

Preparation and Properties of High-Performance Polysilsesquioxanes/Bismaleimide-Triazine Hybrids

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ABSTRACT: A novel kind of high-performance hybrids (coded as POSS-NH₂/BT) with significantly decreased curing temperature, lowered dielectric constant and loss, and improved thermal resistance were developed, which were prepared by copolymerizing bismaleimide with cage octa(aminopropylsilsesquioxane) (POSS-NH₂) to produce POSS-containing maleimide, and then co-reacted with 2,2'-bis(4-cyanatophenyl) isopropylidene. The curing behavior and typical properties of cured POSS-NH₂/BT were systematically investigated. Results show that POSS-NH₂/BT hybrids have lower curing temperatures than BT resin because of the additional reactions between —OCN and amine groups. Compared with BT resin, all hybrids show improved dielectric properties. Specifically, hybrids have slightly decreased dielectric constants and similar depend-

ence of dielectric constant on frequency over the whole frequency from 10 to 10⁶ Hz; more interestingly, the dielectric loss of hybrids is only 25% of that of BT resin at the frequency lower than 10⁵ Hz, whereas all hybrids and BT resin have almost equal dielectric loss when the frequency is higher than 10⁵ Hz. In addition, POSS-NH₂/BT hybrids also show good thermal and thermo-oxidative stability compared with BT resin. All these differences in macroproperties are attributed to the difference in chemical structure between POSS-NH₂/BT hybrids and BT resin. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 120: 360–367, 2011

Key words: hybrids; octa(aminopropylsilsesquioxane); bismaleimide; cyanate ester

INTRODUCTION

The rapid evolution of electronic circuitry have brought increasing requirement of producing smaller, more powerful, and faster multilayer units, leading to increasing demands on dielectric materials with lower dielectric constant and loss and better thermal resistance to obtain high reliability.

Bismaleimide-triazine (BT) resins, derived from the blends of dicyanate ester and bismaleimide resins, claim superiority over the component homopolymers in terms of physical and mechanical characteristics. BT resins generally have advantages of low dielectric constant and loss, good thermal and dimensional stability, excellent chemical resistance, and good retention of mechanical properties at elevated temperature, especially in hot/wet environments.^{1–3} However, BT

resins need to be cured at very high temperature; in addition, their dielectric loss needs to be improved to meet the harsh requirements of increasingly developing electric industry.⁴ High curing temperature not only causes high energy consumption, but also results in big internal stress and many defects of the cured resin and, thereby, declines the overall integrated properties of the resultant materials. These two key disadvantages greatly limit the use of high-performance thermosetting resins in applications requiring outstanding overall properties.

Polyhedral oligomeric silsesquioxanes (POSS) are a family of nanoscale chemical structures that contain a silicon/oxygen core based on (SiO_{1.5})_n, where *n* = 6–12; *R* can be various types of organic groups, one (or more) of which is reactive or polymerizable.^{5–7} POSS molecules are uniformly porous in nanometer-range size; thus, they show very low dielectric constant (2.1–2.7).⁸ It is the combination of an inorganic core covered with an organic shell at the molecular level that has led POSS structures to be labeled as hybrid materials. The copolymerization of POSS macromers with organic monomers has been proved to be an efficient approach to develop various hybrids through the formation of covalent bonds between POSS cages and polymer matrices. Cao et al.⁹ synthesized octa(maleimidophenyl)silsesquioxane, which was then used to modify BT resins.

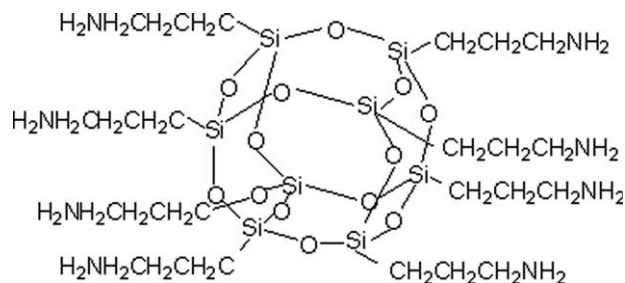
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Scheme 1 The structure of POSS-NH₂.

However, the synthesis of octa(maleimidophenyl)silsesquioxane is a complicate multistep procedure; in addition, the modified BT resin exhibits big brittleness because octa(maleimidophenyl)silsesquioxane has poor toughness because of the presence of short and rigid phenyl structure. Thus, there is an interest to develop new modified POSS/BT hybrids with improved integrated performance.

This article gives the first presentation on the preparation and properties of high-performance hybrids based on BT resin and octa(aminopropyl)silsesquioxane (POSS-NH₂), and the effects of POSS-NH₂ and its content on the integrated properties, including curing behavior and dielectric and thermal properties of BT resin, are investigated systematically.

