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Preparation and Thermal Conductivity of BN/Al₂O₃ Composite Powder and Its EP-based Composites

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Abstract: Several boron nitride (BN)/aluminum oxide (Al_2O_3) composite powders were synthesized by liquid-solid phase co-mixture method. BN was modified by the freeze-thaw method and surface modification. The precursor and as-sintered BN/Al₂O₃ composite powders were obtained by changing the type of surface modification materials and the molar ratios. Additionally, the thermal conductivity of the epoxy resin (EP) -based BN/Al₂O₃ composite materials prepared by mechanical mixture was measured and analyzed. Results show that the dispersion and interfacial compatibility of BN after freeze-thaw process are obviously better than those without freeze-thaw process. The modification effect of dopamine on BN is better than that of polyethylene glycol. When the dopamine is used as the surface modification material and the molar ratio of BN to Al(NO₃)₃ is 1:1, BN powder at micro-level with uniform nano-Al₂O₃ deposition can be obtained, namely the BN/Al₂O₃ micro/nano-composite powder. The thermal conductivity coefficient of this EP-based BN/Al₂O₃ composite reaches 0.62 W·m⁻¹·K⁻¹, which is 3 times and 1.5 times higher than that of the pure EP composite and EP-based composite material prepared by pure micro-sized BN powder, respectively. The deposited nano-Al₂O₃ on BN surface can form multiple thermal conduction paths, thereby improving the thermal conductivity of the EP-based BN/Al₂O₃ composite.

Key words: BN/Al₂O₃; composite powder; thermal conductivity property

High-integration, high-power, and miniaturized electronic components are rapidly developing in the modern electronics, electricity, and light-emitting diode fields. With the reduction in size and volume of electronic components as well as the continuous increase in density, the generated heat is greatly increased, which has an inevitable impact on the working stability and reliability of electronic components, even decreasing the service life^[1-2]. Therefore, improving the heat dissipation performance of electronic components is crucial to solve this problem. High thermal conductivity insulating composite materials are widely used in the electrical industry due to their excellent thermal conductivity, fine insulation performance, low cost, and easy industrial production^[3]. Thus, the high thermal conductivity insulating composite materials attract much attention and have been widely researched to improve the heat dissipation and reliability of electronic components^[4-5].

Polymers have the advantages of electrical insulation, high flexibility, small mass, low cost, excellent mechanical properties, and good processibility, and they are widely used in various industrial fields^[6]. However, the amorphous arrangement and vibration of polymer molecular chains can reduce their thermal conductivity. Most polymers are thermal insulators or the ones with poor thermal conductivity of 0.1-0.5 $W \cdot m^{-1} \cdot K^{-1}$ [7-8]. Instead of designing a new polymer of intrinsically high thermal conductivity, filling the polymer matrix by thermally conductive fillers is an effective method to improve the material thermal conductivity^[9]. Common fillers include the alumina (Al₂O₃)^[10-11], aluminum nitride (AlN)^[12], boron nitride (BN)^[13], silicon nitride $(Si_3N_4)^{[14]}$, silicon carbide (SiC)^[15], graphene^[16], and carbon nanotubes^[17]. Ceramic-based fillers have not only good thermal conductivity but also excellent electrical insulation properties. They can improve the ther-

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mal conductivity while maintaining the electrical insulation properties, thereby presenting great potential in the field of electronic packaging. Due to their excellent electrical insulation, stable chemical properties, and low price, ceramicbased fillers are considered as the high-quality filler to prepare thermally conductive and electrically insulating composite materials, and they are usually used as the packaging substrate material in the electronic packaging field. Hexagonal boron nitride has graphite-like layered structure, excellent electrical insulation performance, good mechanical properties, high thermal conductivity, high thermal shock resistance, low dielectric properties, high temperature resistance, and low density. Therefore, it is an ideal ceramic-based thermally conductive filler. As reported in Ref. [18-19], the thermal conductivity of composite materials with two or more fillers is better than that of the composite materials with single-filler. The composite filler can form effective heat conduction paths in the matrix. Moreover, the chemical surface modification treatment of inorganic fillers can also reduce the thermal resistance of the filler-polymer interface, and improve the dispersion and interfacial compatibility of filler particles in the polymer matrix, therefore effectively improving the thermal conductivity^[20].

