Magnetically oriented 3D-boron nitride nanobars enable highly efficient heat dissipation for 3D integrated power packaging

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Research Article

**Keywords:** Boron nitride nanobarbs, Thermal interface materials, Magnetic alignment, Thermal management

**Posted Date:** April 27th, 2023

**DOI:** https://doi.org/10.21203/rs.3.rs-2836879/v1
Abstract

Increasing power density and miniaturization in 3D packaged power electronics demand innovative thermal management. Yet, the thermal performance of electrically insulated packages for power electronics is currently limited by the ultralow thermal conductivity of conventional thermal interface materials (TIMs) and their poor ability of directing heat to heat sink. Herein, we have prepared highly thermally conductive and electrically insulating TIMs composite based on boron nitride nanobars (BNNB). The polar characteristics of B-N bond in the BNNB outer tube wall-derived h-BN nanosheets facilitates the adsorption of magnetic particles. Modulating the arrangement of 3D-BNNB by an external magnetic field improves the thermal conductivity of composite up to 3.3 W m\(^{-1}\) K\(^{-1}\) at a concentration of 40 wt%, 17.8 times higher than the pure epoxy and also exhibiting significant anisotropy. Moreover, the composite shows a high stiffness of 510 MPa and a high resistivity of 27.2 M\(\Omega\)·cm, demonstrating excellently mechanical and electrically insulating characteristics. Infrared thermography results show that the surface temperature of the composite depends on the orientation of BNNB and its interfacial interaction with the epoxy resin. The magnetic field-oriented modulation of 3D-BNNB can offer a promising solution to achieve the efficient thermal management of 3D integrated power packaging.

1. Introduction

Thermally conductive and electrically insulating materials play a significant role in achieving efficient thermal management for high-density integrated electronic devices.\(^1\)–\(^4\) Hot spots resulting from high temperatures in power-intensive areas are the leading causes of chip failure. Thermal interface materials (TIMs) with high thermal conductivity are often used for connections within electronic devices to prevent safety hazards due to inadequate thermal management.\(^5\)–\(^9\) Therefore, heat needs to be directed in a specific direction toward the heat sink for complex packaging environments. Various TIMs with directional heat transfer have been obtained using graphite flakes, carbon nanotubes, and metal fillers.\(^9\) However, the use of conductive materials in high-density packaged devices can be affected by arcing and cause damage to the device. Therefore, there is a need for electrically insulating and highly thermally conductive TIMs that can also regulate the direction of heat transfer.

Boron nitride (BN) has drawn much attention for its high thermal conductivity and great electrical insulation properties among many thermally conductive fillers.\(^10\)–\(^13\) There are two primary forms of boron nitride fillers, including one boron nitride nanosheet with an in-plane thermal conductivity of up to 400 W m\(^{-1}\) K\(^{-1}\). In contrast, the through-plane thermal conductivity is only about 30 W m\(^{-1}\) K\(^{-1}\). Another is boron nitride nanotubes (BNNT) owning an in-plane thermal conductivity of up to 2400 W m\(^{-1}\) K\(^{-1}\). Phonon scattering tends to occur in randomly mixed boron nitride fillers, making the composite anisotropic and less thermally conductive. Therefore, the orientation of boron nitride filler in the composite can be manipulated to enhance its thermal conductivity and achieve directional heat transfer. Various methods have been applied to investigate the orientation effects including hot pressing,\(^14\),\(^15\) type-casting,\(^16\)–\(^18\) freeze-casting,\(^19\),\(^20\) “thermophoresis”,\(^50\) and magnetic fields.\(^21\)–\(^23\),\(^36\) Boron nitride
nanosheets are easily attached by magnetic particles but have a lower thermal conductivity than BNNT. Compared to boron nitride nanosheets, yet the elongated structure of BNNT makes it challenging to control its orientation, so there is less study on the directional regulation of BNNT. Considering that BNNT is more thermally stable and has a longer thermal path, the vertical arrangement of BNNT would obtain TIMs with high thermal conductivity. However, BNNT alone with smooth surface cannot adsorb magnetic particles well to help modulate the orientation, and the nanotube surfaces should be engineered. Following this, the boron nitride nanobarbs (BNNB) manufactured with irregular barbs on the outer surface nanotube demonstrate great potential to reinforce the interaction with matrix materials and magnetic particles.

