

Thermal and Insulating Properties of Epoxy/Aluminum Nitride Composites Used for Thermal Interface Material

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ABSTRACT: We synthesized an epoxy matrix composite adhesive containing aluminum nitride (AlN) powder, which was used for thermal interface materials (TIM) in high power devices. The experimental results revealed that adding AlN fillers into epoxy resin was an effective way to boost thermal conductivity and maintain electrical insulation. We also discovered a proper coupling agent that reduced the viscosity of the epoxy-AlN composite by AlN surface treatment and increased the solid loading to 60 vol %. For the TIM sample made with the composite adhesive,

we obtained a thermal conductivity of 2.70 W/(m K), which was approximately 13 times larger than that of pure epoxy. The dielectric strength of the TIM was 10 to 11 kV/mm, which was large enough for applications in high power devices. Additionally, the thermal and insulating properties of the TIM did not degrade after thermal shock testing, indicating its reliability for use in power devices. © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 124: 669–677, 2012

Key words: composites; adhesives; thermal properties

INTRODUCTION

With the miniaturization of electronic devices, heat dissipation has emerged as a critical problem that affects the device performance and reliability, especially in high power devices such as high power diode lasers, high-brightness light emitting diodes, and high power transistors.^{1,2} The heat generated by these high power devices during operation needs to be efficiently conducted to the heat sink and then dissipated to ambient environment. Between the high power device and heat sink, there is a layer of thermal interface material (TIM) that needs to be thermally conductive and electrically insulating (Fig. 1). Since the polymers used in TIM like epoxy resins and silicone gels exhibit a poor thermal conductivity between 0.1 and 0.3 W/(m K), they cannot meet the demands of fast heat conduction for state-of-the-art

electronic products. Thus, there is a need to develop a TIM with a high thermal conductivity.

Extensive research has been carried out to enhance the thermal conductivity of the polymers by adding thermally conductive fillers. These fillers can be metallic powders,³ carbon-based fillers,^{4–7} or ceramic powders.^{8–20} The polymers filled with metallic particles or carbon materials can exhibit a thermal conductivity of more than 10 W/(m K), but they are not suitable for applications requiring electrical insulation. Ceramic powders have a high thermal conductivity and are not electrically conductive, so they can be used as fillers for specific applications in high power devices.

Alumina (Al₂O₃),^{8–10} silicon oxide (SiO₂),^{9,11} silicon nitride (Si₃N₄),¹¹ silicon carbide (SiC),^{10,12} boron nitride (BN),^{10,13–15} aluminum nitride (AlN),^{12,15–20} and other ceramic fillers have been included into a polymer matrix to form a composite with satisfactory properties. Among the ceramic fillers, AlN has recently attracted a lot of attention, because it has a thermal conductivity of 266 W/(m K).²¹ Xu and Chung treated the surface of AlN particles with silane coupling agents and achieved a composite with a thermal conductivity of 11.0 W/(m K).¹⁵ Hsieh and Chung fabricated an epoxy molding compound (EMC) filled with AlN powders having an average size of 35.3 μm and obtained a thermal conductivity

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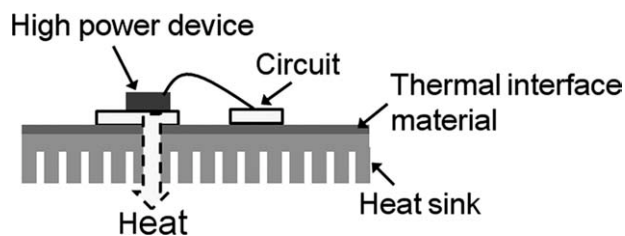


Figure 1 Structural schematic of a high power device with thermal interface material.

of 14 W/(m K).¹⁶ Lee et al. filled the epoxy resin with AlN particles that were coated by two layers of surfactants and achieved a thermal conductivity of 3.39 W/(m K), which was 15 times higher than that of pure epoxy.^{17,22}

For TIM in high power devices, as shown in Figure 1, the processing method used for EMC fabrication^{12,15,16,18,19} is not suitable because the hot pressing method with a high pressure of more than 10 MPa is not suitable for device mounting on the substrate. For this specific TIM application, Ref. 17 demonstrated a processing method to synthesize a composite adhesive with low viscosity and enhanced thermal conductivity. However, their technique of double-layer coating is quite complicated.²² In this study, we aim to perform one-step surface treatment on the AlN particles and synthesize an epoxy-AlN composite adhesive used as the TIM in a high power device such as insulated gate bipolar transistors (IGBT).

EXPERIMENTAL

Materials and surface treatment of AlN

Halogen-free bisphenol-A liquid epoxy resin CYD-128 (supplied by Baling Petrochemical Enterprise, Yueyang, China) was used as the polymer matrix, for which the epoxide equivalent weight is 184 to 194 g/eq and the viscosity is 11 to 14 Pa s at 25°C. The curing agent is an acid anhydride curing agent operated at high temperatures, QS-1858Y, from Qingda Qi-Shi Company, Beijing, China. The AlN powder with a median size of 6.01 μm was provided by Fujian Sinocera Advanced Materials Co., Ltd, Fuzhou, China, as shown in Figure 2. The physical properties of the AlN powder and epoxy resin are shown in Table I. Acetone (analytical reagent grade) was used as the solvent and was supplied by Beijing Chemical Works, Beijing, China.