Bian et al^[21] prepared the epoxy resin (EP) composites filled by dopamine (DA)-modified micro-BN and KH550-modified nano-Al₂O₃ binary filler, and studied the thermal conductivity, dielectric loss, and high-frequency breakdown strength of the composites. When BN content is 22.5wt% and Al₂O₃ content is 7.5wt%, the thermal conductivity of the composites can reach 1.182 W·m⁻¹·K⁻¹, which is about 700% higher than that of the pure EP. Ren et al^[22] prepared the graphene and boron nitride hybrid nanoparticles (GBN) by hydrothermal method, and studied the thermal conductivity and dielectric properties of GBN-containing composites. It is found that the thermal conductivity of GBN/epoxy composites is significantly improved due to the GBN addition.

The h-BN with layered structure belongs to the hexagonal crystal system. The layers are bonded to each other by Van der Waals force. As a result, h-BN is prone to agglomerate in the matrix^[23]. Therefore, two types of surface modifiers were selected in this research. DA is a catechol natural substance containing hydroxyl, amino, and catechol on the surface. Under certain conditions, the substance of polydopamine (PDA) presents strong adhesion^[24]. Polyethylene glycol (PEG) is a non-ionic macromolecule surfactant, which is usually adsorbed on the particle surface by steric hindrance stabilization mechanism, forming micelles and causing repulsion between particles. Thus, the dispersion of the material is improved^[25]. Besides, it is reported that the dispersion of BN and the compatibility with the matrix can be improved by mechanical ball milling^[26-27]. Therefore, h-BN modified by DA, PEG, or Al₂O₂ was prepared by ball milling process in this research.

In this research, the BN/Al₂O₃ composite powder and its EPbased composites were prepared and studied. The BN was treated by freeze-thaw process and chemically modified by different surface modifiers. The BN/Al₂O₃ composite powders were prepared with different molar ratios through the liquidsolid phase mixture method. The microstructure, morphology, and phase were characterized by optical microscope (OM), X-ray diffractometer (XRD), or scanning electron microscope (SEM). The thermal conductivity and mechanism of composites were studied and analyzed.

1 Experiment

The materials used in this research were as follows: micronsized hexagonal boron nitride (BN, Zibo Jingyi Ceramics Technology Co., Ltd) with layered structure and anisotropy properties which are similar to those of graphite; aluminum nitrate (Al(NO₃)₃, Tianjin Damao Chemical Reagent Factory), which is a white substance and soluble in water; citric acid ($C_6H_8O_7$, Tianjin Damao Chemical Reagent Factory); sodium hydroxide (NaOH); hydrochloric acid (Tianjin Damao Chemical Reagent Factory); dopamine hydrochloride (Hubei Maoerwo Biomedical Co., Ltd); polyethylene glycol 6000 (PEG, Fuchen Chemical Reagent Co., Ltd); trismetylaminomethane (Amresco Co., Ltd); bisphenol-A EP (Epikote-828 EL, 186-190g/eq); methyl-tetrahydrophthalic anhydride curing agent; graphite spray (Tianjin Damao Chemical Reagent Factory).

The synthesis of composite materials was divided into two steps: the synthesis of BN/Al_2O_3 composite powder and the synthesis of EP-based BN/Al_2O_3 composites.

(1) Synthesis of BN/Al₂O₃ composite powders

The BN/Al₂O₃ composite powder was synthesized by the solid-liquid phase mixture method.

Step 1: BN was treated by freeze-thaw method. BN was frozen at -20 °C for 24 h and then thawed for 12 h. This process was repeated for 10 times. BN treated by freeze-thaw method and BN without freeze-thaw treatment were used for comparison, and they were named as BN# and BN, respectively.

Step 2: BN and BN# were then modified by DA or PEG, and the resultant products were named as DA@BN, DA@BN#, PEG@BN, and PEG@BN#.

Firstly, 10vol% dilute hydrochloric acid solution was dropped into the 0.01 mol/L trismetyl-aminomethane solution, and the pH value was adjusted to about 8.5 to obtain the Tris-HCI solution. Then, 54 mL Tris-HCI solution, 6 mL ethanol, and 0.03 g dopamine hydrochloride were added to the beaker and stirred for 1 h to obtain the DA solution. Finally, 1.24 g BN or BN# was added to the DA solution for 24 h to obtain the DA@BN or DA@BN#, respectively.

