Materials Research Express

PAPER

Enhanced damping and thermal conductivity of hBN/silicone rubber composites via strong interfacial action

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Keywords: hexagonal boron nitride, damping, thermal conductivity, silicone rubber composites

Abstract
High integration and miniaturization of electronic systems require thermal conductivity and mechanical damping materials. In this study, hexagonal boron nitride (hBN) particles were modified by vinyltrimethoxysilane (V171) and hexadecyltrimethoxysilane (N3116) for comparing, and described as V171-/hBN and N3116-/hBN, respectively. The pristine and surface modified hBN were filled in vinylmethylpolysiloxane (VMQ), and then vulcanized to fabricate hBN/silicone rubber composites. The damping properties of V171-/hBN/silicone rubber composites were significantly higher than that of pristine hBN/silicone rubber composites, while N3116-/hBN/silicone rubber composites were in contrast. The strong interfacial action in V171-/hBN/silicone rubber composites came from the chemical bond by vinyl groups on the surface of V171-/hBN taking part in the vulcanization of VMQ. This strong interfacial action led the deformation of hBN, and the internal friction between layers of hBN significantly increased to dissipate more energy. As a result, V171-/hBN/silicone rubber composites with excellent damping and thermal conductivity as well as good mechanical properties were obtained.

1. Introduction

When electronic systems work, they are often accompanied by heat accumulation, vibration and noise, which reduce the accuracy, reliability, and fatigue life of the devices. Moreover, with the increasing integration and decreasing geometric dimensions of electronic devices, heat accumulation and vibration are rather significant [1]. The polymer composites with outstanding thermal conductivity and damping property can transfer heat and reduce vibration of electronic components [2, 3].

As a typical polymer materials, silicone rubber possesses excellent electrical insulation, high and low temperature resistance and easy process ability, and has been used in fabrication of electronic systems [4]. However, the low thermal conductivity and poor damping properties of pure silicone rubber cannot satisfy some stringent requirements [5]. Recently, carbon materials such as carbon nanotube [6], graphene [7], and graphite [8] have been widely used to improve the thermal conductivity and damping properties of polymers. However, carbon materials with high electrical conductivity will destroy the electrical insulation properties of composites [9]. The hexagonal boron nitride (hBN), well-known insulating filler, has a graphite-like layered structure with outstanding thermal conductivity comparable to graphene. The inner layer is composed of covalently B-N rings, and the interlayer is connected by van der Waals force [10]. Sueyoshi et al [11] prepared composite steels with an iron/hBN sintered core by powder metallurgy, and the internal friction due to the deformation of hBN made a great contribution to the damping of composites. Bustillos et al [12] fabricated photosensitive polymer-based boron nitride nanoplatelets composites via stereolithography three-dimensional printing technique. Due to the interfacial shearing and sliding of boron nitride nanoplatelets, the damping properties of composites enhanced. It is concluded that hBN powder can improve the damping properties of polymer composites.

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Like other inorganic fillers, the surface of hBN is naturally hydrophilic [13], and its compatibility with silicone rubber is poor. Silane coupling agents have been extensively used to reduce the surface hydrophilicity of inorganic fillers and improve the interface interaction and compatibility between polymer matrix and fillers [14]. Gauvin et al. [15] investigated the effect of the interfacial action on the damping properties of carbon fiber/epoxy composites, they found that the damping of composites decreased with increasing the interface adhesion. Other researchers also got similar results [16].

vulcanization of VMQ. A strong interfacial action among hBN and silicon rubber matrix was obtained by forming chemical bond, which accelerated the internal friction of hBN, and increased the damping properties of composites. As a result, hBN/silicon rubber composites with excellent thermal conductivity, mechanical strength as well as good damping properties were obtained.