EXPERIMENTAL

Materials

4,4'-Bismaleimidodiphenol methane (BDM) was obtained from Fengguang Chemical Co. (China). 2',2'-Diallylbisphenol A (DBA) was purchased from Laiyu Chemical Factory (China). POSS-NH₂ (Scheme 1) was synthesized by us according to the procedure in literature.¹⁰ 2,2'-Bis(4-cyanatophenyl) isopropylidene (BADCy) was supplied by Shangyu Shengda Chemical Co., (Zhejiang, China). Ethanol and dioxane (AR) were commercial products and purified before use.

Preparation of uncured POSS-NH₂/BT hybrids

Four formulations (Table I) were evaluated to see the effect of differing stoichiometry of POSS-NH₂ on the processing and performance parameters and also to establish the formulational latitude the system provides.

Twenty grams of BDM and 90 mL of dioxane were charged into a 250-mL flask with a magnetic stirrer. The mixture was heated to 76°C and maintained at that temperature until BDM were all dissolved in dioxane. Appropriate quantities of POSS-NH₂ were dissolved in ethanol, and then the POSS-NH₂/ethanol solution was dropped into the three-necked flask containing BDM/dioxane solution. After that, the mixture was heated to 100°C and maintained at that

temperature for 1 hr under N₂. When the reaction was over, solvents were distilled in vacuum to leave a yellow solid, which was washed with ethanol and dried in vacuum for 24 hr. The resultant product was coded as POSS-NH₂-BDM resin, the structure of which is shown in Scheme 2.

POSS-NH₂-BDM and DBA with a weight ratio of 1 : 0.86 were placed in a beaker equipped with a mechanical stirrer and thermometer. The mixture was heated to 130–135°C and maintained at that temperature range with stirring until a homogeneous liquid was obtained. The mixture was maintained at that temperature for additional 15 min to obtain a prepolymer. Appropriate amount of the prepolymer was slowly added into molten BADCy at 90°C with stirring and kept at 90°C for 30 min to obtain an uncured hybrid, which is coded as POSS-NH₂/BT.

Preparation of BT prepolymer

Appropriate quantities of BDM and DBA were put into a beaker with a mechanical stirrer and a thermometer. The mixture was heated to 130–135°C and maintained within that temperature range with stirring until a clear and brown liquid was obtained. The liquid was maintained at that temperature for additional 15 min to obtain a transparent liquid followed by cooling to 100°C. After that, preweighted BADCy was added into the beaker with stirring to obtain a brown-red transparent liquid, which was BT prepolymer.

Preparation of cured BT resin and hybrids

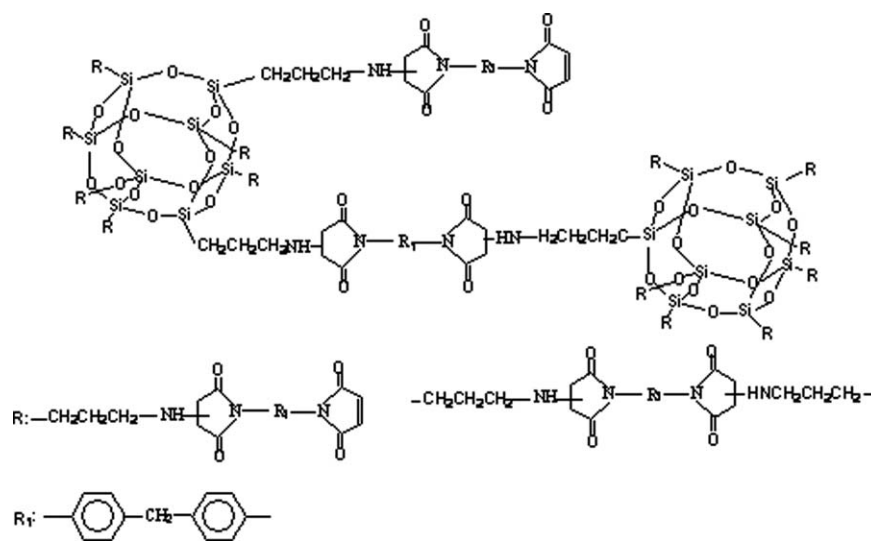
Each uncured sample was thoroughly degassed at 100°C and poured into a preheated (100°C) glass mold followed by curing per the procedure of 150°C/2 hr + 180°C/2 hr + 200°C/2 hr + 220°C/2 hr. After that, the cured sample was demolded and postcured in an air oven at 240°C for 4 hr.

Measurements

Differential scanning calorimetry (DSC) analyses were done by using DSC 200 F3 (NETZSCH, Germany) between 50°C and 320°C at a heating rate of 10°C/min in a nitrogen atmosphere.

TABLE I
Formulations of BT Resin and POSS-NH₂/BT Hybrids

Code	Weight ratio			
	DBA	BDM	POSS-NH ₂	BADCy
BT	0.86	1.00	0.00	2.79
F ₁ #	0.86	0.97	0.03	2.79
F ₂ #	0.86	0.93	0.07	2.79
F ₃ #	0.86	0.90	0.10	2.79



Scheme 2 The structure of POSS-NH₂-BDM.