Besides, 1.68 g PEG was dissolved in 100 mL deionized water and stirred for 1 h. Then, 1.24 g BN or BN# powder was added into the mixture. The obtained solution was stirred for 1 h and ultrasonically dispersed for 3 h to obtain PEG@BN and PEG@BN#.

Step 3: BN/AlO(OH) powder, namely the precursor of BN/ Al₂O₃ composite powder, was prepared. Firstly, 20 mL citric acid (2.5 mol/L) and 50 mL NaOH solution (10 mol/L) were added into 100 mL Al(NO₃)₃ solution (1 mol/L). Then, the mixed solution was stirred evenly to obtain AlO(OH) solution. Afterwards, the BN, BN#, DA@BN, DA@BN#, PEG@BN, and PEG@BN# solutions were separately added to the AlO(OH) solution based on certain molar ratios of BN to Al(NO_3)₃. The solution was stirred for 1 h and mechanically milled for 6 h. Finally, different BN/AlO(OH) powders were obtained after several centrifugation treatments followed by cleaning and drying for 48 h.

Step 4: the obtained BN/AlO(OH) powders were sintered at 1200 °C for 2 h in argon atmosphere to prepare the BN/Al₂O₃ powders.

A series of BN/Al_2O_3 powders were prepared at the molar ratio of BN to $Al(NO_3)_3$ of 1:1 according to the abovementioned processes. Additionally, the experiments with molar ratio of BN to $Al(NO_3)_3$ of 1:0.5 and 1:2 were also conducted to determine the optimal preparation parameters.

The abovementioned precursors were named as DA@xBN/AlO(OH), PEG@xBN/AlO(OH), DA@xBN#/AlO(OH), and PEG@xBN#/AlO(OH). The sintered composite powders were named as DA@xBN/Al₂O₃, PEG@xBN/Al₂O₃, DA@xBN#/Al₂O₃, and PEG@xBN#/Al₂O₃. When x=2, 1, and 0.5, the molar ratio of BN to Al(NO₃)₃ was 1:0.5, 1:1, and 1:2, respectively. Normally, without specific instruction, the discussed products are referred to the ones with molar ratio of 1:1.

(2) EP-based BN/Al₂O₃ composites

According to Ref. [28], the thermal conductivity of the material can be improved by appropriate addition of thermal conductive fillers. When the filler content is more than 30wt%, the mechanical properties of the material decrease significantly. Therefore, 23wt% BN/Al₂O₃ fillers were used in this research.

The DA@BN#/Al₂O₃ powder was filled into EP with the methyl-tetrahydrophthalic anhydride curing agent, then forming the EP-based BN/Al₂O₃ composite. The content of DA@BN#/Al₂O₃ filler was 23wt%, and the mass ratio of EP to methyl-tetrahydrophthalic anhydride curing agent was 3:1. The specimen diameter was 12.5 ± 2 mm and the thickness was about 2.5 mm. The upper and lower surfaces of specimen were sprayed by carbon to ensure the electrical conductivity of the specimen.

The as-prepared composites with molar ratio of BN to $Al(NO_3)_3$ of 1:0.5, 1:1, and 1:2 were named as 2BNAl/EP, 1BNAl/EP, and 0.5BNAl/EP, respectively. EP-based BN composites were also prepared and were named as BN/EP.

XRD analyzer (XRD-7000, Shimadzu, Japan) was used to analyze the phase constitutes with scanning steps from 10° to 90°. The observation of microstructure and morphology of the specimens was conducted by SEM (JEOL). The thermal conductivity of the composites was measured by German NETZSCH LFA467 flash thermal conductivity meter through the laser flash technique, where the heat was transferred from the upper surface to the lower surface of the specimens.

2 Results and Discussion

2.1 DA@BN/AlO(OH) and PEG@BN/AlO(OH) powders

XRD patterns of different BN/AlO(OH) powders are shown in Fig.1. It can be seen that the diffraction peaks of AlO(OH) are at 14.5° , 28.1° , 38.3° , and 49° for the PEG@2BN/



Fig.1 XRD patterns of different BN/AlO(OH) powders

AlO(OH) powder, indicating the (020), (120), (031), and (051) crystal planes, respectively. Meanwhile, the characteristic diffraction peaks at 26.7° , 41.6° , and 50.1° correspond to the (002), (100), and (102) crystal planes, respectively.

