

Novel High-Performance Wave-Transparent Aluminum Phosphate/Cyanate Ester Composites

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ABSTRACT: Advanced wave-transparent composites are the key materials for many cutting-edge industries including aviation and aerospace, which should have outstanding heat resistance, low dielectric constant and loss as well as good mechanical properties. A novel kind of high-performance wave-transparent composites based on surface-modified aluminum phosphate $\text{AlPO}_4(\text{KH-550})$ and cyanate ester (CE) was first developed. The dielectric and dynamic mechanical properties of $\text{AlPO}_4(\text{KH-550})/\text{CE}$ composites were investigated intensively. Results show that $\text{AlPO}_4(\text{KH-550})/\text{CE}$ composites have decreased dielectric loss and higher

storage moduli than pure CE resin; in addition, the composites with suitable $\text{AlPO}_4(\text{KH-550})$ concentration remain the outstanding thermal property and low dielectric constant of pure CE resin. The reasons attributing to these results are discussed from the effects of $\text{AlPO}_4(\text{KH-550})$ on the key aspects such as morphology, curing mechanism, and interfacial adhesion of composites. © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 123: 1576–1583, 2012

Key words: aluminum phosphate; cyanate ester; thermomechanical property; dielectrical properties; interface

INTRODUCTION

Advanced wave-transparent composites are the key materials in many cutting-edge industries including aviation and aerospace, and their main features are outstanding heat resistance, excellent dielectric properties (low dielectric constant and loss), and good mechanical properties. Wave-transparent composites can be divided into inorganic^{1–3} and organic^{4–6} matrix composites; the former has good wave-transparent property only in the centimeter frequency range, while the latter can be used in millimeter frequency range and exhibits wide frequency feature; moreover, usually has good processing characteristics, high mechanical properties, and low price, and so it has occupied the important position.

It is known that a high performance matrix is the necessary and key component for developing

advanced polymeric composites; hence, a matrix with outstanding heat resistance, excellent dielectric properties, and reasonable process needs to be first developed for advanced wave-transparent composites. However, present high-performance resins do not completely meet the needs required by advanced wave-transparent composites. On the other hand, more and higher demands have been proposed owing to the rapid development of modern industries. Therefore, high-performance resin matrix for advanced wave-transparent composites has been the hot subject in the field of materials science and engineering.

Cyanate ester (CE) resin is one of high performance resins for advanced composites, which has excellent dielectric properties, outstanding thermal and mechanical properties as well as good processing characteristics, and thus has been considered as the most potential matrix for fabricating advanced structural/functional composites.^{7,8} However, its thermal property needs to be improved to meet restrict requirements by next generation products for aerospace and aviation industries.^{9–11} Many methods have been developed to modify CE resin, and some good results have been achieved, but it is found to be difficult to obtain a resin system with both thermal and dielectric properties; in other words, the improvement of thermal resistance is generally based on decreasing the dielectric property of original CE resin.

Developing organic/inorganic composites have been a vital subject, because composites have benefits

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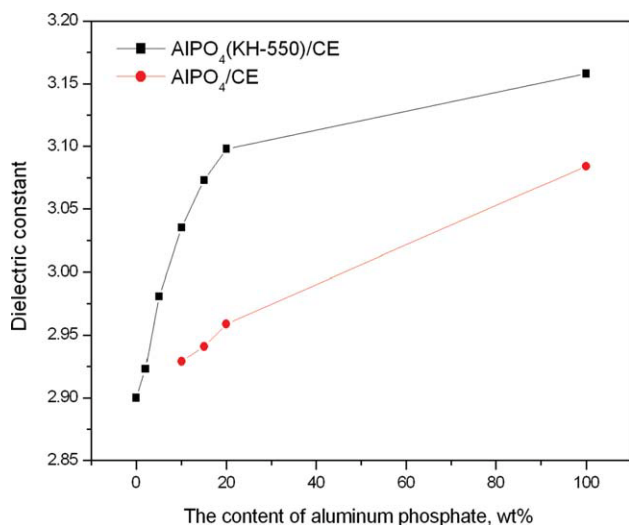


Figure 1 Dependence of dielectric constant on the content of aluminum phosphate in composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

for merging advantages of both inorganic and organic materials.^{12,13} To date, some inorganic filler/CE composites have been studied such as aluminum borate whisker/CE, carbon nanotube/CE, polyhedral oligomeric silsesquioxane/CE, and polysulfone/CE,^{14–17} and results have proved that these composites have better thermal properties than original CE resin. On the other hand, the effect of inorganic fillers on the dielectric properties of polymeric matrices has been investigated,^{18,19} and results suggest that dielectric properties of inorganic/CE composites are greatly depended on that of inorganic materials. So, to develop a novel kind of CE resin-based composites with excellent dielectric properties, new inorganic component should be selected.