2. Experimental

2.1. Materials
Poly(methyl-vinyl siloxane rubber (VMQ, 110–2, with density of 0.98 g·cm$^{-3}$) was provided by Dongjue Silicone Co. Ltd., China. Hexagonal boron nitride (hBN, BNH10, with the density of 2.27 g·cm$^{-3}$, $d_{50} = 10 \, \mu$m), was purchased from Eno High-Tech Material Co. Ltd., China. Vinyltrimethoxysilane (V171) and hexadecyltrimethoxysilane (N3116) were received from Qufu Chenguang Chemical Co. Ltd., China. Ethanol, acetic acid, and 2,5-dimethyl-2,5-bis(tert-butyl) peroxide (DBPH) were obtained from Aladdin Industrial Corporation, China. All reagents were used without further purification.

2.2. Surface modification of hBN particles
The silane coupling agent (1.5 wt% of the weight of hBN particles), water (the molar ratio of water/silane coupling agent was 3:1), and ethanol (the same quality as silane coupling agent) were put into a glass vessel, then the mixture was kept at 35 °C for 0.5 h in order to hydrolyze silane coupling agent. In addition, the hBN particles were added to a SHR-10C high-speed mixer (Jiangsu Zhangjiagang Beier Machinery Co. Ltd., China). When the temperature of hBN particles increased to 110 °C, the hydrolyzed silane coupling agent solution was injected homogeneously to the high-speed mixer and stirred for 30 min. The modified hBN particles were washed with absolute ethanol for 3 times, and dried in a vacuum oven at 80 °C for 8 h. The hBN particles modified by N3116 and V171 were demonstrated as N3116–hBN and V171–hBN, respectively.

2.3. Preparation of hBN/silicone rubber composites
The preparation method of hBN/silicone rubber composites was similar to published papers [5]. VMQ was firstly kneaded in an open mill (JTC-7523, Zhanjiang Machinery Factory, China). A definite proportion of pristine hBN particles were then kneaded with masticated VMQ. And then the obtained masterbatch was mixed with DBPH (0.6 wt% of the weight of VMQ). The above blends were compression molded 15 min at 160 °C under 10 MPa, followed by cooling to room temperature. Finally, pristine hBN/silicon rubber composites were obtained by second vulcanized at 200 °C for 4 h. The preparing process of V171–hBN/silicone rubber and N3116–hBN/silicone rubber composites was the same as that of pristine hBN/silicone rubber composites.

2.4. Characterization
The interaction between silane coupling agent and hBN particles was analyzed by X-ray photoelectron spectroscopy (XPS, ESCALAB250, Thermo, USA). Before measurement, pristine and modified hBN particles were wrapped with filter paper, and extracted by Soxhlet extractor with acetone as extraction solvent for 24 h.

Thermo gravimetric analysis was tested according to ASTM E2402–11(2017) by a TG–209–F3 instrument (NETZSCH, Germany) from 35 °C to 900 °C at a temperature rate of 20 °C·min$^{-1}$ under nitrogen atmosphere.

Dynamic mechanical performance was carried out with a DMA 242E instrument (NETZSCH, Germany) in a compressed mode. Samples were heated from −50 °C to 180 °C at a heating rate of 5 °C·min$^{-1}$ and under a frequency of 1 Hz.

The mechanical properties of hBN/silicone rubber composites were measured by electronic universal testing machine (CMT4304, Shenzhen Sans, China). The tensile strength and elongation at break were measured according to ISO 37:2017 by type II dumbbell specimen at a stretching rate of 500 mm·min$^{-1}$ and under a frequency of 1 Hz.

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Thermal conductivity of samples was determined by thermal conductivity apparatus (TC.3000, Xi An Xiatech Electronics Co. Ltd., China) based on a transient hot-wire technique at room temperature. In this
method, a thin metallic wire, as a line heat source and temperature sensor, was suspended between two flat testing samples of 2 mm thickness.

Electrical resistivity was measured on a digital high-resistance-meter PC68 (Shanghai Precision Instrument Manufacture, China) using a sample with a diameter of 60 mm and a thickness of 1 mm.