Dielectric property was analyzed in a frequency range of 10–10⁶ Hz at room temperature by using a Novocontrol Concept 80 Analyzer (Germany). The sample dimension was $(25 \pm 0.2) \times (25 \pm 0.2) \times (3 \pm 0.1) \text{ mm}^3$.

Dynamic mechanical analysis (DMA) scans were performed in single-cantilever mode by using a TA Instruments DMA Q800 dynamic mechanical analyzer (USA) from 50°C to 320°C at a heating rate of 3°C/min and a frequency of 1 Hz. The glass-transition temperature (T_g) is defined as the peak temperature of $\tan \delta$ -temperature plot.

Fourier transform infrared (FT-IR) spectroscopy spectra were recorded between 400 and 4000 cm^{-1} , with a resolution of 2 cm^{-1} on a Nicolet FTIR 5700 spectrometer (USA). Thermogravimetric (TG) analyses from 50°C to 780°C were performed by using PerkinElmer TGA-7 (USA) at a heating rate 20°C/min in an air or nitrogen atmosphere, respectively. The initial decompose temperature (T_{di}) was defined as the point of intersection of the tangent of onset temperature and the tangent of the maximum degradation rate temperature. A scanning electron microscope (Hitachi S-4700, Japan) was used to observe the morphology of the fractured surfaces of samples.

RESULTS AND DISCUSSION

Curing behavior

Figure 1 shows the DSC curves of BT prepolymer and uncured POSS-NH₂/BT hybrids. It can be seen that each hybrid has one exothermic peak as BT prepolymer does, but the whole peak of the former shifts to lower temperature compared with that of the latter, and in addition, the peak of the hybrid with more content of POSS-NH₂ appears at lower temperature. For example, the whole peak of F₃# shifts to

lower temperature by about 13°C compared with that of BT prepolymer, demonstrating that hybrids have improved curing processing than BT prepolymer.

The curing mechanism of BT prepolymer mainly consists of "Diels-Alder" and "Ene" reactions between BDM and DBA as well as the cyclotrimerization of BADCy as shown in Scheme 3. The curing reactions of POSS-NH₂/BT hybrids are more complicated than BT prepolymer. Besides above reactions during the curing of BT prepolymer, the curing reactions of POSS-NH₂/BT hybrids also include the Michael addition (Scheme 2) between POSS-NH₂ and BDM and the reaction between POSS-NH₂ and BADCy (Scheme 4). The reaction between maleimide and cyanate ester is still controversial. Snow¹¹ reported

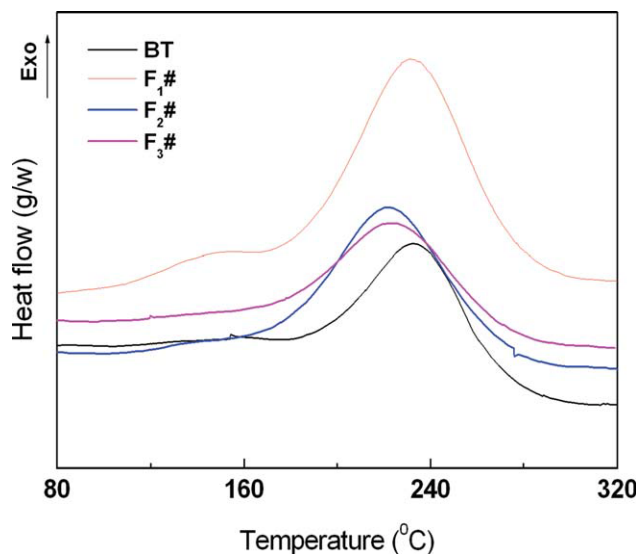
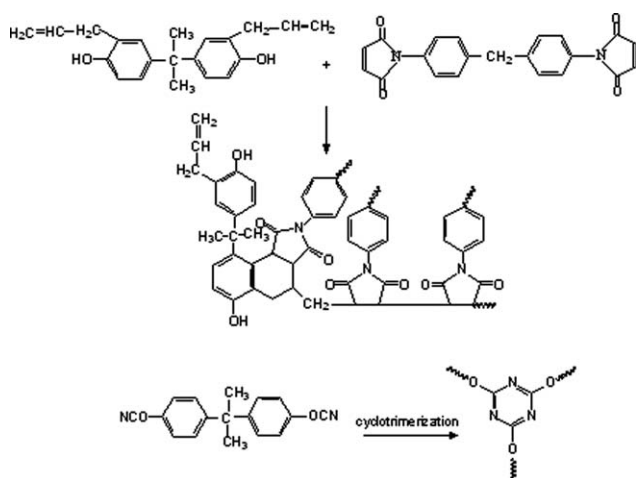


Figure 1 DSC curves of BT prepolymer and uncured POSS-NH₂/BT hybrids. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://www.interscience.wiley.com).]