Aluminum phosphate (AlPO_4) is an inorganic material with excellent thermal properties and modulus, which is stable even at the temperature as high as 1500 or 1800°C^{20,21}; moreover, it has outstanding dielectric properties, and so AlPO_4 -based composites have been one important sort of advanced wave transparent materials.

Based on outstanding features of CE resin and AlPO_4 as well as organic/inorganic composites, a novel kind of composites based on CE and AlPO_4 , coded as AlPO_4/CE , is developed; in addition, the dielectric and thermal properties of AlPO_4/CE composite are mainly investigated in this work.

EXPERIMENTAL

Materials

2,2'-Bis(4-cyanatophenyl) isopropylidene (CE) was brought from Shangyu Chemical Ltd. of Zhejiang in China. Bisphenol-A diglycidyl ether (E51) was made

by Sanmu Group of Jiangsu in China, its epoxy value is 0.52 mol/100 g. γ -Aminopropyl triethoxysilane (KH-550) was obtained from Shuguang Chemical Group Co. of Nanjing in China. Ethanol was purchased from Zhanwang Chemical Reagent Co. of Wuxi in China. AlPO_4 was brought from Meixing Chemical of Shanghai in China.

Surface treatment of AlPO_4

AlPO_4 was added into an appreciated quantity (1 wt % of AlPO_4) of ethanol solution of KH550 with thoroughly stirring for 2 h to form a homogenous system. After that the system was filtrated to wipe off the solvent at room temperature and subsequently dried at 120°C for 24 h under vacuum. The resultant product was coded as AlPO_4 (KH550).

Preparation of AlPO_4 (KH550)/CE composites

Appropriate quantity of CE was heated to 150°C and maintained at that temperature with stirring till a clear liquid was obtained. And then, AlPO_4 (KH550) was put into the melted CE liquid to form a mixture, and the mixture was maintained at 150°C for 2 h. After that, 1 phr E51 epoxy resin was added into the mixture with stirring. The mixture was kept at 150°C for additional 2 h. The resultant mixture was poured into a preheated (160°C) metal mold, and then the mold was put into a mold press and cured following the protocol of 170°C/1 h + 200°C/2 h with a pressure of 15 MPa. After that, the cured sample was demolded and postcured in an air oven at 240°C for 4 h. The resultant composite was coded as $n\%\text{AlPO}_4$ (KH550)/CE, where n represents the content of AlPO_4 in the composite.

Measurements

Fourier transform infrared (FTIR) spectra were recorded between 400 and 4000 cm^{-1} with a resolution of 2 cm^{-1} on a Prostar LC240 infrared spectrometer (USA).

Differential scanning calorimeter (DSC) measurements were performed with a DSC 2010 supported by TA Instruments (USA), ranging from room temperature to 300°C, at a heating rate of 10°C/min under nitrogen atmosphere.

A scanning electron microscope (Hitachi S-4700/Japan) was used to observe the morphology of samples. The resolution of the secondary electron image is 1.5 nm under 15 kV. All samples should be dried at 100°C for 6 h before test.

Dielectric properties were measured using a S914 dielectric loss (DL) test device with a QBG-3DQ instrument in the temperature of 25°C and a frequency of 10 MHz.