3. Results and discussion

3.1. The modification of hBN particles

The pristine and modified hBN particles were characterized by XPS analysis, and the results were shown in figure 1. In the spectrum of pristine hBN (figure 1(a)), two strong peaks at 399 eV and 191 eV were attributed to the binding energy of N 1s and B 1s, respectively. In the spectra of V171-hBN and N3116-hBN (figure 1(b) and (c)), a small peak at 103 eV was the binding energy of Si 2p [17], and peaks at 533 eV and 285 eV corresponded to the binding energy of O 1s and C 1s, respectively. Due to the introduction of silane coupling agent residue on the surface of hBN, O 1s and C 1s peaks of V171-hBN and N3116-hBN increased by comparing with pristine hBN.

The C 1s peak of V171-hBN (figure 2(a)) could be deconvoluted into three components at 283.1, 284.0 and 284.9 eV, which were attributed to C–Si, C–H and C=C component, respectively [17]. The C 1s peak of N3116-hBN (figure 2(b)) could be deconvoluted into three components at 283.1, 283.9 and 284.0 eV, which were attributed to C–Si, C=C and C–H component, respectively [17]. This observation indicated that silane coupling agents V171 and N3116 were effectively combined on the surface of hBN fillers.

TGA was further performed to characterize the pristine and modified hBN samples. As depicted in figure 3, the residual mass percent at 900 °C of pristine hBN, V171-hBN and N3116-hBN was 98.54, 98.04 and 97.68%, respectively. The –OH groups on the surface of pristine and modified hBN samples, as well as V171 or N3116 residua on the surface of V171-hBN or N3116-hBN were removed at 900 °C. By comparing the residual mass percent of pristine and modified hBN samples, the grafted organic residua of V171-hBN and N3116-hBN was 0.50% and 0.86%, respectively [18].

3.2. Damping property

The effect of the component at the surface of fillers on the damping properties of the hBN/silicone rubber composites were investigated by DMA at 1Hz. Because the actual working temperature of the composites was much higher than its glass transition temperature (Tg) value, the temperature range chosen in this paper was –50 to 180 °C. The relationship of mechanical damping factor (tanδ) in the DMA graph versus temperature was shown in figure 4. The content of fillers in pristine hBN/silicone rubber, V171-hBN/silicone rubber and N3116-hBN/silicone rubber composites was set the same as 50 vol%. It could be seen that an additional small peak at –7 °C appeared in curve of the pristine hBN/silicone rubber composites. The compatibility between the pristine hBN and the silicone rubber matrix was poor, and some defects and/or voids existed in the interface which
might benefit to the relaxation of silicone rubber small-size units [19]. On the other hand, the compatibility between fillers and matrix was effectively improved by surface modification of hBN using N3116 or V171, and the internal defects and/or voids in composite were reduced. As a result, the V171-hBN/silicone rubber and N3116-hBN/silicone rubber composites had no weak peak at −7 °C.

At most temperature conditions, the damping property of composites increased in sequence of N3116-hBN/silicone rubber, pristine hBN/silicone rubber and V171-hBN/silicone rubber (table 1). In fillers/polymer composites, the dielectric loss (tanδ) was related to molecular movements, viscoelasticity besides the certain defects that contributed towards damping such as dislocations, grain boundaries, phase boundaries and various interfaces [20]. In this paper, the damping property of silicone rubber matrix itself is weak (tanδ, 0.1–0.2), so the damping effect caused by silicone rubber molecular chain motion is weak [21]. The damping properties of the pristine hBN/silicone rubber, V171-hBN/silicone rubber and N3116-hBN/silicone rubber composites might mainly be affected by the friction among fillers and the interaction between the fillers and the matrix. Similar to graphite, hBN was also a filler with sheet structure. In hBN/silicone rubber composites, the layered structure of filler facilitates enhanced damping because of large filler surface area involved. This will contribute to higher energy dissipation because of friction among fillers. Therefore, compared with silicone rubber, the damping properties of hBN/silicone rubber composites are obviously improved [22].