For the DA@BN/AlO(OH) powder, it can be seen that the positions of diffraction peak hardly change, but their diffraction intensity is decreased with increasing the Al(NO₃)₃ content. The diffraction peak intensity of BN/AlO(OH) powder is further decreased by the DA-modification, compared with that after PEG-modification. An organic layer is formed on BN surface after DA- or PEG-modification, which reduces the intensity of diffraction peaks. It is found that the diffraction peak at $2\theta > 60^{\circ}$ almost disappears with increasing the content of Al(NO₃)₃. Thus, it can be inferred that the modification effect on BN for the BN/AlO(OH) powder becomes worse with increasing the Al(NO₃)₃ content.

The morphologies of BN/AlO(OH) powders with different modifiers (PEG, DA) are shown in Fig. 2. Combined with Fig. 1, it can be seen that the AlO(OH) is barely attached



Fig.2 SEM morphologies of PEG@BN/AlO(OH) powder (a) and DA@BN/AlO(OH) powder (b)

to BN in the PEG@BN/AlO(OH) powder (Fig. 2a), and some small AlO(OH) exists on DA@BN surface, as shown in Fig. 2b. Therefore, the effect of AlO(OH) deposition on BN surface by DA-modification is better than that by PEGmodification.

2.2 DA@BN#/AIO(OH) powder

It is noticed that the BN powder is a layered material and has clustered agglomeration structure, and it is difficult to achieve uniform deposition of AlO(OH) on BN powder surface. Therefore, the freeze-thaw method was used to pretreat BN powder before the preparation of BN/AlO(OH) powder.

To investigate the effect of freeze-thaw treatment, XRD patterns of the DA@xBN#/AlO(OH) powders are shown in Fig.3. When the molar ratio of BN to Al(NO₃)₃ is 1:1, the BN peaks at 26.7°, 41.6°, and 50.1° are more obvious, compared with those at other molar ratios. When the molar ratio of BN to Al(NO₃)₃ is 1:0.5 and 1:2, the intensity of BN peaks is decreased. Furthermore, it can be seen that the AlO(OH) peaks appear at 14.5°, 28.1°, 38.3°, and 49°. With increasing the Al(NO₃)₃ content, the diffraction peaks of AlO(OH) slightly change.

As shown in Fig. 1, the AlO(OH) peak intensity increases significantly, indicating that the deposition of AlO(OH) on BN after freeze-thaw treatment is greatly improved. Moreover, more active sites are provided, which facilitates the AlO(OH) deposition on BN surface.

SEM morphologies of DA@xBN#/AlO(OH) powders are



Fig.3 XRD patterns of different DA@xBN#/AlO(OH) powders

shown in Fig.4. Compared with Fig.2b, it can be clearly seen that the distribution of AlO(OH) is relatively uniform, and there are some tiny AlO(OH) particles. As shown in Fig.4b, when the molar ratio of BN to Al(NO₃)₃ is 1:1, some tiny AlO(OH) particles are deposited on BN surface. As shown in Fig. 4c, a large number of tiny AlO(OH) particles are deposited on BN surface, presenting the particle aggregation effect. Therefore, with increasing the Al(NO₃)₃ content, more and more tiny AlO(OH) particles are deposited on BN surface, even forming the aggregation phenomenon. Therefore, it can be concluded that the deposition state of tiny AlO(OH) particles is positively proportional to the Al(NO₃)₃ content.

2.3 DA@BN#/Al₂O₃ powder

Fig. 5 shows XRD patterns of DA@xBN#/Al₂O₃ powders after sintering at 1200 °C. It can be seen that the diffraction peak intensity of BN at 2θ =27°, 42°, and 50° is decreased with increasing the Al(NO₃)₃ content. Moreover, Al₂O₃ has obvious characteristic peaks at 2θ =7.4°, 33.4°, 57.9°, and 66.7°, which correspond to (002), (107), (303), and (1114) crystal planes, respectively. When the molar ratio of BN to Al(NO₃)₃ is 1:2, three obvious Al₂O₃ diffraction peaks can be observed at 2θ =18°-26°.