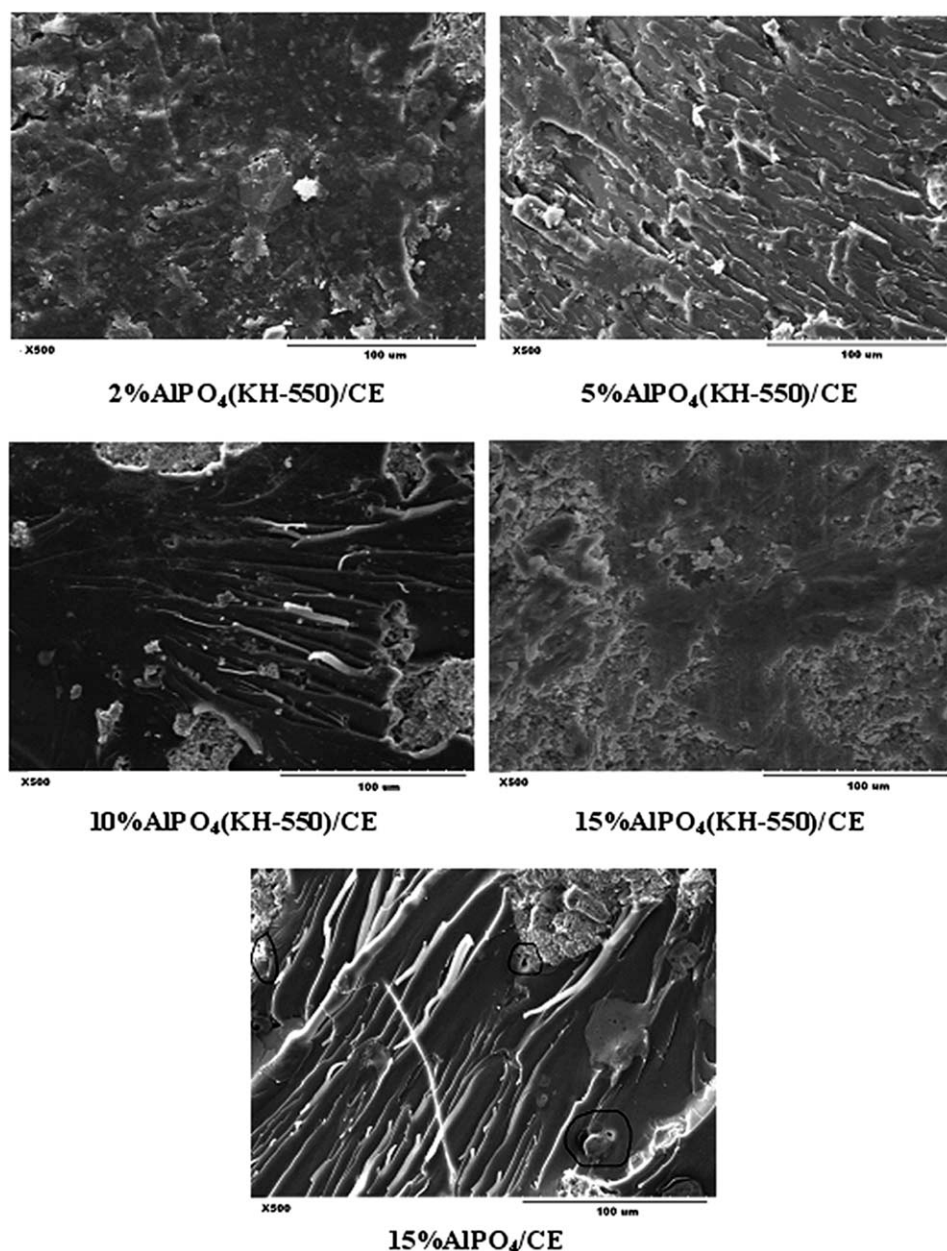


Figure 2 SEM micrographs of CE resin and AlPO₄(KH-550)/CE composites (a) 2%AlPO₄(KH-550)/CE, (b) 5%AlPO₄(KH-550)/CE, (c) 10%AlPO₄(KH-550)/CE, (d)15%AlPO₄(KH-550)/CE, and (e)15%AlPO₄/CE.

Dynamic mechanical analysis (DMA) scans were performed using TA DMA Q800 apparatus from TA Instruments (USA). A single cantilever clamping geometry was used. DMA tests were carried out from room temperature to 320°C with a heating rate of 3°C/min at 1 Hz. The glass transition temperature (T_g) is defined as the peak (maximum) temperature in the $\tan \delta$ -temperature plot.

RESULTS AND DISCUSSION

Dielectric property

A necessary and important property of high-performance wave-transparent composites is their excel-

lent dielectric properties including low dielectric constant and loss factor. Figure 1 shows the relationship of dielectric constant of AlPO₄(KH-550)/CE composites on the content of AlPO₄(KH-550). It can be seen that AlPO₄(KH-550)/CE composites have higher dielectric constant than CE resin. In the case of AlPO₄(KH-550)/CE composites, the composite with a larger content of AlPO₄(KH-550) tends to have higher dielectric constant. As it is known that the dielectric constant of a material is a function of its capacitance that is proportional to the quantity of the charge stored on either surface of the material in an applied electric field.²² With regard to a composite, its dielectric constant is dictated mainly by the