Three silane coupling agents (KH-550, KH-560, and KH-792) and two titanate coupling agents (TC-2 and TC-27) were used without further purification, as all agents have a purity of over 97%. KH-560 was provided by Nanjing Xinhui Scientific Co., Ltd, Nanjing, China. KH-550 and KH-792 were supplied by Nanjing Yudeheng Fine Chemical Co., Ltd, Nanjing, China, and TC-2 and TC-27 were from Anhui

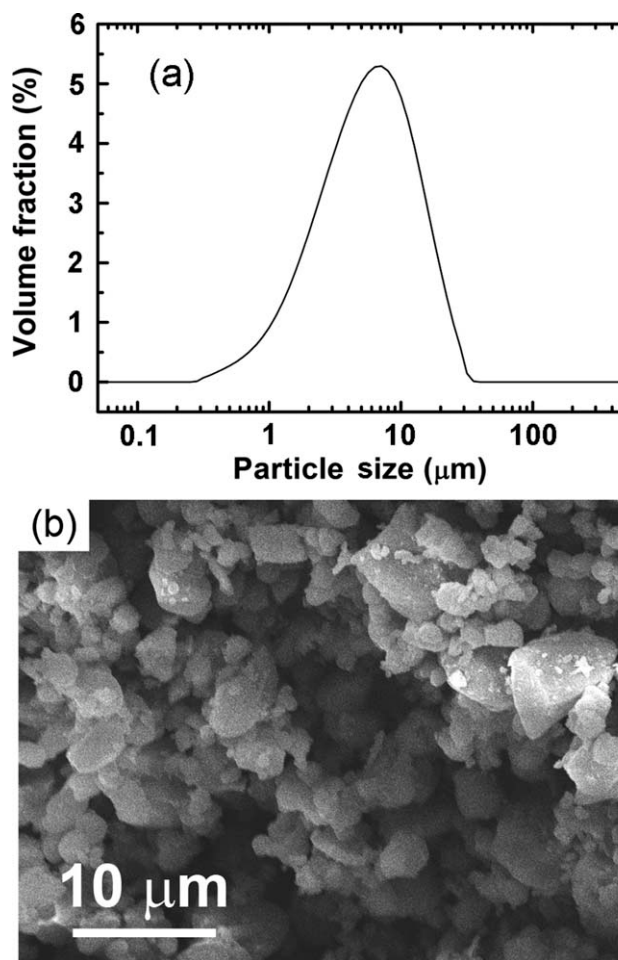


Figure 2 (a) Size distribution and (b) SEM image of AlN fillers.

Taichang Chemical Co., Ltd, Chuzhou, China. The names and molecular formulas of the coupling agents are shown in Table II.

The surface treatment of the AlN powder was carried out as follows: (i) mixing fillers and coupling agent in acetone with mechanical stirring at room temperature for 30 min, (ii) dispersing through ultrasonication at 50°C for 10 min, (iii) evaporating acetone at 60°C for about 0.5 to 1 h, and (iv) baking the treated AlN powder at 100°C for 6 h.

Sample preparation

The procedure for sample preparation is shown in Figure 3(a): (i) mixing the epoxy and the curing

TABLE I
Physical Properties of Epoxy Resin and AlN Powder

Materials	Thermal conductivity (W/(m·K))	Dielectric strength (kV/mm)	Density (g/cm ³)	Purity (%)
Epoxy	0.1–0.3	10–30	1.15	>99.8
AlN	150–250	>20	3.24	98.5

TABLE II
Coupling Agents

Symbol	Chemical name	Molecular formula
KH-550	γ -Aminopropyl-triethoxy silane	$\text{NH}_2(\text{CH}_2)_3\text{Si}(\text{OC}_2\text{H}_5)_3$
KH-560	γ -Glycidoxypropyl-trimethoxy silane	$\text{CH}_2\text{OCHCH}_2\text{O}(\text{CH}_2)_3\text{Si}(\text{OCH}_3)_3$
KH792	[3-(2-Aminoethyl)aminopropyl]-trimethoxy silane	$\text{NH}_2(\text{CH}_2)_2\text{NH}(\text{CH}_2)_3\text{Si}(\text{OCH}_3)_3$
TC-2	Titanate coupling agent TC-2	$(\text{CH}_3)_2\text{CHOTi}[\text{OPOOH-PO}(\text{OR})_2]_3$
TC-27	Titanate coupling agent TC-27	$(\text{CH}_3)_2\text{CHOTi}[\text{OPO}(\text{OR})_2]_3 \cdot [\text{R-C}_6\text{H}_4\text{-O-(CH}_2\text{CH}_2\text{O)}_x]_m \cdot \text{PO}(\text{OH})_m$

agent with mechanical stirring at 60°C for 5 min, (ii) surface modification of the AlN fillers as described in the Materials and surface treatment of AlN, (iii) mixing the epoxy/curing agent and AlN fillers first with a vigorous mechanical stirring at 60°C for 0.5 h, and then dispersing through ultrasonication at 60°C for 0.5 h, (iv) degassing the precuring mixture (adhesive) in a vacuum oven at 80°C for 2 h, (v) molding to make a thin-film sample before finally curing the sample. To prepare a thin-film sample, some adhesive was poured on a piece of stainless steel foil after which another piece of foil was placed on the top of the adhesive. Weights were then placed on top of the foil to exert pressure. With an estimated pressure of 1.5 kPa provided by weights on the top foil, the thickness of the sample can be controlled within a range of 100 to 300 μm . The adhesive with the foils was then cured in an oven to form a TIM sample [Fig. 3(b) shows the curing curve].