Scheme 3 The curing reactions of BT prepolymer.

that pure cyanate ester cannot cure by itself at all, but why the self-curing of cyanate ester generally takes place is attributed to the presence of adventitious impurities. For this reason, catalysts are usually used to reduce the curing temperature and shorten the curing time of cyanate ester. It has been reported that hydrogen donors (e.g., phenols, acids, etc.) are suitable catalysts for the cyclotrimerization of cyanate ester.

In this study, the decrement in curing temperature is ascribed to the reactions between $-\text{OCN}$ and amine groups.¹² Amine groups can react with $-\text{OCN}$ groups upon mild heating or in the presence of base catalysts, interestingly, the product of the reaction is the catalyst for the formation of triazine rings. The amino groups on POSS-NH₂ can react easily by adding across the triple bond of $-\text{OCN}$ in the molecules of BADCy (Scheme 4). This causes POSS-NH₂ macromer to react with cyanate ester monomer at temperatures far below the temperatures for curing cyanate ester.

Dielectric property

Figures 2 and 3 show the dielectric constant and loss of cured BT resin and POSS-NH₂/BT hybrids,

respectively. All hybrids have similar dependence of dielectric constant on frequency as BT resin; in addition, the former has slightly lower dielectric constant than the latter. This can be attributed to the low dielectric constant of POSS. Many investigations have proved that the cubic silica core with the homogeneity nanopore of POSS can increase the free volume of polymers¹³; moreover, the molecules of POSS have lower polarity and thus low dielectric constant.

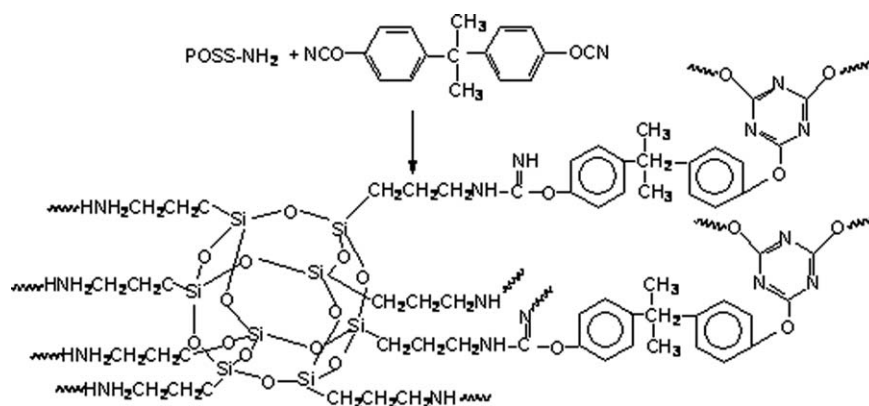
In case of dielectric loss, compared with BT resin, all hybrids not only have similar dependence of dielectric loss on frequency as BT resin, but also have significantly decreased loss at low frequency. All hybrids and BT resin have almost equal values of dielectric loss at the frequency higher than 10⁵ Hz.

It is known that the dielectric properties of a polymer are not only determined by the orientation and relaxation of dipoles, but also influenced by the structure of cured resin. Compared with BT resin, POSS-NH₂/BT hybrids have different chemical structure as stated above. There are more polar groups in hybrids than in BT resin, but these polar groups are thought to be restricted by the POSS structure in the hybrids; thus, POSS-NH₂/BT hybrids show lower dielectric loss than BT resin. However, with the increase in frequency, dipole orientation polarization may not happen because longer time is needed; in other words, the dielectric loss at high frequency is mainly related to the polarity of the chemistry, and thus, all hybrids and BT resin have almost equal dielectric loss.

Thermal resistance

Heat-resistance is generally characterized by T_g and TG behavior. The former indicates the movement capacity of molecular chains with the increase in temperature, and the latter denotes the thermal stability of molecular chains.

Generally, DMA is an effective method for measuring the T_g value of a thermosetting network. In this article, T_g is defined as the peak (maximum)



Scheme 4 Reaction between $-\text{NH}_2$ and $-\text{OCN}$ groups.