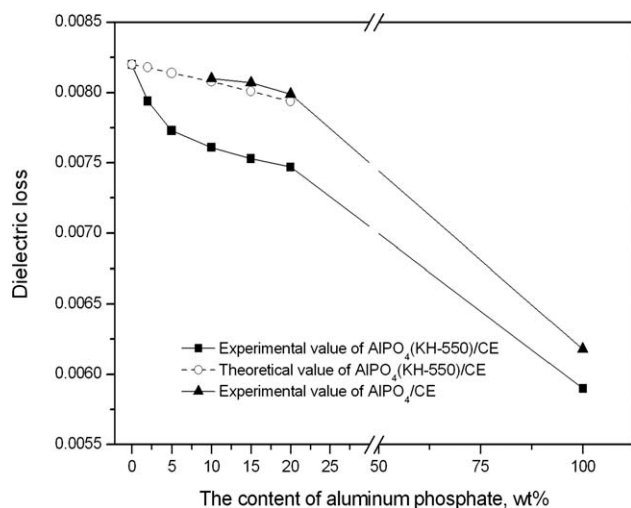


Figure 3 Dependence of dielectric loss on the content of aluminum phosphate in composites.

polarity of its matrix and those interfaces in subsurface (the area immediately next to the sample surfaces). The small addition of AlPO_4 or $\text{AlPO}_4(\text{KH-550})$ increases the quantity of the accumulated charge owing to an additional contribution from the polarization of matrix/filler interfaces and thus increases the dielectric constant. Because the additional contribution in $\text{AlPO}_4(\text{KH-550})/\text{CE}$ composite is noticeably higher than that in AlPO_4/CE composite resulting from the molecular polarity of KH-550, so in the case of the same filler loading, $\text{AlPO}_4(\text{KH-550})/\text{CE}$ composites have significantly higher dielectric constant than AlPO_4/CE composites. In addition, due to the poor interfacial adhesion between AlPO_4 and CE resin, the prepared sample tends to have porosities, which also results in lower dielectric constant.

The interfacial adhesion between the matrix and fillers can be proved apparently by SEM images (Fig. 2) of composites after impact tests. It can be

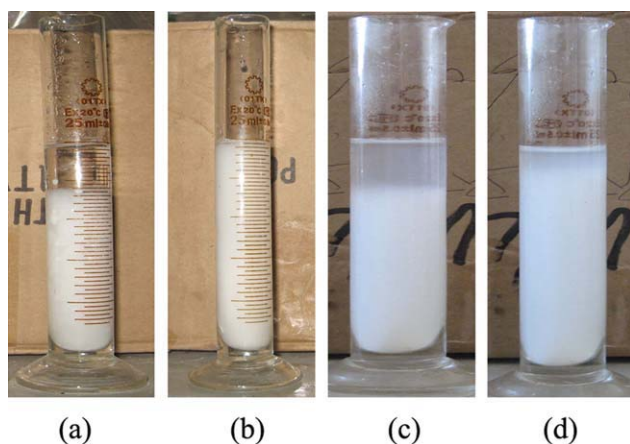


Figure 4 The photos of AlPO_4 and $\text{AlPO}_4(\text{KH-550})$ in water or liquid paraffin. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://www.interscience.wiley.com).]

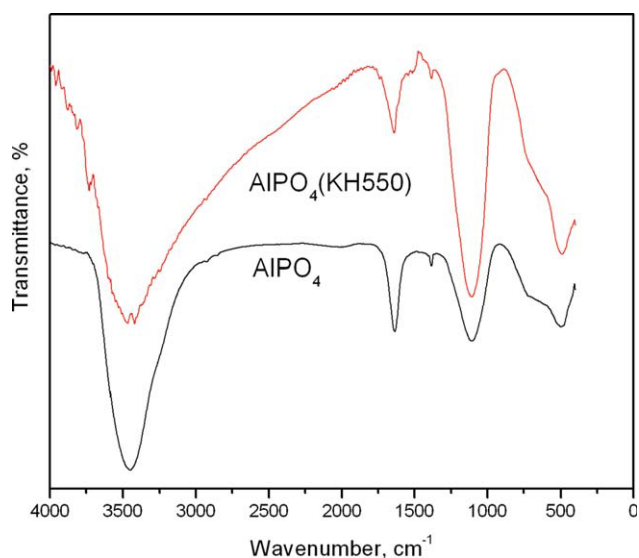
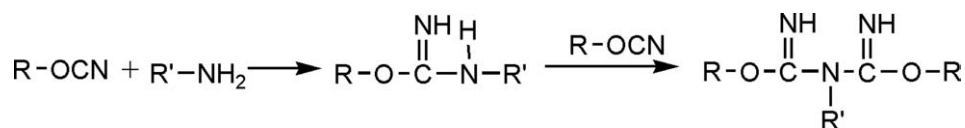