Characterization and testing

The thermal conductivity, κ , was obtained from $\kappa = \rho \times \alpha \times C_p$, where ρ is the density of the sample, α is the thermal diffusivity and C_p is the heat capacity. The thermal diffusivity was measured at room temperature by a Fourier Transform-Thermal Analysis system, which can accurately characterize the thin-film samples within a thickness of hundreds of micrometers.²³ The heat capacity was measured by a TA DSC Q-2000 tool, in which samples (4 mg powder) were heated under flowing nitrogen gas with a heating rate of 10°C/min. The thermal conductivity data in this article was obtained at 25°C. At higher temperatures, the thermal conductivity value calculated from the equation above will be larger due to its larger heat capacity.

The dielectric strength, E_b , was calculated by $E_b = V_b/h$, where h and V_b represent the thickness of the thin-film sample and the breakdown voltage, respectively. V_b was measured by a CY-2671A Auto Breakdown Tester.

The viscosity of the precuring mixture was measured with a Physica MCR300 Modular Compact Rheometer using a volume of 20 mL.

The natural fracture surface of the composite samples was observed by scanning electron microscopy (SEM: JEOL JSM-6460LV, Japan).

Thermal shock testing was performed in a Thermal Shock Tester CTS01F. The temperature range is from -25 to 80°C, as shown in Figure 4.

The size distribution of AlN powder was measured by a Malvern Mastersizer 2000 Laser Diffraction Particle Size Analyzer.

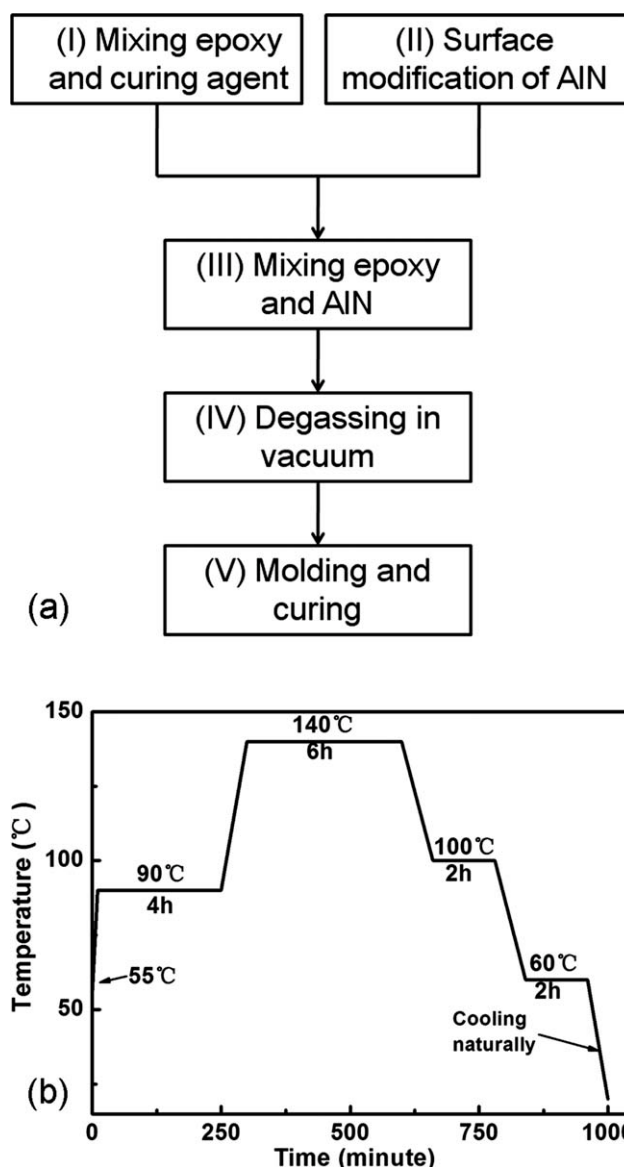


Figure 3 (a) Process flow of the epoxy-AlN composite material, and (b) procedure for curing.

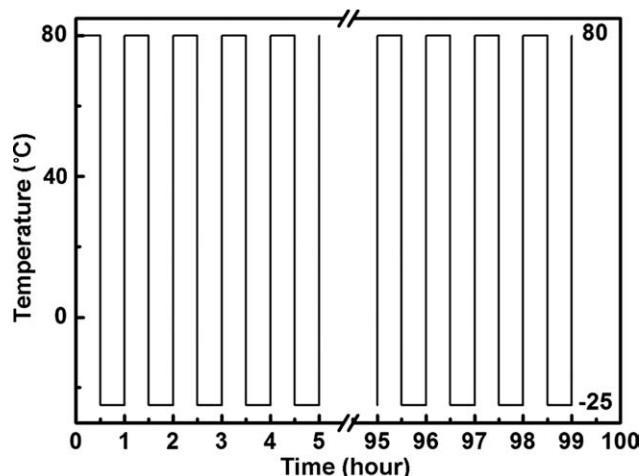


Figure 4 Temperature variation during thermal shock testing.