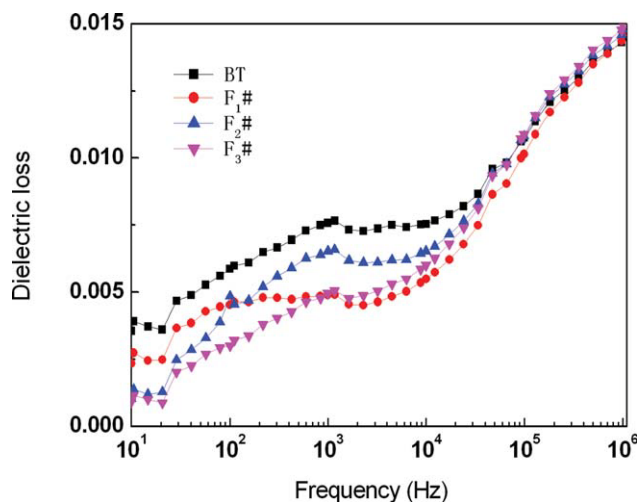


Figure 2 Dependence of dielectric constant of cured BT resin and POSS-NH₂/BT hybrids. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

temperature in the $\tan \delta$ -temperature plot from DMA tests. Figure 4 shows the overlay plots of $\tan \delta$ versus temperature for cured BT resin and POSS-NH₂/BT hybrids. It can be seen that BT resin and F₁# hybrid show a single peak, whereas other two hybrids possess similar shapes consisting of a strongly sharp peak with a small shoulder at low temperature. By Gaussian fitting,¹⁴ each curve can be divided into two peaks; one big peak appears at about 259°C, and another small peak appears at about 233°C, indicating that F₂# and F₃# hybrids have multiphase structure and, thus, two T_g values.

To find the reason behind the above phenomena, FT-IR analyses are conducted to check whether the curing of hybrids is complete or not. Figure 5 shows

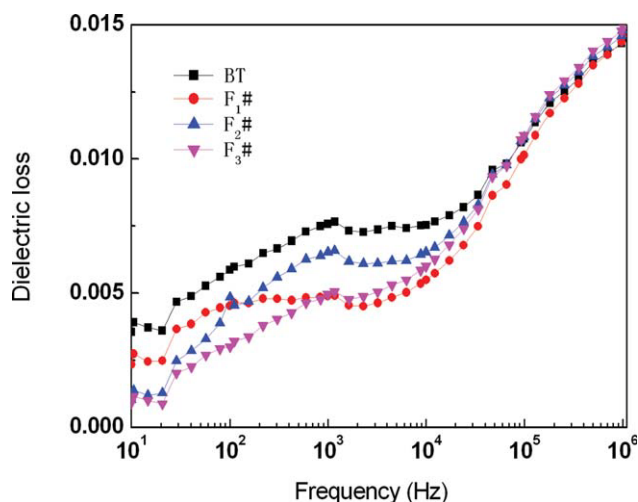


Figure 3 Dependence of dielectric loss of cured BT resin and POSS-NH₂/BT hybrids. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

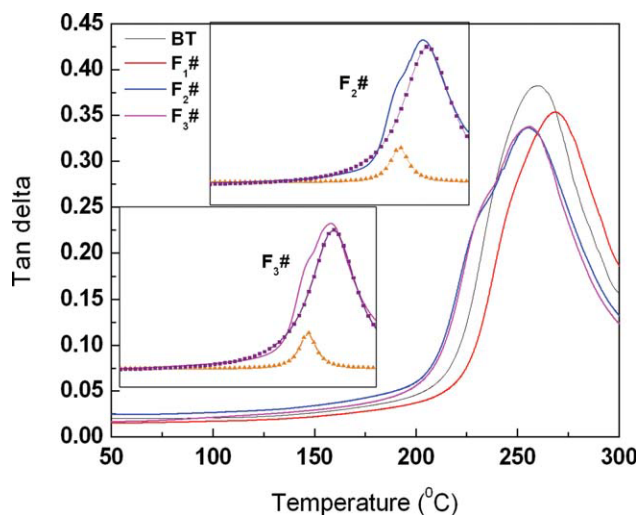


Figure 4 Overlay curves of $\tan \delta$ versus temperature for cured BT resin and POSS-NH₂/BT hybrids. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

the FT-IR spectra of cured BT resin and POSS-NH₂/BT hybrids; the spectrum of BT prepolymer is also supplied for comparison. In the spectrum of BT prepolymer, the absorption peaks at 3067 and 686 cm^{-1} belong to the stretching vibration of C=C group, and the peak near 2263 cm^{-1} is assigned to the stretching vibration of —OCN group. However, in case of each spectrum of any hybrid, these characteristic absorption peaks completely disappear, and a new absorption peak appears at 1370 cm^{-1} , which belongs to sym-triazine rings.¹⁵ These results confirm that under the curing and postcuring conditions developed herein, the curing of all hybrids is complete. In other words, the presence of the shoulder in Figure 4 does not result from the incomplete curing.