Figure 5 FTIR spectra of AlPO_4 and $\text{AlPO}_4(\text{KH-550})$. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://www.interscience.wiley.com).]

seen that there are porosities in the SEM image of 15% AlPO_4/CE composite—these porosities result from the poor adhesion—are original were the places of AlPO_4 , which fell off during the impact test owing to the poor interfacial adhesion between CE resin and AlPO_4 ; while such phenomenon does not appear in the SEM images of $\text{AlPO}_4(\text{KH-550})/\text{CE}$ composites because of the good interfacial adhesion between CE resin and $\text{AlPO}_4(\text{KH-550})$ (further discussion on the interfacial adhesion in DMA part will also confirm this point).

Figure 3 shows the dependence of DL factor on the content of $\text{AlPO}_4(\text{KH-550})$ in composites, it can be seen that $\text{AlPO}_4(\text{KH-550})/\text{CE}$ composites have lower DL than CE resin, and the higher is the $\text{AlPO}_4(\text{KH-550})$ content, the lower is the DL. In addition, with the same filler content, $\text{AlPO}_4(\text{KH-550})/\text{CE}$ composite has lower DL than AlPO_4/CE composite, indicating that the suitable surface treatment of AlPO_4 is necessary to fabricate composites with improved dielectric properties.

It is known that DL factor of a reinforced polymer composite is determined by those of both matrix and reinforcement as well as the interface polarization.²¹ The surface treatment of AlPO_4 changes its polarity and dispersion in the organic matrix, which can be evaluated by its dispersion in liquids with different polarities and its interfaces with organic phase. Figure 4 shows the photos of AlPO_4 and $\text{AlPO}_4(\text{KH-550})$ in water or liquid paraffin, it can be seen that AlPO_4 condenses in the bottom of water or liquid paraffin, indicating that AlPO_4 is neither a hydrophile nor lipophile compound; while this circumstance is significantly improved for $\text{AlPO}_4(\text{KH-550})$. The reason behinds this phenomenon can be



Scheme 1 The reactions between $-NH_2$ and $-OCN$ groups.

attributed to the nature of KH-550. The organic molecular chains of KH-550 provide $AlPO_4(KH-550)$ with lipophile property, resulting in a good dispersion of $AlPO_4(KH-550)$ in liquid paraffin; while the $-NH_2$ groups in KH-550 can form hydrogen bonds with water, leading to a good dispersion of $AlPO_4(KH-550)$ in water. The formation of hydrogen bonds can be confirmed by the split two peaks at $\sim 3450\text{ cm}^{-1}$ in the FTIR spectra shown in Figure 5.

In the case of $AlPO_4(KH-550)/CE$ composites, strong interfaces between CE resin and $AlPO_4(KH-550)$ are achieved through the chemical reactions between $-NH_2$ groups in KH-550 molecules and $-OCN$ groups in CE resin (Scheme 1); in addition, there still are hydrogen bonds between $-OH$ groups on the surface of $AlPO_4$ resulting from the hydrolysis of KH-550.²² These chemical reactions reduce the polarity groups in CE resin and filler phase.

It is worth noting that generally when the interfacial adhesion is strong and the interface has low polarization, the influence of the interface on DL factor is negligible²¹; but when the interfacial adhesion is weak, the influence of the interface on DL factor is not negligible. Hence the interface polarization of $AlPO_4(KH-550)/CE$ composite is negligible, but that of $AlPO_4/CE$ composite is not negligible. On the other hand, note that the good interfacial adhesion between $AlPO_4(KH-550)$ and CE will restrict the mobility of chain segments and thus leading to decreased DL; while the poor interfacial adhesion does not play this influence.

Briefly, the influence of the interfacial adhesion on the dielectric properties contains two aspects, a good adhesion will reduce the interfacial polarization and restrict the mobility of chain segments, leading to reduced DL; a bad adhesion brings the interfacial polarization, resulting in increased DL. Based on the above-mentioned analyses, it can be stated that the DL factor of composite with $AlPO_4(KH-550)$ is therefore dominated by the ratio of CE resin and $AlPO_4(KH-550)$, which will decrease with increasing the content of $AlPO_4(KH-550)$, because $AlPO_4(KH-550)$ has much lower DL factor than that of CE resin, while the DL factor of $AlPO_4/CE$ composite is dominated by not only the ratio of CE resin and $AlPO_4$ but also the interface polarization. In other words, the overall polarization of $AlPO_4(KH-550)/CE$ composites is lower than that of $AlPO_4/CE$ composites; hence, $AlPO_4(KH-550)/CE$ composites have lower DL factor than $AlPO_4/CE$ composites.