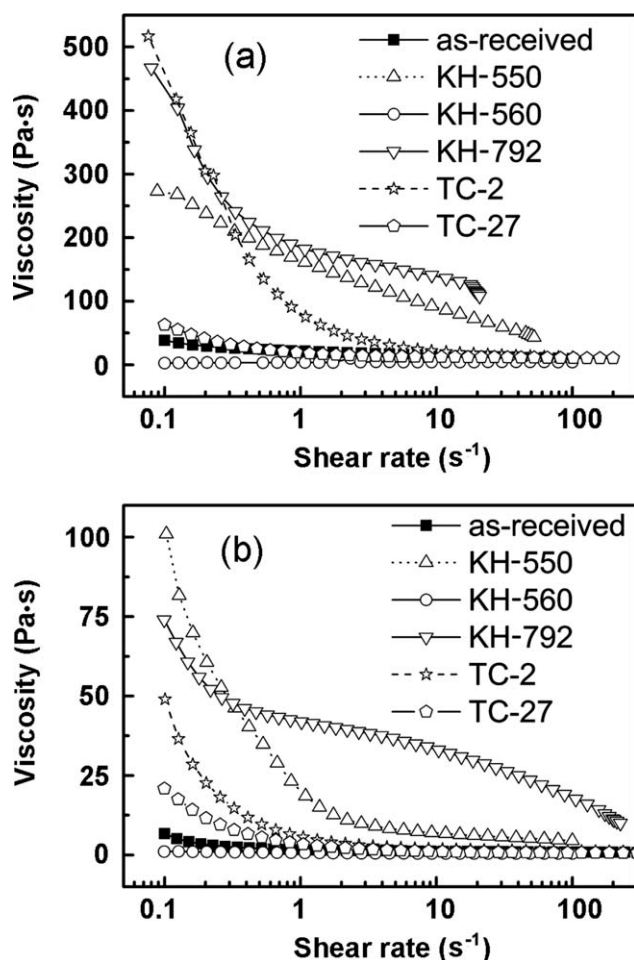


Figure 5 Viscosity of an epoxy-AlN mixture containing 40 vol % AlN treated with various coupling agents at (a) 40°C and (b) 80°C. Dark squares represent the data for the as-received AlN powder, up-triangles represent the data for the AlN fillers treated with KH-550, circles represent the data for the AlN fillers treated with KH-560, down-triangles represent the data for the AlN fillers treated with KH-792, stars represent the data for the AlN fillers treated with TC-2, and pentagons represent the data for the AlN fillers treated with TC-27.

Fourier transform-infrared (FT-IR) spectrometry was performed on the coupling agents or AlN powder surfaces with a Nicolet 6700 FT-IR.

RESULTS AND DISCUSSION

Viscosity and thermal properties

When a composite adhesive is used for the TIM in a high power device (Fig. 1), a low viscosity is required to reduce the TIM thickness to achieve good coverage on the substrate before the curing stage. Generally, the viscosity of the epoxy-AlN mixture will be raised dramatically at high solid loading by increasing the volume fraction of the AlN fillers,²⁴ so the surface modification of AlN fillers is critical for reducing the viscosity.^{17,22} We tested the effects of five kinds of silane and titanate coupling agents on the viscosity of the epoxy-AlN precuring mixture. Figure 5(a,b) show the viscosity of the precuring mixture with an AlN loading of 40 vol % at

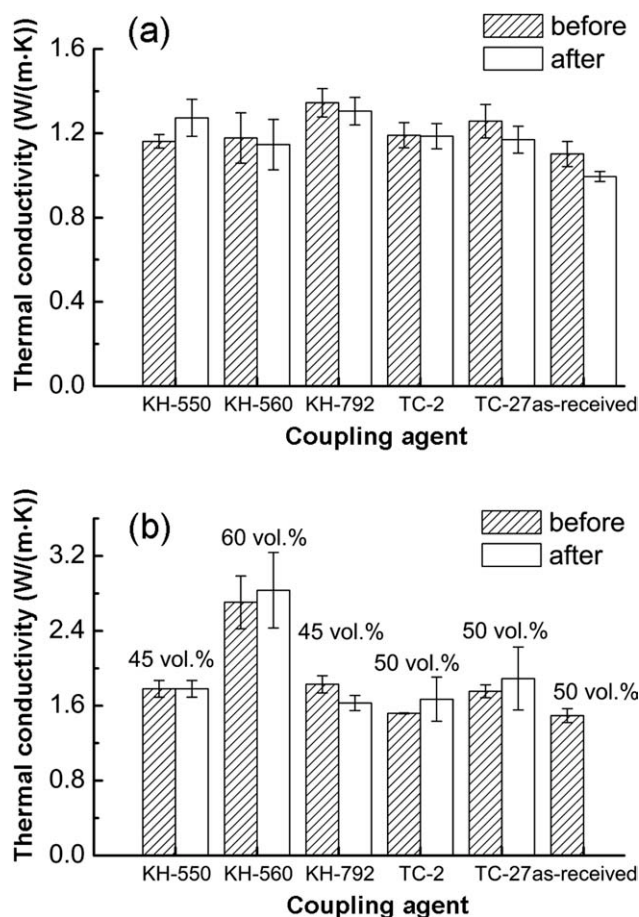


Figure 6 Thermal conductivity of TIM samples with (a) an AlN loading of 40 vol % and (b) maximum volume fractions. The shaded bars and blank bars represent the data before and after 99 thermal shock cycles, respectively. The maximum loading is 45 vol % for KH-550 and KH-792, 50 vol % for TC-2 and TC-7, and 60 vol % for KH-560.

