The third exothermic peak (III) appeared at above 300°C. Combined with the FTIR analysis results, the first exothermal peak was attrib-

uted to 1,3-dipolar cycloaddition between azide groups and ethynyl or ethynylene groups to form the polymer with 1,4-disubstituted and 1,5-disubstituted 1,2,3-triazoles. The second peak related to the polymerization of ethynyl or ethynylene to form polyene, benzene ring, and naphthalene ring,<sup>18</sup> and the third peak attributed to polymerization of ethynylene, as well as decomposition of cured resin successively, as shown in Scheme 1.

Figure 4 shows the  $^{13}\text{C}$  CP/MAS spectra of the PDMSEPE-DAMBP samples cured at 80°C for 12 h or then postcured at 250°C. The peaks at 74.18 and 79.10 ppm were attributed to the ethynyl group and 90.19, 103.08 ppm were assigned to the ethynylene group. The peak at 49.46 ppm corresponded to the methylene group. When PDMSEPE-DAMBP was cured at 80°C, a small resonance centered at 150.1 ppm was observed in Figure 4, which was assigned to C=C in the triazole ring. This phenomenon indicated 1,3-dipolar cycloaddition between azide groups and ethynyl or ethynylene groups took place to form 1,4-disubstituted and 1,5-disubstituted 1,2,3-triazoles when PDMSEPE-DAMBP was cured at 80°C for 12 h. After curing at 80°C for 12 h, 120°C for 2 h, 160°C for 2 h, 180°C for 2 h, 210°C for 2 h, and 250°C for 2 h, the signals at 74.18 and 79.10 ppm for ethynyl vanished. The signals in the range from 145 to 158 ppm broadened with the increase in postcuring temperature and this peak can be assigned to the C=C carbons due to the polymerization of ethynyl or ethynylene. The  $^{13}\text{C}$  CP/MAS NMR analyses further confirmed the reactions at different curing stages.



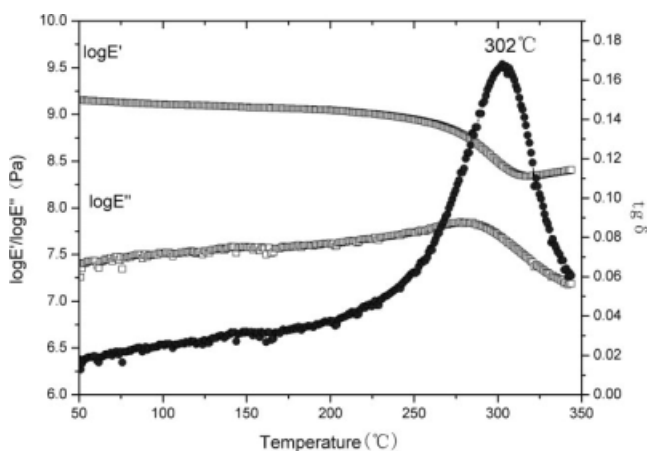
**Figure 4**  $^{13}\text{C}$  CP/MAS NMR spectra of PDMSEPE-DAMBP. (1) PDMSEPE-DAMBP resin cured at 80°C for 12 h; (2) PDMSEPE-DAMBP resin cured at 80°C for 12 h, 120°C for 2 h, 160°C for 2 h, 180°C for 2 h, 210°C for 2 h, and 250°C for 2 h. SSB, spinning side band.



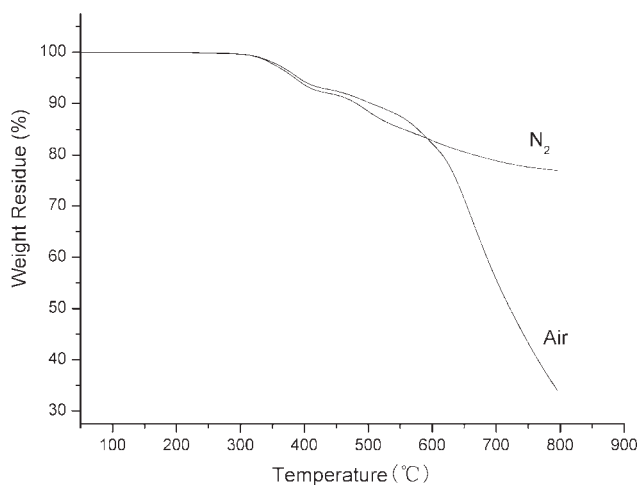
**Figure 5** DMA curves of PDMSEPE-DAMBP resin cured at 80°C for 12 h.

### Thermal properties of the cured PDMSEPE-DAMBP resin

Glass transition temperature of the cured PDMSEPE-DAMBP resin was detected by DMA. Generally, the damping peak of  $T_g\delta$  is identified as the glass transition temperature in DMA analysis curves because a large decrease in modulus occurs at this point. As shown in Figure 5, PDMSEPE-DAMBP resin cured at 80°C for 12 h presented two transitions in the range from 100 to 250°C. A small damping peak in  $T_g\delta$ - $T$  curve and minor drop of storage modulus ( $E'$ ) in  $\log E'$ - $T$  curve could be observed in the range from 125 to 150°C, and the cured resin exhibited well-defined dynamical damping peaks centered at 138°C. With temperature increasing slowly during the experiment, the storage modulus ( $E'$ ) was recovered from the drop due to crosslinking of the PDMSEPE-DAMBP resin and another high glass transition occurred at 195°C.  $T_g$  of the cured resin



**Figure 6** DMA curves of PDMSEPE-DAMBP resin cured at 80°C for 12 h, 120°C for 2 h, 160°C for 2 h, 180°C for 2 h, 210°C for 2 h, and 250°C for 2 h.