Figure 3 also provides the theoretical DL values of each composite calculated by a mixture rule as shown in eq. (1).²³

$$DL_c = DL_m V_m + DL_f V_f \quad (1)$$

where V_f and V_m refer to the volume fraction of the filler and the matrix, respectively; DL_f and DL_m represent the DL values of the composite and the neat matrix, respectively.

The reason leading to the difference between theoretical and experimental DL values is that eq. (1) does not consider the influence of interfaces on the DL. On the other hand, Figure 3 shows that the difference between theoretical and experimental DL tends to be enhanced with increasing the content of $AlPO_4(KH-550)$ in composites, this phenomenon can be explained by the relationship of interfacial adhesion with the content of $AlPO_4(KH-550)$ as discussed earlier.

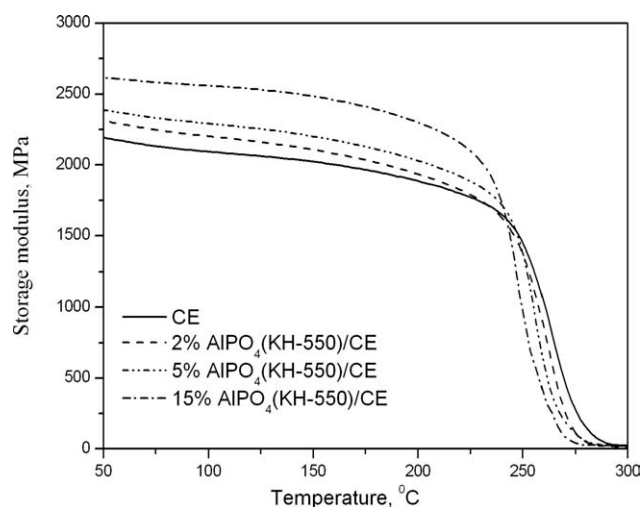


Figure 6 Overlay curves of storage modulus versus temperature for CE resin and $AlPO_4(KH-550)/CE$ composites.

TABLE I
The Coefficient C Values of $AlPO_4(KH-550)/CE$ Composites

Material	C
CE	/
2% $AlPO_4(KH-550)/CE$	1.0260
5% $AlPO_4(KH-550)/CE$	1.0184
15% $AlPO_4(KH-550)/CE$	1.0019

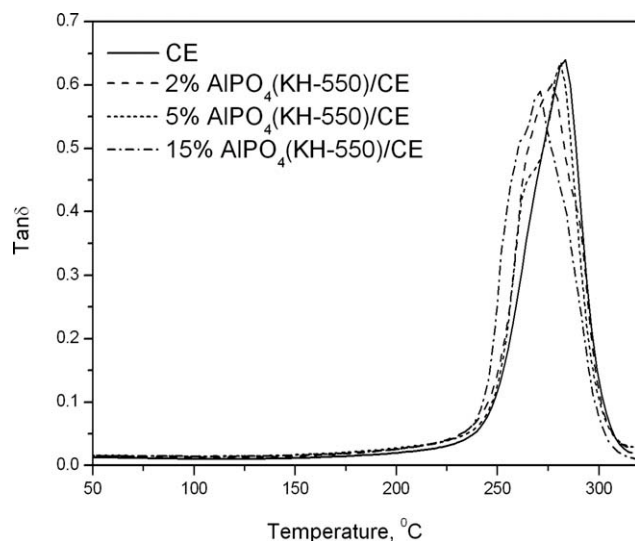


Figure 7 Overlay curves of $\tan \delta$ versus temperature for CE resin and $\text{AlPO}_4(\text{KH-550})/\text{CE}$ composites.