In this work, a high thermal conductivity composite is synthesized by mixing BNNB adhered with Fe₃O₄ nanoparticles into the epoxy resin and then applying an external magnetic field to manipulate their orientation. The polarity of the irregular barb of BNNB improves the attachment of magnetic particles and facilitates magnetic modulation and association with matrix materials. The thermal conductivity of the vertically oriented samples reached 3.3 W m⁻¹ K⁻¹ when filled at a concentration of 40 wt%. The orientation of BNNB in the composite was characterized by scanning electron microscopy (SEM), and the orientation and chemical structure of the composite were analyzed by X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FT-IR). The temperature changes of the composite during the heating of the heating table were captured by an infrared camera, and the mechanical and electrical properties of the highly oriented composite were also tested for practical applications. The mechanism of thermal conductivity enhancement is explained by a study of orientation and the Maxwell-Garnett effective medium approximation model. The BNNB provides new possibilities for directional heat transfer in high-power-density electronic devices.

2. Results and discussion

BNNB is a structure in which irregular hexagonal boron nitride crystals are formed on the outer surface of BNNT. The h-BN in the outer tube wall interlocks with the matrix material like mechanical barbs, thus optimizing the cross-linking between the materials. The barb maximizes the surface distance and thus weakens the effect of van der Waals forces, making agglomeration less likely. The polarity of the B-N bond allows solid interfacial interactions between BNNB and the matrix material, thus enhancing the mechanical properties of the composite. Figures 1 (a-c) show the morphology of BNNB as observed by SEM. BNNB is intrinsically non-magnetic and tends to be randomly arranged in epoxy resins. To enhance its orderliness, we resorted to its barb characteristic. Superparamagnetic iron oxide is electrostatically adsorbed onto h-BN nanosheets on the outer surface of BNNB under the catalysis of a cationic surfactant. Figs. 1 (d-f) show Fe₃O₄ nanoparticles uniformly distributed on the surface of BNNB, and (g) shows Fe₃O₄@BNNB dispersed in deionized water being attracted by a permanent magnet. The magnetically functionalized Fe₃O₄@BNNB powder is then mixed into a low-viscosity aqueous epoxy resin slurry, which is dispersed uniformly using tip ultrasonication. An external magnetic field of 30–50 mT was applied to both ends of the sample and held for 12 hours to orientate the magnetic BNNB to arrange
itself. The samples are dried in a vacuum at 70°C for 12 hours to remove air bubbles and free water, and the final composite is cured using a heated oven at 120°C for 3 hours. Figure 1(h) shows the fabricated composite block, and Fig. 2 shows the process flow for the fabrication of the composite material.

To comparatively investigate the effect of BNNB orientation on thermal conductivity, we have synthesized not only composites with vertical BNNB distribution (V-$\text{Fe}_3\text{O}_4@\text{BNNB}$) but also samples with horizontal BNNB distribution (H-$\text{Fe}_3\text{O}_4@\text{BNNB}$) and random BNNB distribution without magnetic control (R-$\text{Fe}_3\text{O}_4@\text{BNNB}$). Figures 3(a-c) show schematic diagrams of the Fe$_3$O$_4$@BNNB arrangement directions for the three types of samples, while (d-i) show the brittle fracture and polished surface morphologies of these composites characterized by SEM. We observe an orderly and homogeneous arrangement of BNNBs with magnetic modulation and a disordered arrangement of BNNBs without magnetic modulation. Thus, the action of the magnetic field allows the Fe$_3$O$_4$@BNNB to achieve directional displacements. We have found that BNNBs form a continuous thermal conductivity channel with the sufficient concentrations. SEM does not allow us to know the composition and crystalline phase of the sample, so we turn to the X-ray diffraction techniques. As shown in Fig. 4(a), the diffraction peaks of the composites were measured using X-ray diffraction techniques. For the pure epoxy resin, the diffraction peaks correspond to (002), (100), (102), and (004).$^{24,25,48}$ For the composites with the addition of Fe$_3$O$_4$@BNNB powder, four new diffraction peaks appear, belonging to (220), (311), (511), and (440), respectively.$^{26,27}$ These peaks attest to the presence of BNNB and Fe$_3$O$_4$ nanoparticles in the composite, and these diffraction peaks increase with the mass fraction of Fe$_3$O$_4$@BNNB. As shown in Fig. 4(b), we can learn about the orientation of BNNB in the epoxy resin. The (002) diffraction peak representing the horizontal orientation and the (100) diffraction peak representing the vertical orientation in the three composites were analyzed and compared. The strength of the diffraction peaks can indicate differences in the degree of orientation $\delta$:

$$\delta = \frac{I_{100}}{I_{002} + I_{100}} \times 100\%$$

Where $I_{002}$ and $I_{100}$ is the intensity of the horizontally and vertically oriented diffraction peak, respectively.$^{21,28}$ At a BNNB filling mass fraction of 40 wt%, the vertical orientation of the V-$\text{Fe}_3\text{O}_4@\text{BNNB}$ composite is calculated to be 52%, and the orientation of R-$\text{Fe}_3\text{O}_4@\text{BNNB}$ is 24%. With an orientation of 6.3% for H-$\text{Fe}_3\text{O}_4@\text{BNNB}$, the magnetically regulated vertical orientation is approximately twice as high as the random arrangement. The intensity of the X-ray diffraction peaks is mainly influenced by the h-BN nanosheets on the outer surface of the BNNB because the nanosheets are affixed to the outer wall in the same direction as the BNNB. Therefore, it can be shown that there are more boron nitride nanobars arranged vertically after the vertical magnetic modulation. The orientation of R-$\text{Fe}_3\text{O}_4@\text{BNNB}$ is approximately four times that of H-$\text{Fe}_3\text{O}_4@\text{BNNB}$, suggesting that after horizontal magnetic modulation, and only a few BNNBs remain vertically aligned. As shown in Fig. 4(c-d), the Fourier transform infrared
spectroscopy (FT-IR) analysis of the V-Fe$_3$O$_4$@BNNB composite shows new characteristic peaks at 1374 cm$^{-1}$, 807 cm$^{-1}$ and 604 cm$^{-1}$ compared to the pure epoxy. The characteristic peaks locating at 1374 cm$^{-1}$ and 807 cm$^{-1}$ are B-N and B-N-B stretching vibrations, respectively, and the characteristic peak of 604 cm$^{-1}$ is Fe-O.$^{29,30}$ These IR characteristic peaks demonstrate compositions such as boron nitride and iron tetroxide, and the peaks are positively correlated with Fe$_3$O$_4$@BNNB mass fraction.

The thermal conductivities of those composites are then measured following the laser flash method. The calculation equation is $\kappa = \alpha C_p \rho$. Where $\kappa$ is the thermal conductivity, $\alpha$ is the thermal diffusion coefficient, $C_p$ is the specific heat capacity, and $\rho$ is the density of the composite, respectively.$^{41,42}$ As shown in the histogram of Fig. 5(a), it is clear that the thermal conductivity of the composites with BNNB arranged vertically is higher than that of the random distribution and much greater than that of the horizontal arrangement. The thermal conductivity of R-Fe$_3$O$_4$@BNNB and H-Fe$_3$O$_4$@BNNB composites is only 1.674 W m$^{-1}$ K$^{-1}$ and 0.506 W m$^{-1}$ K$^{-1}$, respectively, even when the BNNB mass fraction reaches to 40 wt%. However, the thermal conductivity of the V-Fe$_3$O$_4$@BNNB composite enlarges to 3.3 W m$^{-1}$ K$^{-1}$. As presented in Fig. 5(b), the thermal conductivity of the composites increases for all three orientations as the mass fraction of nanobars increase from 0.5wt%, 10wt%, 20wt% to 40wt%. Moreover, the increase in thermal conductivity is somewhat more significant for V-Fe$_3$O$_4$@BNNB and less for H-Fe$_3$O$_4$@BNNB. It is observed that when the BNNB mass fraction is less than 20 wt%, the improvement in thermal conductivity with increasing the proportion of filler is significantly smaller than the case over 20 wt%. This is because when there is less filler, it does not create a continuous thermal conductivity channel in the composite. When the filler ratio reaches a specific value, the thermal conductivity of the composite increases substantially, which has been demonstrated in TIMs materials with other types of filler. Figure 5(c) shows the percentage increase in thermal conductivity of BNNB vertically arranged over randomly arranged composites, with V-Fe$_3$O$_4$@BNNB increasing by 59.2%, 73.1%, 79.5%, and 87.6% over R-Fe$_3$O$_4$@BNNB for BNNB filling mass fractions of 0.5wt%, 10wt%, 20wt% and 40wt% respectively. Therefore, it is sufficient to show that the magnetic modulation of the perpendicular alignment contributes significantly to the thermal conductivity enhancement of the composite. The histogram in Fig. 5(c) shows the percentage increase in thermal conductivity of the V-Fe$_3$O$_4$@BNNB and R-Fe$_3$O$_4$@BNNB composites, respectively, relative to the pure epoxy resin. When BNNB is filled to 40 wt%, the R-Fe$_3$O$_4$@BNNB composite has a 9-fold increase in thermal conductivity relative to the pure epoxy, while the V-Fe$_3$O$_4$@BNNB composite has a 17.8-fold increase. The red pentagon in Fig. 5(d) shows the thermal conductivity of the composites obtained in this work. The thermal conductivity is somewhat higher than that obtained in most literature using nanosheets and nanotubes for the same filling concentration.$^{28,29,31,32,43–47}$