For N3116-hBN/silicone rubber composites, a long alkyl chain (−(CH₂)₁₅CH₃) from N3116 was grafted on the surface of hBN, it is well known that long alkyl chain has good lubricity, and the friction among fillers

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**Figure 2.** C 1s spectra of samples. (a) V171-hBN, (b) N3116-hBN.

**Figure 3.** TGA curves of samples.
decreased. As a result, the dielectric loss of N3116-hBN/silicone rubber composites were lower than that of pristine hBN/silicone rubber composites at large temperature range. On the other hand, the compatibility between fillers and silicone rubber matrix was improved in N3116-hBN/VMQ composites, and damping effect caused by defects such as voids was weakened. It is reported that composites with poor interfacial compatibility sometimes exhibit more damping loss than composites with good interfacial compatibility [23].

For V171-hBN/silicone rubber composites, the vinyl functional groups from V171 were grafted on the surface of hBN, it could take part in the vulcanization reaction of VMQ, and a chemical bond formed in the interface between fillers and silicone rubber matrix which enhanced the interfacial action between fillers and matrix. This strong interfacial action led the deformation of hBN, and the internal friction between layers of hBN significantly increased to dissipate more energy. The mechanism of improving the damping performance of V171-hBN/silicone rubber composites was shown in figure 5. This effect on damping property improving was obviously larger than damping property reduction from interfacial compatibility. As a result, the tanδ of V171-hBN/silicone rubber composites were considerably higher than that of pristine hBN/silicone rubber composites when the temperature was higher than 15 °C, and the temperature range of tanδ > 0.5 of V171-hBN/silicone rubber composites (40 °C–180 °C) was larger than that of pristine hBN/silicone rubber composites (50 °C–147 °C).

Figure 6 shows the curves of tanδ versus temperature of V171-hBN/silicone rubber composites with different filler content. The tanδmax of composites with 50 vol% filler content is slightly higher than that of with 55 vol% filler content, and significantly higher than that of with 45 vol% filler content. This is because with the increase of filler content, the internal friction between the hBN layers increases. Although the filler content of 55 vol% is higher than that of 50 vol%, tanδ of composites slightly reduces when the temperature is higher than 68 °C. This may due to that the filler content of 55 vol% has exceeded the threshold value, the fillers tend to agglomerate, the interfacial action in V171-hBN/silicone rubber reduces, and the internal friction between the hBN layers decreases. In addition, a greater number of polymer chains is restricted in their movement [24].

### 3.3. Mechanical properties

Figure 7 shows the curves of tensile strength and elongation at break of pristine hBN/silicone rubber, V171-hBN/silicone rubber and N3116-hBN/silicone rubber composites with different volume number of fillers. The tensile strength of three composites all increases to a maximum value and subsequently decrease with further
Figure 5. The mechanism of improving the damping performance of V171-hBN/silicone rubber composites.

Figure 6. $\tan \delta$ of V171-hBN/silicone rubber composites vs temperature with different fillers content.

Figure 7. Mechanical properties of composites. (a) Tensile strength, (b) elongation at break.
loading. The tensile strength of V171-hBN/silicone rubber and N3116-hBN/silicone rubber composites is significantly higher than that of pristine hBN/silicone rubber composites. When the fillers content is less than 50 vol%, the tensile strength of N3116-hBN/silicone rubber is higher than that of V171-hBN/silicone rubber, while the result was opposite when the fillers content was higher than 50 vol%. This may due to the different compatibility between V171-hBN or N3116-hBN and matrix. The grafted residue of V171 and N3116 on the hBN surface is 0.50% and 0.86%, respectively (figure 3). When the fillers content is lower than 50 vol%, the compatibility effect from organic residue of modified fillers may dominant, otherwise, the compatibility effect from chemical bond existing between fillers and silicone rubber matrix, which comes from the vinyl functional groups of V171 residue participating the vulcanization reaction of VMQ, become obviously. The elongation at break of V171-hBN/silicone rubber composites is higher than that of N3116-hBN/silicone rubber and pristine hBN/silicone rubber composites at any filler content, it can be concluded that strong interfacial action from chemical bond between fillers and matrix take advantage to elongation at break of composites.