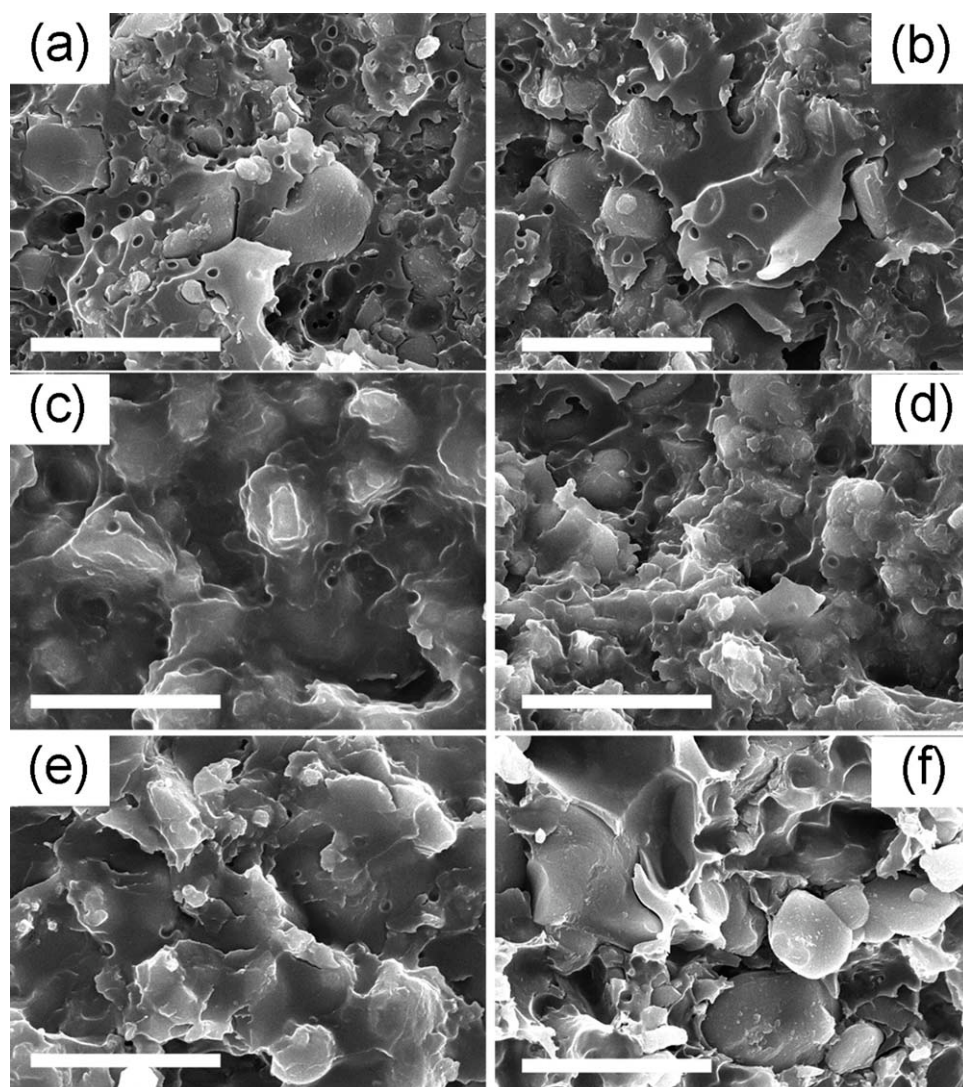


Figure 7 SEM images of the natural fracture surfaces of TIM samples containing 40 vol % AlN powder whose surface was modified with various coupling agents: (a) as-received, (b) KH-550, (c) KH-560, (d) KH-792, (e) TC-2, and (f) TC-27. The scale bar is 10 μm .

40°C and 80°C, respectively. In Figure 5, the quantity of each coupling agent was 5 wt % of the AlN fillers. Only KH-560 reduced the viscosity of the mixture, while others dramatically increased the viscosity, which indicated that KH-560 can improve the compatibility between AlN fillers and epoxy resins and promote a better dispersion of the treated AlN fillers in the epoxy matrix.

The thermal conductivity data of the cured composite thin films with a thickness of $\sim 200 \mu\text{m}$ were summarized in Figure 6. Since the thermal conductivity increases by increasing the volume fraction of AlN fillers,^{17,19,20,24} we focused our study on high AlN loadings. In Figure 6, the shaded bars and the blank bars represent the thermal conductivity before and after 99 thermal shock cycles, respectively, and the measurement was conducted 10 times for each sample. In Figures 6(a) and 7, all the samples have

an AlN loading of 40 vol %. The thermal conductivity of the composite TIM was 1.10 W/(m K) with the as-received AlN fillers, and was raised 10 to 15% by applying various coupling agents on the surface of AlN fillers. Because the coupling agents helped to reduce the gaps between the treated fillers and the epoxy matrix, which was confirmed by the SEM images in Figure 7, the thermal conduction between the fillers and the epoxy improved.

Figure 6(b) then displays the thermal conductivity of the TIM samples with a maximum loading of the AlN fillers. Without surface modification, the maximum loading of the untreated AlN fillers was 50 vol % because the viscosity of the precuring mixture at a higher loading was too large to fabricate a continuous thin film. Because KH-560 reduced the viscosity of the epoxy-AlN composite, we were able to fill AlN fillers into the epoxy up to 60 vol % and form a