Dynamic mechanical property

The overlay plots reflecting storage modulus (E') as a function of temperature for $\text{AlPO}_4(\text{KH-550})/\text{CE}$ composites and CE resin are shown in Figure 6. $\text{AlPO}_4(\text{KH-550})/\text{CE}$ composites have higher storage moduli in the glassy state than CE resin; moreover, that with larger content of $\text{AlPO}_4(\text{KH-550})$ has higher E' value. The great improvements in E' value clearly confirm the reinforcing effect of $\text{AlPO}_4(\text{KH-550})$ in composites. The effectiveness of fillers on the modulus of a composite can be described by the coefficient (C), which can be calculated by eq. (2).²⁴

$$C = \frac{(E'_G/E'_R)_{\text{composite}}}{(E'_G/E'_R)_{\text{resin}}}$$

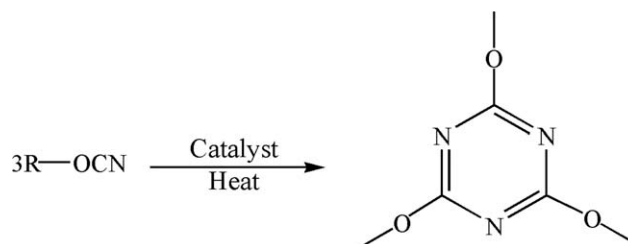
where E'_G and E'_R are the storage moduli in the glassy and rubbery region, respectively.

High C value means that the effectiveness of fillers on the modulus of composite is small. The modulus of each composite at 100 and 200°C is regarded as E'_G and E'_R , then the coefficient C values of all $\text{AlPO}_4(\text{KH-550})/\text{CE}$ composites are calculated by eq. (2), and corresponding data are summarized in Table I. It can be seen that the composition of $\text{AlPO}_4(\text{KH-550})/\text{CE}$ composites has an obvious effect on C values. Specifically, the composite with larger content of $\text{AlPO}_4(\text{KH-550})$ has smaller C value, suggesting that the addition of $\text{AlPO}_4(\text{KH-550})$ to CE resin retards the motion of molecular segments in CE resin.

Figure 7 shows overlay $\tan \delta$ -temperature curves of CE resin and $\text{AlPO}_4(\text{KH-550})/\text{CE}$ composites, and it can be seen that the content of $\text{AlPO}_4(\text{KH-550})$ has significant effect on the shapes of $\tan \delta$ peaks. The

shape of $\tan \delta$ peak may be used as a convenient indicator of the morphology state of the phases within multiphase materials.²⁵ The broadness of a relaxation indicates the complexity of the morphology of a material. $\text{AlPO}_4(\text{KH-550})/\text{CE}$ composites indeed tends to have wider $\tan \delta$ peak than CE resin, indicating that $\text{AlPO}_4(\text{KH-550})/\text{CE}$ composites have more complex morphology than CE resin. Figure 7 shows that CE resin only has a single peak, while the incorporation of $\text{AlPO}_4(\text{KH-550})$ tends to add a shoulder in the low temperature side of the main peak, suggesting that $\tan \delta$ peaks of composites are composed of two overlapped peaks. In detail, compared to the peak of CE resin that of 2% $\text{AlPO}_4(\text{KH-550})/\text{CE}$ composite becomes wider and shifts slightly toward low temperature; while either 5% $\text{AlPO}_4(\text{KH-550})/\text{CE}$ or 15% $\text{AlPO}_4(\text{KH-550})/\text{CE}$ composite shows one main peak with an obvious shoulder in the side of lower temperature, meaning that there is more than one phase in these composites. The reason behind this phenomenon is the different structures between composites and CE resin. It is known that crosslinked triazine ring is the structure of cured CE resin via the polymerization of $-\text{OCN}$ groups (Scheme 2); while, with regard to $\text{AlPO}_4(\text{KH-550})/\text{CE}$ composites, besides triazine structure, there is new additional structure resulting from the reaction between $-\text{NH}_2$ groups in KH-550 molecules and $-\text{OCN}$ groups in CE resin (Scheme 1). These two kinds of structures will be reflected in the $\tan \delta$ -temperature curves. When the content of $\text{AlPO}_4(\text{KH-550})$ is very small (e.g., 2% $\text{AlPO}_4(\text{KH-550})/\text{CE}$), the additional new structure occupies very small proportion in the whole network structure, and its peak is too small to be clearly observed. With increasing the content of $\text{AlPO}_4(\text{KH-550})$, the proportion of the additional new structure in the whole network structure increases, and so the composite has obvious $\tan \delta$ peak. On the other hand, because $\tan \delta$ peaks of the two structures appear in two close temperature ranges, $\text{AlPO}_4(\text{KH-550})/\text{CE}$ composites exhibit a main $\tan \delta$ peak with a shoulder.