To further assess the effect of the degree of orientation of the filled BNNB on the thermal conductivity enhancement, the enhancement efficiency $\eta$ is defined as

$$\eta = \frac{\kappa_v - \kappa_r}{\kappa_r} \times 100\%$$
Where $\kappa_v$ and $\kappa_r$ is represents the thermal conductivity of the BNNBs arranged vertically and randomly in the composite, respectively.\textsuperscript{23} When there is more BNNB in the vertical direction, the thermal conductivity enhancement efficiency $\eta$ is more significant and the thermal conductivity is thus higher. Due to the particular characteristics of the BNNB structure, the more vertical rows there are, the easier it is to create good thermal paths in the long strips.

In all TIMs, the microscopic interfacial thermal resistance of the filler within the material must be considered, similar to the way TIMs are used to improve macroscopic contact thermal resistance. In this study, the filler particles and the substrate are insulated, so the interfacial heat transfer between BNNB and the epoxy resin is mainly via phonons. However, phonon transport at the interface between different materials is prone to scattering, and phonon scattering will affect the thermal conductivity of the composite.\textsuperscript{33, 51} The Maxwell Garnett effective medium approximation can be used to analyze the effect of thermal resistance at the interface between the filler particles and the matrix on the thermal conductivity of the composite\textsuperscript{34, 35}, as determined by

$$K = K_p \left[ \frac{3K_m + 2f (K_p - K_m)}{(3 - f) K_p + K_m f + \frac{R_B K_m K_p f}{H}} \right]$$

Where $R_B$ is the microscopic interfacial thermal resistance between the filler particles and the substrate, $K_p$ is the thermal conductivity of the filler particles, $K_m$ is the thermal conductivity of the substrate, $f$ is the filling concentration, and $H$ is the thickness of the filler particles.\textsuperscript{40} As shown in Fig. 5(e), this barb structure maximizes the surface distance and weakens the Van der Waals forces so that no agglomeration occurs. The polarity of the B-N bond allows the formation of interfacial bonds with strong interfacial interactions between BNNB and the matrix material. This reduces phonon scattering and decreases the microscopic interfacial thermal resistance $R_B$ increasing the thermal conductivity $K$ of the composite. Therefore, the cross-linking between BNNB and the matrix material enhances the thermal conductivity and mechanical properties of the composite.

The mechanical and electrical properties of the composites were further verified in order to make them more suitable for use in 3D chip packaging.\textsuperscript{38, 39} As shown in Fig. 6(a), BNNB arranged vertically at 25°C has a modulus of elasticity of 510 MPa, which is greater than that of the randomly arranged and horizontally arranged composites. The strong interfacial bonding of the BNNB in contact with the epoxy resin results in stronger interactions with each other than the usual boron nitride nanosheets, thus enhancing the mechanical properties of the composite. The vertically aligned composite filler particles are uniformly subjected to deformation stresses so that they are less prone to deformation when used as TIM, yet are more elastic than pure epoxy resins. As shown in Fig. 6(b), the resistivities of the composites
were tested for three concentrations in a vertical row. We found that although the resistivity decreased with increasing BNB concentration, probably due to the increase in attached \( \text{Fe}_3\text{O}_4 \), the resistivity of each material remained large. The resistivity at 10wt\% is 87.6 M\( \Omega \cdot \text{cm} \), and even at 40wt\%, it is still 27.2 M\( \Omega \cdot \text{cm} \). The good electrical insulation properties of the composite material meet the requirements of insulating packages for high power density electronics. To evaluate the thermal performance of several BNNB composites, they were placed on an electrically heated plate and heated continuously. Infrared thermography observes the temperature changes on the composites' surface. In Fig. 6(c), it is evident that the V-\( \text{Fe}_3\text{O}_4 \)@BNNB composite heats up faster, with R-\( \text{Fe}_3\text{O}_4 \)@BNNB being the next fastest and H-\( \text{Fe}_3\text{O}_4 \)@BNNB the slowest. Thus, it can be demonstrated that the thermal conductivity of vertically arranged composites is higher. That heat can pass quickly along the thickness direction and dissipate heat more effectively. Figure 6(d) shows that in less than 10 seconds, the surface temperature of the BNNB vertically lined up sample increased from 25°C to 45°C, approximately 10°C higher than the randomly lined up sample. After some time, the heating rate of the three samples slowed down, and the temperature of the samples tends to be stable after 150s. The V-\( \text{Fe}_3\text{O}_4 \)@BNNB sample has the highest surface temperature, and the H-\( \text{Fe}_3\text{O}_4 \)@BNNB sample has the lowest surface temperature.