With increasing the Al(NO₃)₃ content, the intensity of Al₂O₃ diffraction peaks is increased continuously, whereas that of BN is decreased. It can be concluded that when the molar ratio of BN to Al(NO₃)₃ is 1:1, the diffraction intensity of BN and Al₂O₃ is moderate. Then, with increasing the Al(NO₃)₃ content, the deposition of Al₂O₃ on BN surface becomes more obvious firstly and then decreased.

SEM morphologies of different BN/Al₂O₃ powders after sintering at 1200 °C are shown in Fig.6. It can be seen that the microstructures of BN/Al₂O₃ are composed of the micronsized BN with deposited nano-Al₂O₃ particles. According to Fig.6a, the nano-Al₂O₃ particles are deposited compactly onto the BN surface, and it may form a flat structure on BN. As shown in Fig. 6b, the nano-Al₂O₃ are tightly condensed and deposited on the BN surface. It can be observed from Fig.6c that the large BN is closely wrapped by Al₂O₃ particles. However, compared with Fig.6b, due to the agglomeration of Al₂O₃ particles, relatively larger Al₂O₃ particles are formed. As a result, with increasing the Al(NO₃)₃ content, more and more Al₂O₃ particles are deposited on BN surface. However, when the molar ratio of BN to Al(NO₃)₃ is 1:2, severe agglomeration



Fig.4 SEM morphologies of DA@2BN#/AlO(OH) powder (a), DA@1BN#/AlO(OH) powder (b), and DA@0.5BN#/AlO(OH) powder (c)



Fig.5 XRD patterns of different DA@xBN#/Al₂O₃ powders after sintering at 1200 °C

occurs. Therefore, the optimal molar ratio of BN to $Al(NO_3)_3$ is 1:1.

2.4 Thermal conductivity and mechanism of EP-based DA@BN#/Al,O, composites

The thermal conductivity of EP-based $DA(a)xBN#/Al_2O_3$ composites with x=2, 1, and 0.5 is 0.44, 0.62, and 0.33 W·m⁻¹·K⁻¹, which increases by 118%, 212%, and 62%, compared with that of pure EP (0.2 $W \cdot m^{-1} \cdot K^{-1}$), respectively. Besides, the thermal conductivity of EP-based DA@1BN#/ Al₂O₂ composite is 3 times and 1.5 times higher than that of the pure EP composite and BN/Al₂O₂ powder, respectively. It is clear that among these specimens, the EP-based DA@xBN#/ Al₂O₃ composite has the highest thermal conductivity when the molar ratio of BN to Al(NO₃)₃ is 1:1. This result indicates that excess addition of Al(NO₃)₃ leads to the aggregation of deposited Al₂O₃ particles, thereby decreasing the thermal conductivity. Since the crystal structure and physicochemical properties of BN are similar to those of graphite, it can be deduced that BN is composed of layered structures and the layers are bonded to each other by Van der Waals force. As a result, BN is prone to agglomerate in EP matrix.

In this research, DA and PEG were used to modify BN. Some strong polar groups, such as -OH and -NH₃, are introduced onto the BN surface during modification. Thus, the surface adhesion, dispersion, and interfacial compatibility of BN are improved. Additionally, the introduction of functional groups increases the active sites on BN surface, which is helpful for the adhesion of Al_2O_3 particles onto the BN surface. In the preparation process of BN/Al₂O₃ composite powder, the freeze-thaw treatment and the mechanical ball milling process can improve the dispersion of filler and the compatibility of matrix interface to a certain extent^[29].

The thermal conductivity of the polymer composites containing thermal conductive fillers is achieved by forming continuous transmission network or path through the filler of effectively high thermal conductivity. In the EP-based $DA@xBN#/Al_2O_3$ composites, the main thermal conduction paths are formed by BN, and other thermal conduction paths can be formed when Al_2O_3 particles are deposited on the BN surface. Therefore, multiple thermal conduction paths are formed and the thermal conductivity is improved. The thermal conductivity of EP-based composites is improved by the combination of BN and Al_2O_3 filler at low filler content.