On the other hand, the shapes of $\tan \delta$ peak for all $\text{AlPO}_4(\text{KH-550})/\text{CE}$ composites suggest that all composites have two apparent T_g values, which are



Scheme 2 Formation of triazine rings.

TABLE II
Information of Tan δ Peaks of CE Resin and AlPO₄(KH-550)/CE Composites from DMA Tests

Material	Shape (temperature from low to high)	T_g (°C)	Peak intensity
CE	Single peak	283	0.64
2%AlPO ₄ (KH-550)/CE	A main peak, a shoulder	276, 285	0.60
5%AlPO ₄ (KH-550)/CE	A shoulder, a main peak	268, 281	0.64
15%AlPO ₄ (KH-550)/CE	A shoulder, a main peak	260, 271	0.59

summarized in Table II. Similar phenomena also appear in other polymers and polymeric composites.^{25–28} When a composite has very small content of AlPO₄(KH-550) (up to 5 wt %), its two T_g values are similar with the single T_g value of CE resin; while when a composite has somewhat large content of AlPO₄(KH-550), its two T_g values tend to shift toward lower temperature range. The phenomenon can be mainly interpreted by two facts that the addition of AlPO₄(KH-550) to CE resin changes the curing reaction and thus the cured structure. First, in AlPO₄(KH-550)/CE composites, AlPO₄(KH-550) acts as physical interlock points in crosslinked structure, which not only provides a sterically hindered environment for curing reactions of composites, but also may restrain chain mobility, especially in the network formed by rigid aromatic rings. Hence, it is difficult for composites to attain the same level of curing as pure CE resin. Second, $-NH_2$ groups on the surfaces of AlPO₄(KH-550) can catalyze the polymerization of CE; but when the content of AlPO₄(KH-550) is small, the main reaction is still the formation of triazine rings,²⁴ and so the combined effect of AlPO₄(KH-550) is hindering the curing of CE resin, which can be further confirmed by DSC curves of pure CE and 15% AlPO₄(KH-550)/CE as shown in Figure 8. It can be seen that the addition of AlPO₄(KH-550) to CE can significantly shift the whole curing peak from 212 to 255 to 208–270°C,

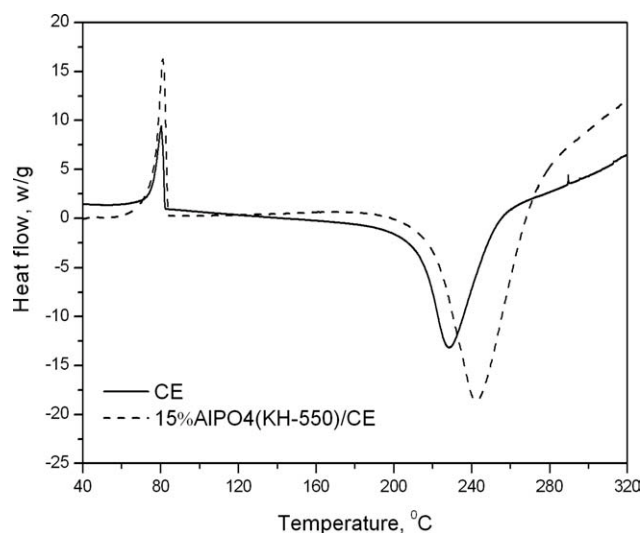


Figure 8 DSC curves of CE and 15%AlPO₄(KH-550)/CE.

suggesting that using the same curing and postcuring procedures, AlPO₄(KH-550)/CE composite has relatively lower crosslinking degree than CE resin.

Compared to CE resin, AlPO₄(KH-550)/CE composites not only have different T_g values and shapes of tan δ peak but also exhibit significantly smaller intensity of tan δ peaks. According to Matsuoka,²³ the greater is the motion associated with the transition, the greater is the intensity of tan δ peak. Table II suggests that AlPO₄(KH-550) decreases the molecular motion owing to its interaction with CE resin and thus leads to smaller intensity of tan δ peak.

CONCLUSIONS

A novel kind of high-performance wave-transparent composites based on surface-modified AlPO₄, AlPO₄(KH-550), and CE was developed. AlPO₄(KH-550)/CE composites have decreased DL and higher storage moduli than pure CE resin; in addition, the composites with suitable content of AlPO₄(KH-550) remain the outstanding glass transition temperature and low dielectric constant as pure CE resin does. The results are attributed to the effect of AlPO₄(KH-550) on the key aspects such as morphology, curing mechanism, and interfacial adhesion of composites.

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