3. Conclusion

In summary, by attaching magnetic particles to the nanobarbs and regulating the BNNB orientation with the help of a magnetic field, the thermal conductivity of the epoxy resin is significantly increased. At a BNNB concentration of 40 wt\%, the through-plane thermal conductivity of the vertically aligned nanotubes composite reached 3.3 W m\(^{-1}\) K\(^{-1}\), an increase of 87.6\% over the randomly aligned one and 17.8 times that of the pure epoxy. The higher thermal conductivity of vertical orientation is explained by analyzing XRD patterns to establish a link between BNNB orientation and thermal conductivity enhancement efficiency \( \eta \). In addition, the structural peculiarities of the nanobarbs, h-BN microcrystals form interfacial bonds with the epoxy resin, enhancing cross-linking with the epoxy resin, reducing phonon scattering and lowering the microscopic interfacial thermal resistance, which in turn improves the thermal conductivity of the composite. The modulus of elasticity of the three types of arrangement was also tested, with the V-\( \text{Fe}_3\text{O}_4 \)@BNNB sample having a modulus of 510 MPa. It exhibited better elasticity than epoxy and was less prone to deformation than random and horizontal displacements. The composite filled with BNNB at 40wt\% concentration still has a resistivity of 27.2 M\( \Omega \cdot \text{cm} \), which meets the insulation requirements of TIM. Infrared thermal imaging experiments demonstrate the better thermal performance of the V-\( \text{Fe}_3\text{O}_4 \)@BNNB composite in practical applications. This work provides a cost-effective method of preparing a new type of TIM with high thermal conductivity, offering a new option for the thermal management of packaging for advanced electronics.

4. Experimental section

Materials
The oriented epoxy composites were prepared using BNNB supplied by BNNano. BNNB (average length 20 µm, internal nanotube diameter 60 nm, h-BN Barb size 80 nm, purity > 90%) was purchased from Dalian Yibang Technology Company Limited, China, which is the sole distributor of BNNano in China. The epoxy resin is a low-viscosity bisphenol F type (EPON-862) purchased from Shenzhen Yongxin Plastic Chemical Company Limited, China. Superparamagnetic nano Fe₃O₄ (20nm) purchased from Andi Metal Materials Company Limited, China.

**Preparation of Fe₃O₄@BNNB**

1g BNNB was added to 50 ml of deionized water and mechanically stirred for 1h to form a homogeneous dispersion. 0.5g of Fe₃O₄ nanoparticles coated with cationic surfactant was added to 50 ml of deionized water. The tip was ultrasonically dispersed for 10 min, and the two parts of the solution were mixed. They mechanically stirred at 90°C for 1h, and the tip was ultrasonically dispersed for 5 min to ensure strong adsorption of Fe³⁺ by B-N polarity. The product was repeatedly washed with deionized water to remove the supernatant and the residual Fe₃O₄ nanoparticles at the bottom. The product was dried under vacuum at 70°C for 8h to obtain Fe₃O₄@BNNB powder.