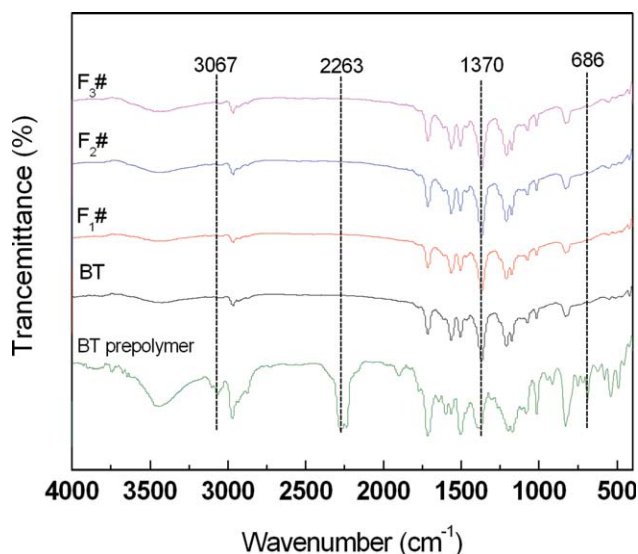


Figure 5 FT-IR spectra of BT prepolymer, cured BT resin, and hybrids. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

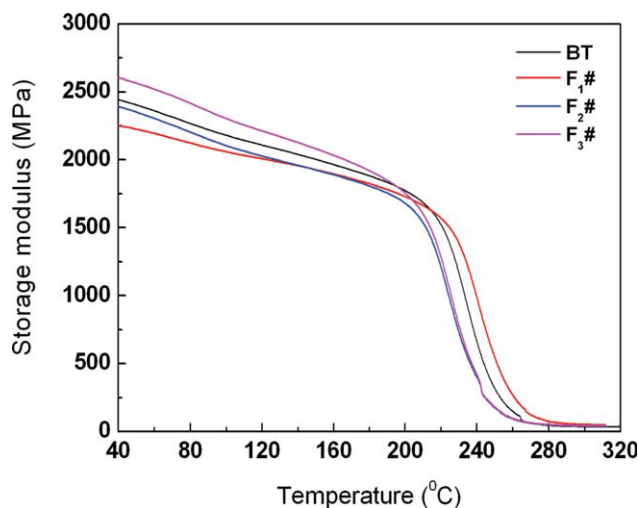


Figure 6 Storage moduli of cured BT resin and POSS-NH₂/BT hybrids. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

As discussed in the curing behavior before which POSS-NH₂/BT hybrids have different chemical structure from BT resin, the difference is enhanced when the content of POSS-NH₂ is big, which is reflected by the broad shapes of their $\tan \delta$ -temperature curves from DMA measurements. Generally, it is proposed that POSS has two competitive effects on the T_g of POSS/polymer hybrids: first, POSS cages on the segmental level could restrict the motion of macromolecular chains and, thus, improve T_g value; second, the presence of the bulky POSS cages enlarges the distance of cross-linking points; in other words, the addition of POSS tends to decrease the cross-linking degree of the network and, thus, the T_g value. In case of developed POSS-NH₂/BT hybrids, there are two additional factors affecting the cross-linking degree of hybrids. As stated above, POSS-NH₂ reacts with BDM by Michael addition reaction, enlarging the distance between the cross-linking points and, thus, resulting in decreased cross-linking degree; on the other hand, POSS-NH₂ substantially reduces the curing temperature and accelerates the whole curing process, and thus, under the same curing and postcuring procedures, hybrids have bigger cross-linking degree than BT resin. Obviously, the above four aspects play a combined role in any hybrid. When the content of POSS-NH₂ in hybrid is low, the positive effect for improving T_g plays domain role, so F1# has improved T_g value; however, with the increase in the content of POSS-NH₂, the negative effect attributed to the presence of POSS-NH₂ cannot be neglected, which is reflected in the plots of $\tan \delta$ versus temperature from DMA analyses.

To confirm the influence of POSS on the cross-linking degree, the cross-linking densities (X_{density})

of BT resin and POSS-NH₂/BT hybrids are calculated by following semiempirical equation based on DMA analyses.¹⁶

$$\log_{10} G' = 7 + 293X_{\text{density}}$$

where G' is the storage modulus (Fig. 6) of the cured sample in the rubbery plateau region; herein, G' is chosen as the modulus at the temperature which is 40°C higher than T_g . The corresponding cross-linking densities of BT resin and hybrids are depicted in Figure 7. It can be seen that F1# displays the biggest cross-linking density, whereas other hybrids have similar X_{density} as BT resin, reflecting that low POSS-NH₂ content tends to increase the X_{density} of resultant network and, thus, improve the T_g value.

Figure 4 also shows that the $\tan \delta$ peak of all hybrids has bigger half-width than that of BT resin; moreover, with the increase in POSS-NH₂ content, the $\tan \delta$ peak does tend to become wider, reflecting that the complexity of the morphology of hybrids increases.¹⁷ This statement is in good agreement with the different chemical structure of hybrids and is further confirmed by observing the fracture surfaces of hybrids and BT resin as shown in Figure 8. It can be seen that BT resin exhibits a single morphology; however, hybrids show a two-phase morphology. With the increase in the content of POSS-NH₂, more POSS-NH₂ phase can be observed. Note that the dispersion of POSS-NH₂ is homogeneous because of the reaction between POSS-NH₂ and BT resin.