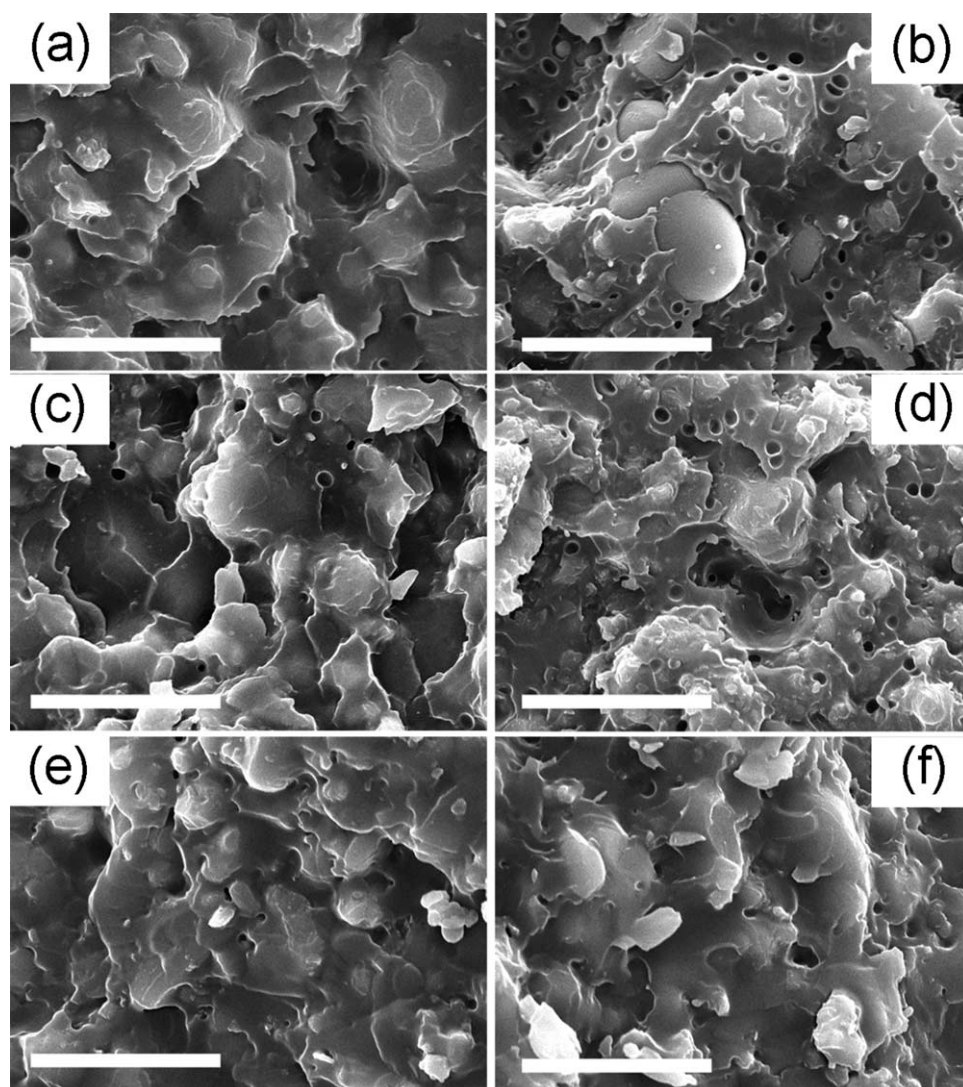


Figure 8 SEM images of the natural fracture surfaces of TIM samples containing (a) 50 vol % as-received AlN fillers, (b) 45 vol % AlN fillers treated with KH-550, (c) 60 vol % AlN fillers treated with KH-560, (d) 45 vol % AlN fillers treated with KH-792, (e) 50 vol % AlN fillers treated with TC-2, and (f) 50 vol % AlN fillers treated with TC-27. The scale bar is 10 μm .

continuous TIM sample with a proper thickness. KH-550 and KH-792 dramatically increased the viscosity (shown in Fig. 5), thus reducing the maximum loading of the AlN fillers to 45 vol % for these two coupling agents. The thermal conductivity of the sample containing 60 vol % AlN fillers treated with KH-560 increased to 2.70 W/(m K), which was approximately 13 times higher than that of the pure epoxy resin at 0.21 W/(m K). KH-560 proved to be a very effective coupling agent for enhancing the filler loading, which in turn increased the thermal conductivity of the composite material. For other coupling agents, the maximum filler loading was not increased, thus limiting the enhancement of the thermal conductivity. Figure 8 shows the natural fracture surfaces of the TIM samples with a maximum volume fraction. Generally, the samples with KH-

560 have a smoother interface between the fillers and the epoxy matrix than the samples using other kinds of coupling agents.

The data in Figure 6 also shows that the thermal conductivity of the composite thin films does not degrade after 99 thermal shock cycles (-25 to 80°C), which means that the TIM made with an epoxy-AlN composite adhesive was suitable for the applications in high power devices.

We also varied the amount of KH-560 and investigated its effects on viscosity and thermal conductivity. The viscosity of the precuring mixture and the thermal conductivity of the composites containing 40 vol % AlN fillers treated with 1 wt %, 3 wt %, and 5 wt % KH-560 are summarized in Figures 9 and 10, respectively. The viscosity of the mixture with KH-560 was one order of magnitude less than that of the