**Preparation of composite materials**: The epoxy resin EPON-862 was first mixed with deionized water at a volume ratio of 1:5, then a certain amount of Fe₃O₄@BNNB powder according to the preparation concentration requirement, and the curing agent and dispersant were added and stirred mechanically for 3h first. The tips were sonicated for 10min in a water bath at 70°C to obtain a mixture solution. The mixture was then poured into a cubic silica gel grinder with 20mm sides. The applied magnetic field is fixed at both ends of the mold to regulate the orientation of the Fe₃O₄@BNNB. They are dried in a vacuum at 70°C for 12h when the free water evaporates, and the resin cross-linking and magnetic field effects occur simultaneously. The samples are then cured at 120°C for 3h to obtain the desired composite.

**Characterization experiments**

The cross-sectional morphology of both Fe₃O₄@BNNB particles and composites were observed by scanning electron microscopy (SEM, Apreo 2, USA). The orientation of the composite was analyzed using an X-ray diffractometer (XRD, Rigaku Ultima IV, Japan) with Cu Kα radiation (λ = 1.54056 Å). The compositions of the composites were analyzed by Fourier Transform Infrared Spectroscopy (Nicolet iS50 FTIR, USA). The thermal diffusion coefficient through the surface of the composite was measured using a laser flash thermal conductivity meter (LFA 467, Germany). The specific heat capacity of the composites was measured using a differential scanning calorimeter (DSC25, TA, USA). The modulus of elasticity of the composite is measured using a dynamic thermomechanical analyzer (DMA Q800, TA, USA). The thermal imaging camera (FLIR-T7250, Sweden) was used to observe temperature changes on the surface of the composite to verify its ability to dissipate heat.

**Declarations**
Supporting Information

The Supporting Information is available free of charge on the Springer nature Publications website at XXX.

Author Contribution

Jian Wang and Jia-Yue Yang wrote the main manuscript text and prepared figures 1-6. Chao Yang, Dezhi Ma, Mowen Zhang, Zhiqiang Li and Xing Li participated in the investigation and experiment. Zhiyuan He, Zhiwei Fu and Linhua Liu are involved in project management. All authors reviewed the manuscript.

Funding

The authors are thankful to the National Key R&D Program of China Grant number 2020YFB2008900, the Key Realm R&D Program of Guang Dong Province under Grants (2020B010173001), and the National Natural Science Foundation of China (Grant no. 52076123).

The authors declare no competing interests.

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**Figures**

(a-c) show the original morphology of BNNB and the surface morphology of h-BN microcrystals, (d-f) show the morphology of Fe₃O₄@BNNB and the surface attachment of nano-Fe₃O₄ to h-BN microcrystals, (g) shows Fe₃O₄@BNNB being attracted to permanent magnets in deionized water, (h) shows the synthesized composite block.
Figure 2

shows the process flow for the preparation of the composite.
Figure 3

(a-c) Schematic diagrams of the vertical, random, and horizontal alignment of Fe$_3$O$_4$@BNNB, respectively. (d, g), (e, h) and (f, i) are the morphologies of the V-Fe$_3$O$_4$@BNNB, R-Fe$_3$O$_4$@BNNB and H-Fe$_3$O$_4$@BNNB composites as observed by SEM.
Figure 4

(a) shows the difference in the XRD patterns of Random-BNNB and Fe$_3$O$_4$@BNNB. (b) shows the effect of the (002) and (100) diffraction peaks in the XRD patterns of V-Fe$_3$O$_4$@BNNB, R-Fe$_3$O$_4$@BNNB, and H-Fe$_3$O$_4$@BNNB on the degree of orientation. (c) shows a comparison of FT-IR spectra of pure epoxy resin and V-Fe$_3$O$_4$@BNNB composites with different concentrations. (d) shows 3D FT-IR waterfall plots of pure epoxy resin and V-Fe$_3$O$_4$@BNNB composites with different concentrations.
Figure 5

(a) Comparison of the thermal conductivity of the three composites. (b) Change trend of thermal conductivity of the three composites with BNNB filling concentration. (c) Line and histogram of the percentage increase in thermal conductivity. (d) Comparison of the thermal conductivity of the composites for this work with other literature. (e) shows simulations of interfacial bonding interactions and phonon transport at the microscopic interface between h-BN microcrystals of BNNB and epoxy resin.
Figure 6

(a) Modulus of elasticity of the composites for the three BNNB arrangements. (b) shows the resistivity of the V-Fe$_3$O$_4$@BNNB composite. (c) shows thermal images of the surface of three composites at different moments in time. (d) shows the surface temperature variation curves for the three composites.

Supplementary Files

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