The TG behavior can be reflected by TG analyses. Figures 9 and 10 show the TG and DTG curves of all samples in N₂ and in air. The typical data, such as T_{di} , the temperature of maximum degradation rate (T_{max}), and char yield (Y_c) at 780°C, obtained from these curves, are listed in Table II. It is not

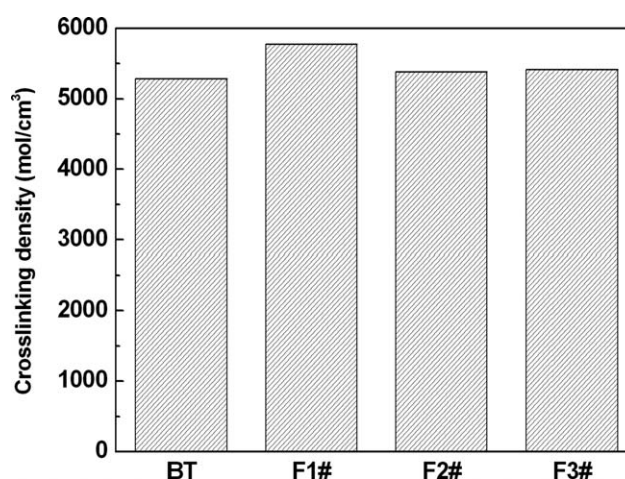


Figure 7 The cross-linking densities of BT resin and hybrids.

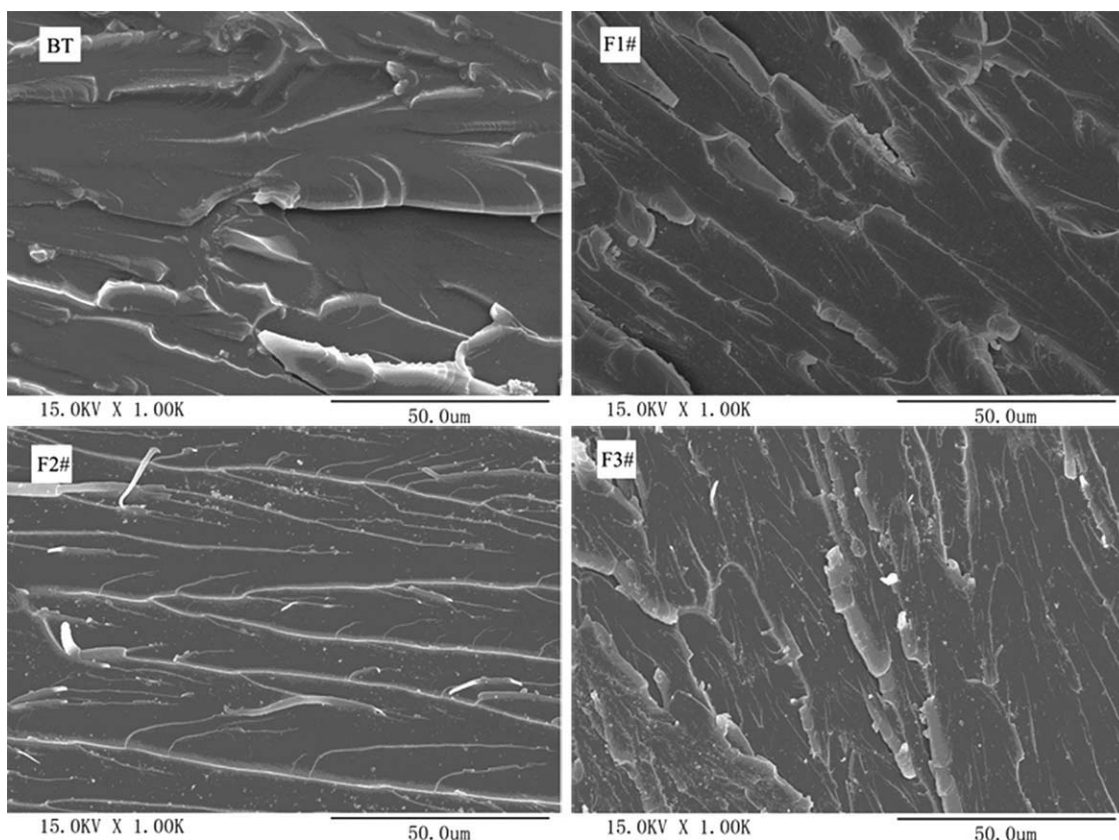


Figure 8 SEM micrographs of the fracture surface of cured BT resin and POSS-NH₂/BT hybrids.