**Figure 7** TGA curve of cured PDMSEPE-DAMBP resin.

increased with the increment of the postcuring temperature, and it reached 302°C when postcured at 250°C for 2 h, as shown in Figure 6.

The thermal stability and thermo-oxidative stability of the cured PDMSEPE-DAMBP resin were determined by analyses of TGA in  $N_2$  and air. As shown in Figure 7, the thermal decomposition temperature of the cured resin at 5% weight loss ( $T_{d5}$ ) arrived at 385°C and the char yield (wt %) was 76.9% at 800°C in  $N_2$ . This showed good thermal stability of the cured resin. The TGA analyses (in air) of the cured PDMSEPE-DAMBP resin was shown in Figure 7 and the results were tabulated in Table I. The thermal oxidative decomposition temperature of the cured resin at 5% weight loss ( $T_{d5}$ ) arrived at 391°C and the char yield (wt %) was 33.9% at 800°C in air. The thermal decomposition temperature in  $N_2$  was close to the thermal oxidative decomposition temperature. This indicated the resin possessed good thermal oxidative stability. As compared with PDMSEPE resin

**TABLE I**  
Mechanical Properties of Carbon Fiber (T700)-Reinforced PDMSEPE-DAMBP Resin Composites

Processing condition	Testing temperature (°C)	Flexural strengths (MPa)	Flexural modulus (GPa)
(1)	RT	1830 ± 80.2	119 ± 2.4
(2)	RT	1833 ± 76.5	126 ± 1.4
(3)	RT	1861 ± 70.7	117 ± 2.8
(1)	200	1476 ± 19.1 (81%)	125 ± 1.8
(1)	250	1278 ± 17.5 (70%)	126 ± 1.6

Processing condition: (1) at 80°C for 12 h under pressure of 3 MPa; (2) at 80°C for 12 h under pressure of 3 MPa and then postcured at 120°C for 2 h, at 170°C for 2 h, at 210°C for 2 h and at 250°C for 2 h under pressure of 3 MPa; (3) at 80°C for 12 h under pressure of 3 MPa and then postcured at 120°C for 2 h, at 170°C for 2 h, at 210°C for 2 h, and at 250°C for 2 h under free pressure.

( $T_{d5}$  631°C), the  $T_{d5}$  of cured PDMSEPE-DAMBP was 385°C and decreased greatly in  $N_2$ . The main reason for this phenomenon was due to the presence of weak C—N bonds. Due to the instability of C—N bond under high temperatures, thermal decomposition of C—N bonds in cured resin take place easily.<sup>19</sup>

### Properties of carbon fiber-reinforced PDMSEPE-DAMBP composites

The PDMSEPE-DAMBP resin was used as a matrix for carbon fiber reinforced composites. A unidirectional prepreg was pressed at 80°C for 12 h under pressure of 3 MPa and then postcured under the pressure of 3 MPa or at free-pressure conditions. As shown in Table I, the PDMSEPE-DAMBP resin composites possessed excellent mechanical properties after the composite was cured at 80°C for 12 h and the influences of postcure conditions on mechanical properties of the composites were negligible. Thereby, the PDMSEPE-DAMBP resin was suitable for low-temperature molding composites. The composites also exhibited high flexural properties at high temperatures. More remarkable was that the retention of the flexural strength at 200 and 250°C reached 81% and 70%, respectively. Therefore, the PDMSEPE-DAMBP resin would be expected to be used as a matrix for advanced composites.

### CONCLUSIONS

A novel silicon-containing PTA resin (PDMSEPE-DAMBP resin) was prepared from DAMBP and PDMSEPE by 1,3-dipolar cycloaddition polymerization. The PDMSEPE-DAMBP resin underwent multi-stage cure process on FTIR, DSC, and <sup>13</sup>C-NMR. The curing of PDMSEPE-DAMBP resin went through three stages with increases in temperature. The cycloaddition of ethynyl and ethynylene with azides took place first at the temperature in the range of 65–175°C, then the polymerization of ethynyl and ethynylene occurred at temperature in range of 175–250°C, and finally the polymerization of ethynylene appeared at the temperature above 250°C. The cured

PDMSEPE-DAMBP resin exhibited good thermal properties as evidenced by TGA and DMA results. The glass transition temperature of the cured resin reached 302°C and thermal decomposition temperature ( $T_{d5}$ ) was 385°C and 391°C in  $N_2$  and air, respectively. The carbon fiber (T700) reinforced composites showed flexural strength arrived at 1833 MPa at room temperature and a property retention at 1278 MPa at 250°C, suggesting the PDMSEPE-DAMBP resin could be expected to be used as a matrix of low-temperature molding composites.

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