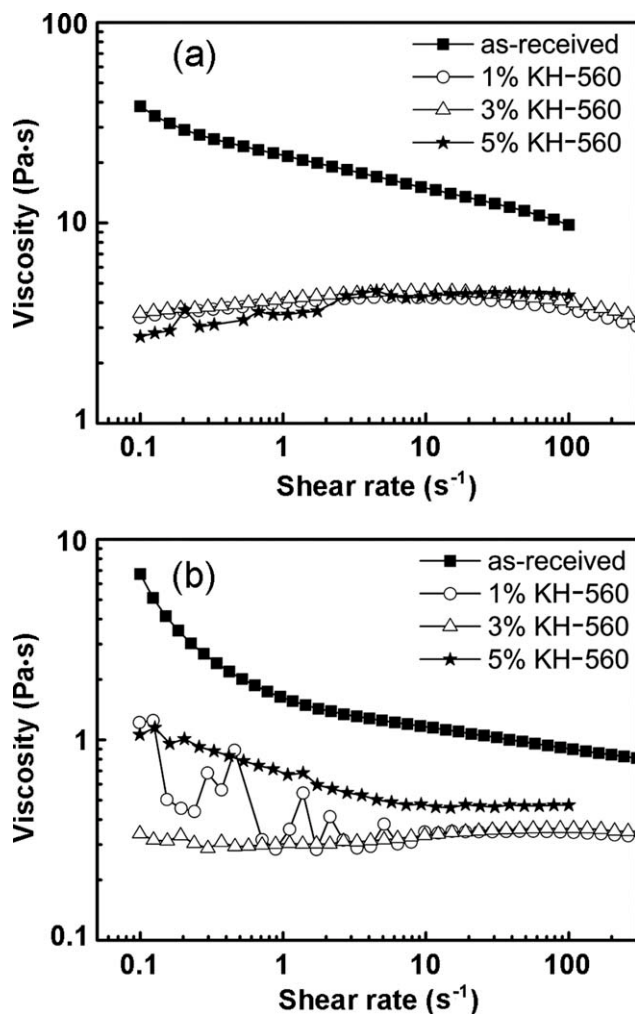


Figure 9 Viscosity of epoxy-AIN mixture containing 40 vol % AIN fillers treated with KH-560 at (a) 40°C and (b) 80°C. Dark squares represent the data for the as-received AIN powder, circles represent the data for the AIN fillers treated with 1 wt % KH-560, up-triangles represent the data for the AIN fillers treated with 3 wt % KH-560, and stars represent the data for the AIN fillers treated with 5 wt % KH-560.

mixture without KH-560, and the measured data displayed no visible correlations with increasing KH-560. For these tests, 1 wt % KH-560 gave the highest thermal conductivity. There were no obvious trends for the viscosity and thermal conductivity as the amount of KH-560 was varied.

Insulating properties

The dielectric strength of pure epoxy was measured to be 11.0 kV/mm. Figure 11(a,b) outline the dielectric strength data for the samples with an AIN volume fraction of 40% and with maximum loadings, respectively. The dielectric strength maintained at 10 to 11 kV/mm after AIN fillers were added and displayed no obvious degradation. After 99 thermal

shock cycles, the reduction of the dielectric strength was less than 10% for most of the samples. A dielectric strength of 10 kV/mm is large enough for the applications in power devices such as IGBT. If a larger dielectric strength is needed, other curing agents such as methyl-hexahydrophthalic anhydride (MHHPA) could be used.²⁵

FT-IR analysis

Figure 12(a,b) show the FT-IR spectra of pure coupling agents and AIN powder treated with the coupling agents, respectively. For the AIN powder treated with KH-560, TC-2, and TC-27, their spectra display some characteristic peaks of these coupling agents. The peaks between 2840 cm^{-1} and 3000 cm^{-1} correspond to the C-H stretching vibrations, which are the evidence of the presence of these coupling agents.^{22,26} In addition, for KH-560, the peaks that represent the Si-(CH₂)₃- and Si-O-CH₃ vibration can be clearly seen at 1194 cm^{-1} and 1080 cm^{-1} ,²⁷ respectively; for TC-2, the peaks indicating the C-H asymmetrical bending vibration in CH₃ or the -CH₂-scissoring vibration and the P=O stretching vibration can be seen at 1460 cm^{-1} and 1150 to 1220 cm^{-1} , respectively; for TC-27, the peaks indicating the benzene ring's bending vibration, the C-H asymmetrical bending vibration in CH₃ or the -CH₂-scissoring vibration, and the P=O stretching vibration can be seen at 1510 cm^{-1} , 1460 cm^{-1} , and 1150 to 1250 cm^{-1} , respectively.^{26,28} However, there is no characteristic peak in the FT-IR spectra for the AIN powder treated with KH-550 and KH-792, which indicates that the coating of KH-550 and KH-792 on the surface of the AIN powder is not effective and

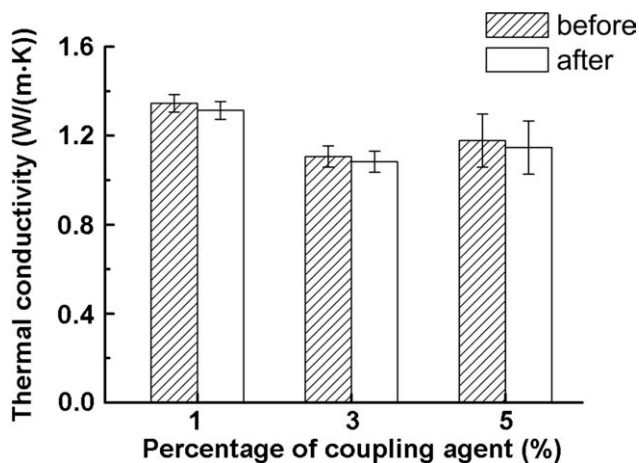


Figure 10 Thermal conductivity of TIM samples containing 40 vol % AIN fillers treated with KH-560 with different weight percentages. The shaded bars and blank bars represent the data before and after 99 thermal shock cycles, respectively.

could be the reason why the viscosity of the precuring mixture with KH-550 and KH-792 is much higher than that of the precuring mixture with other coupling agents (Fig. 5).

Combining the FT-IR spectra, viscosity and thermal conductivity data, it can be concluded that an effective coating of coupling agent on the surface of AlN powder is very critical to the properties of an epoxy-AlN composite material.