The results show that the thermal conductivity of EP-based BN#/Al₂O₂ composites is greater than that of pure EP-based BN composites. Compared with the single BN filler, the composite filler of Al₂O₂ and BN can form a 3D structure in EP matrix, which is beneficial to establish an effective heat conduction and transmission path. It can be seen that the synergistic effect of fillers at different scales improves the thermal conductivity of materials^[30]. However, with increasing the Al(NO₃)₃ content, the thermal conductivity of 0.5BNAl/EP composite is decreased. Because the interface adhesion between BN/Al₂O₂ and EP is weak, serious phonon scattering occurs and large interface thermal resistance is generated. In addition, the high surface energy generated by the large specific surface area due to the excessive Al₂O₃ particles results in the difficulty of Al₂O₃ particle dispersion in EP matrix. Thus, the agglomeration phenomenon hinders the phonon transmission and further affects the heat transfer performance of material. Therefore, the thermal conductivity of the prepared composites is decreased with increasing the $Al(NO_3)_3$ content. Fig. 7 shows the schematic diagram of production and multi-thermal conduction paths of the EPbased BN/Al₂O₂ composite. The dispersion of composite filler is effectively improved by mechanical ball milling and mixture. The 3D structure composed of BN and Al₂O₃



Fig.6 SEM morphologies of DA@2BN#/Al₂O₃ powder (a), DA@1BN#/Al₂O₃ powder (b), and DA@0.5BN#/Al₂O₃ powder (c) after sintering at 1200 °C



Fig.7 Schematic diagram of production and multi-thermal conduction paths of EP-based BN/Al₂O₃ composites

provides effective thermal conduction paths and numerous interface contact points. Therefore, the thermal conductivity of EP-based material is effectively improved.

When the molar ratio of BN to $Al(NO_3)_3$ is 1:1, the BN/ Al₂O₃ composite filler can form an effective heat conduction path in EP. However, when the $Al(NO_3)_3$ content is insufficient or excessive, it may not be able to form the effective thermal conductivity paths, thereby leading to the decrease in thermal conductivity.

3 Conclusions

1) The freeze-thaw treatment is beneficial to change the BN properties. The dispersion and interfacial compatibility of BN are obviously improved by freeze-thaw treatment.

2) The modification effect of dopamine (DA) on BN is better than that by polyethylene glycol (PEG).

3) The microstructures of BN/Al_2O_3 are composed of the micron-sized BN with deposited nano- Al_2O_3 particles. When the molar ratio of BN to $Al(NO_3)_3$ is 1:1, the deposition of Al_2O_3 on BN is uniform and optimal.

4) When BN is treated by freeze-thaw method, DA is used as the surface modifier, and the molar ratio of BN to $Al(NO_3)_3$ is 1:1, the thermal conductivity of this epoxy resin (EP)-based BN/Al₂O₃ composite is 3 times and 1.5 times higher than that of the pure EP composite and EP-based composite material prepared by pure micro-sized BN powder. The BN with deposited Al₂O₃ can form multiple thermal conduction paths, which improve the thermal conductivity of EP-based BN/ Al₂O₃ composites.

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BN/Al_2O_3 复合粉末及其聚合物基复合材料的制备与导热性能

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摘 要:采用固-液相共混法制备了多种 BN/Al₂O₃复合粉末,通过冻融法和表面修饰法对 BN 进行了改性处理,改变表面修饰剂类型和 摩尔比得到了前驱体和烧结态 BN/Al₂O₃复合粉末,并利用机械混合法制备了聚合物基 BN/Al₂O₃复合材料,并测试分析了其导热性能。 结果表明,经冻融处理的 BN 分散性和界面相容性明显优于未经冻融处理的 BN。多巴胺对 BN 的改性效果优于聚乙二醇。采用多巴胺作 为表面修饰剂且 BN 与 Al(NO₃)₃的摩尔比为 1:1 时,能够得到纳米 Al₂O₃均匀包覆的微米 BN 粉末,即 BN/Al₂O₃微纳复合粉末,其聚合物 基复合材料的导热系数可达 0.62 W·m⁻¹·K⁻¹,是纯聚合物导热系数的 3 倍,是采用纯微米 BN 粉末制备的聚合物基复合材料导热系数的 1.5 倍。在 BN 表面附着的 Al₂O₃可以形成层状热传导通道,能够有效提高聚合物基 BN/Al₂O₃复合材料的热导率。 关键词: BN/Al₂O₃;复合粉末;导热性能

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