3.4. Thermal conductivity

Figure 8 shows the thermal conductivity of composites with different content of filler. With the increase of hBN volume content, thermal conductive particles contact closely with each other, forming more thermal conductive paths, which leads to the increase of thermal conductivity of hBN/silicone rubber composites. In addition, the thermal conductivity of the composites increases significantly when the content of hBN is higher than 50 vol%. Once the thermal network is formed through fillers, the composite exhibited permeability behavior and the thermal conductivity increased sharply [25]. The V171-hBN/silicone rubber and N3116-hBN/silicone rubber composites exhibit higher thermal conductivity than pristine hBN/silicone rubber composites with the same hBN loading. The thermal conductivity depended not only on the content of filler, but also on the synergistic effect of matrix and filler. In this study, the vinyl functional group on the V171-hBN surface can participate in the VMQ vulcanization reaction process, which not only improved the compatibility but also enhanced the interfacial action. So that the thermally conductive particles could more closely contact each other to form a network or chain-like thermal conduction channel, and the heat energy could be more easily transferred in V171-hBN/silicone rubber composites. Many researchers have also concluded that composites with better interfacial adhesion can possess higher thermal conductivity [26, 27].

The fracture surface morphology (figure 9) of composites with 50 vol% hBN is observed by scanning electron microscopy (SEM). The pristine hBN has obvious agglomeration due to the poor interface compatibility. In contrast, the dispersion of V171-hBN and N3116-hBN in silicone rubber matrix is more homogeneous, and the compatibility between filler and matrix is improved effectively. The results are in good accordance with the mechanical and thermal conductive properties of composites.

3.5. Electrical insulation

Figure 10 shows the curves of volume resistivity of pristine and modified hBN/silicone rubber composites varying with volume fraction of fillers. The results indicate that the volume resistivity of the composites
decreases with the increasing filler content, and the trend is basically the same. Different surface modification methods had little effect on the electrical insulation properties of the modified hBN/silicone rubber composites. All of them were good electrical insulating materials [28].

From figures 7, 8, and 10, when the filler content was fixed at 50 vol%, the thermal conductivity, tensile strength, elongation at break, and volume resistivity of V171-hBN/silicone rubber composites attained 2.51 W/(m·K), 2.64 MPa, 108%, and $2.1 \times 10^{14}$ Ω·cm, respectively.
4. Conclusions

X-ray photoelectron spectroscopy and thermal gravimetric analysis revealed that V171 was successfully grafted onto the surface of hBN. The tan\(\delta_{\text{max}}\) (0.65/143 °C) and the temperature range of tan\(\delta\) > 0.5 (40°C–180°C) of V171-hBN/silicone rubber composites (hBN 50 vol%) were considerably higher than that of pristine hBN/silicone rubber composites (hBN 30 vol%), which were 0.53/86 °C and 50 °C–147 °C, respectively. When the filler content reached 50 vol%, the thermal conductivity, tensile strength, elongation at break, and volume resistivity of V171-hBN/silicone rubber composites attained 2.51 W/(m·K), 2.64 MPa, 108%, and 2.1 \(\times\) 10\(^{14}\)Ω·cm, respectively. The composites had good damping, thermal conductivity, mechanical, and insulation properties, which could lead to significant technological advances in electronic systems.

Acknowledgments

The authors are grateful for the support and funding from Anhui Science and Technology Department, China (17030901076).

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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