CONCLUSIONS

A thermal interface material made with epoxy resin-based adhesive containing aluminum nitride powder was synthesized and characterized. To enhance the thermal conductivity of the TIM, the surface treatment of AlN fillers was explored by applying vari-

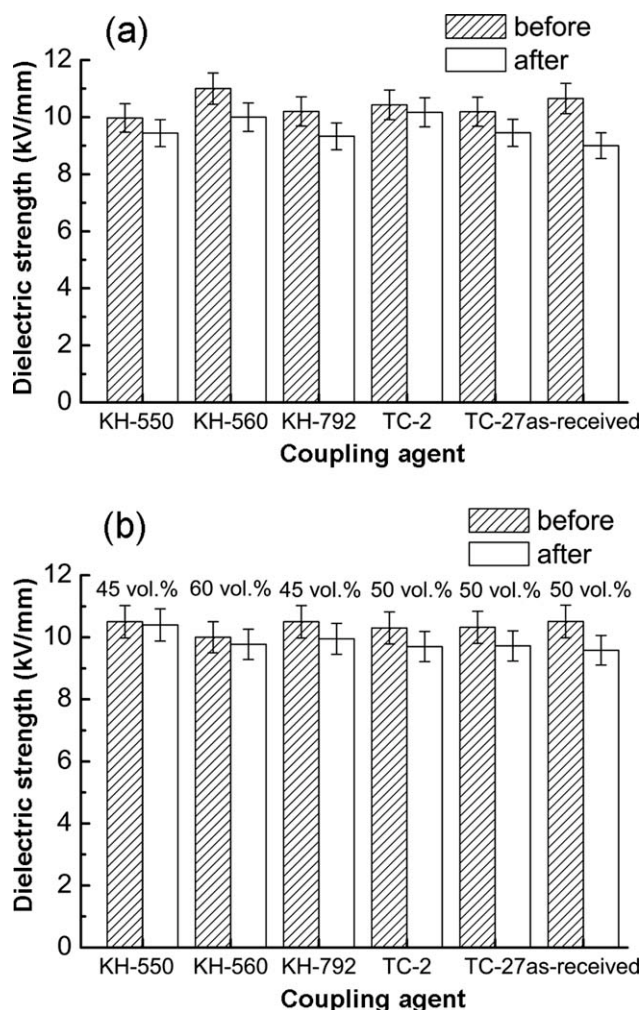


Figure 11 Dielectric strength of TIM samples with (a) an AlN loading of 40 vol % and (b) maximum volume fractions. The shaded bars and blank bars represent the data before and after 99 thermal shock cycles, respectively. The maximum loading is 45 vol % for KH-550 and KH-792, 50 vol % for TC-2 and TC-7, and 60 vol % for KH-560.

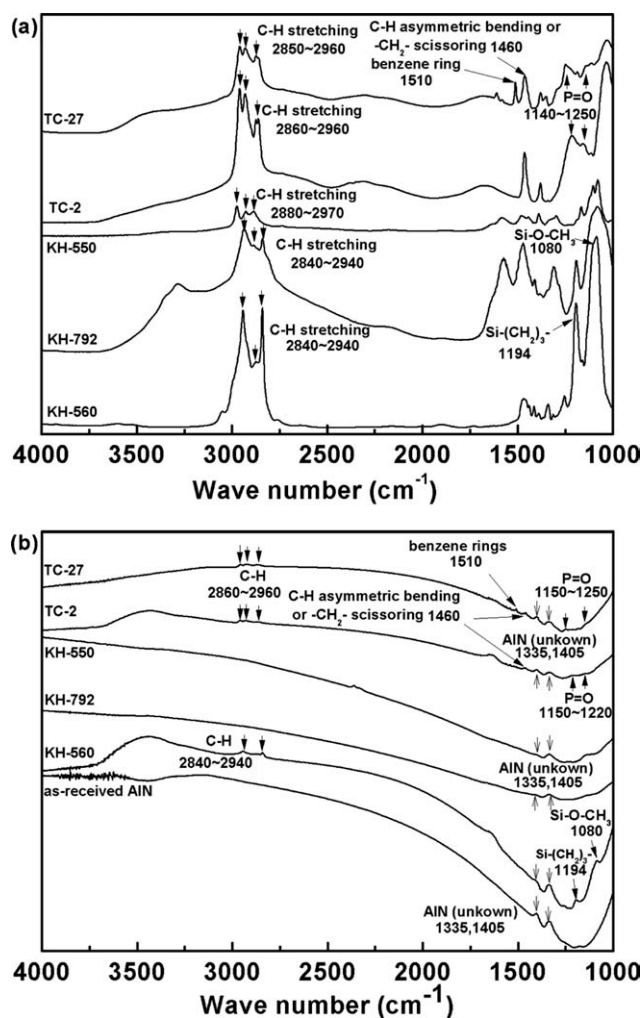


Figure 12 (a) FT-IR spectra of pure coupling agents, and (b) FT-IR spectra of AlN powder treated with 5 wt % coupling agents.

ous coupling agents. Silane KH-560 was confirmed to be a proper coupling agent that reduced the viscosity of the epoxy-AlN mixture. With KH-560, the solid loading was increased to 60 vol % and a thermal conductivity of 2.70 W/(m K) at room temperature was obtained, which was approximately 13 times that of pure epoxy. FT-IR analysis showed that an effective coating of the coupling agent was achieved for silane KH-560, but not for silane KH-550 and KH-792, which means that a coupling agent needs to be carefully selected to improve the dispersion of the AlN fillers in the epoxy resin and the contact between AlN fillers and the epoxy matrix.

For insulating properties, the composite TIM had a dielectric strength of 10 to 11 kV/mm, which meets the requirement for some applications such as IGBT. Additionally, the thermal and insulating properties of the TIM did not degrade after thermal shock testing, which indicates that the epoxy-AlN

composite adhesive can be reliably used as a TIM in high power devices.

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