surprising to find that all typical data in N₂ atmosphere are higher than those in air atmosphere because there is oxidation in air atmosphere. It can be seen that: (1) All hybrids have similar T_{di} value in either N₂ or air atmosphere as BT resin, reflecting that all hybrids have similar thermal stability as BT resin, which is famous for its outstanding thermal resistance. (2) According to DTG curves, the TG

curve of BT resin shows one step in N₂ atmosphere or two steps in air atmosphere, whereas the thermo-degradation of any hybrid can be divided into two steps no matter in what atmosphere. Specifically, in N₂ atmosphere, the first step of hybrids is the domain degradation stage, which takes place at the temperature (about 448°C) similar as that of BT resin. In air atmosphere, the first step of

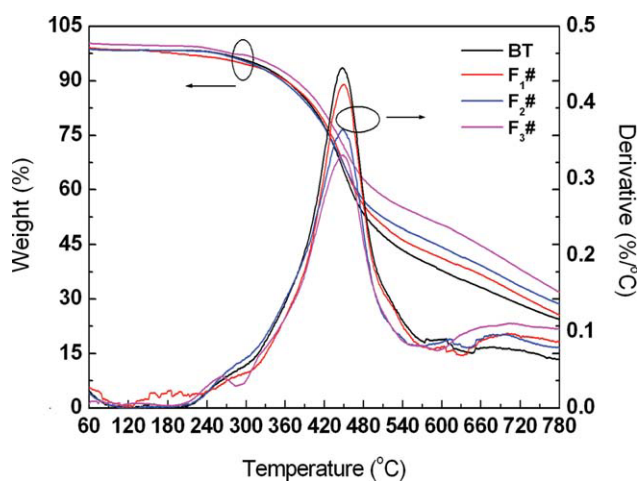


Figure 9 TG and DTG curves of cured BT resin and POSS-NH₂/BT hybrids in N₂ atmosphere. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

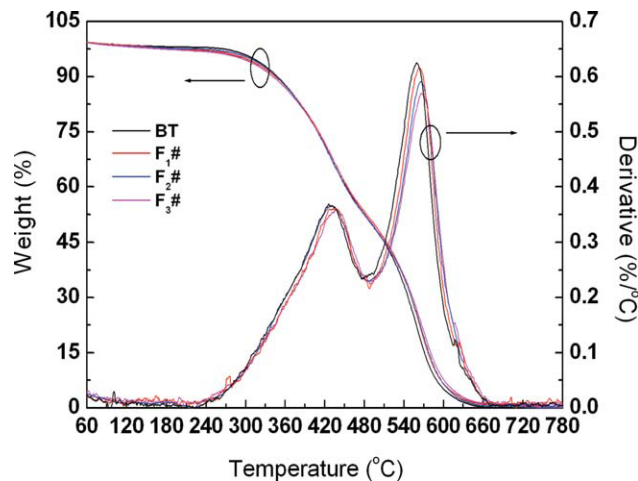


Figure 10 TG and DTG curves of cured BT resin and POSS-NH₂/BT hybrids in air atmosphere. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

TABLE II
Characteristic Data from TG Analyses of BT and POSS-NH₂/BT Hybrids

System	T_{di} (°C)		T_{max} (°C)		Y_c at 780°C (%)	
	In N ₂	In air	In N ₂	In air	In N ₂	In air
BT	380	311	448	431, 559	24.2	0
F ₁ #	387	315	451, 698	431, 564	26.2	0
F ₂ #	368	309	447, 697	430, 566	28.7	0
F ₃ #	370	311	449, 691	435, 569	32.5	0.28

thermodegradation for hybrids and BT resin takes place at similar temperature; however, the second step of hybrids appears at somewhat higher temperature than that of BT resin, indicating that the addition of POSS-NH₂ into BT resin forms more stable structure, which will degrade at high temperature. (3) Because of the excellent thermal stability of POSS, it is easy to know that hybrids have bigger char yield than BT resin and that Y_c value increases with the increase in POSS-NH₂ content; however, it is worthy to note that the increased Y_c value is bigger than the increase in POSS-NH₂ content, suggesting that there is a positive synergistic improvement. This can be contributing to the chemical junction between POSS-NH₂ and BT.

CONCLUSIONS

A novel kind of hybrids based on POSS-NH₂ and BT is developed. Compared with BT resin, POSS-NH₂/BT hybrids exhibit significantly decreased curing temperature, improved dielectric properties, and thermal resistance. The decreased curing temperature may be ascribed to the reactions between —OCN with amine groups; the improved dielectric properties (lower dielectric constant and loss) can be attributed to the lower polarity of POSS. The effect of POSS-NH₂ on the thermal stability of BT resin is very complex, which contributed to good thermal resistance of POSS, the change in cross-linking density, and the restriction of the macromolecular motion. The outstanding integrate properties of POSS-NH₂/BT hybrids supply a great potentiality for them to be used as the matrix for advanced composites or

high-performance adhesive in many cutting edge fields.

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