



Research Article

Thermal and mechanical properties study of boron nitride nanosheets decorated by silver/epoxy nanocomposites



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Abstract

Epoxy is widely used in mechanical and electronic industries due to its excellent adhesive properties and mechanical performances. However, the poor thermal conductivity limits its application, especially in high density equipment. In order to improve the thermal conductivity of epoxy, silver nanoparticles decorated boron nitride nano-sheets (Ag-BN) were prepared and incorporated into the epoxy matrix to obtain nanocomposites (EP-AB) in this work. The EP-AB behaves a significant enhancement of 1089% at an Ag-BN loading of 25 vol% compared to the pure epoxy. The thermogravimetric curve shows that the thermal stability of nanocomposites is improved with the addition of Ag-BN. Moreover, dynamic thermomechanical analysis reveals that Ag-BN can effectively reduce the segmental mobility of the epoxy matrix, which helps to improve the stiffness, decrease the mechanical loss, and increase the glass transition temperature of EP-AB. Those three properties gain an optimum value when the content of Ag-BN is 20 vol%. Also, the nanocomposites perform more stability in mechanical properties than pure epoxy under a varied frequency. The Cole–Cole plot of storage modulus and loss modulus shows that the pure epoxy and nanocomposites with low nanomaterial content are homogenous and the uniformity begins to decrease when the addition of Ag-BN is 20 vol%.

Keywords Silver · Boron nitride · Epoxy · Thermal properties · Mechanical properties

1 Introduction

In recent years, the continual increase in power density generates a large number of heat in mechanical and electronic equipment [1, 2]. Epoxy is widely used in those areas, such as coatings, adhesives, electronic devices, and aerospace due to its excellent adhesive properties, mechanical properties, dimensional stability and electrical performances [3, 4]. However, epoxy has a poor thermal conductivity of 0.1–0.5 W/mK at room temperature [5–7]. During the operation of equipment, mechanical force with high pressure and frequency will result in a large amount of heat. Low thermal conductivity hinders the dissipation of heat, resulting in decomposition, charring, and

mechanical damage of epoxy, which can shorten the service life of the equipment and even lead to the paralysis of the system [8, 9].

Many polymer nanocomposites consist of polymers and thermally conductive nanomaterial were reported to obtain polymers with high thermal conductivity [10–12]. The commonly used nanomaterial include carbon(carbon nanotubes [13], carbon nanofibers [14], graphene [15], etc.), oxides (aluminum oxide (Al₂O₃) [16], beryllium (BeO) [17], (titanium oxide(TiO₂) [18], etc.), nitrides(boron nitride(BN) [19], aluminum nitride (AlN) [20], (silicon nitride) Si₃N₄ [21], etc.), carbide (silicon carbide (SiC) [22], etc.), metals (aluminum (Al) [23], silver (Ag) [24], copper(Cu) [25], etc.), titanate (barium titanate (BaTiO₃)

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[26], strontium titanate (SrTiO_3) [27], etc.) as well as the mixture thereof.

Hexagonal boron nitride (h-BN) is rated as one of the most nanofillers to enhance the thermal conductivity of polymers due to its outstanding thermal conductive, thermal stability, and mechanical properties [28]. Boron nitride nanosheets (BNNS) is a derivative of h-BN, which is a two-dimensional material with a bare (002) crystal plane that can enhance phonon transport and highly thermally conductive [29]. By adding 25 wt% of BNNS into epoxy, the thermal conductivity of the composite has an enhancement of 180% compared to the pure epoxy resin, while the temperature corresponding to 10% of the thermal weight loss increases by nearly 20 °C. Surface modification of BNNS and the hybrid nano-fillers have become one of the research hotspots [30, 31]. Yao et al. [32] prepared a high thermal conductivity polyvinyl alcohol-paper by using polyvinyl alcohol as a matrix and BNNS/AgNP/SiC hybrid as a ternary mixed filler.

While improving the thermal conductivity of polymers, those nanomaterial also have an effect on the mechanical properties. The incorporation of noncovalently functionalized boron nitride nanoflake into epoxy yields a high elastic modulus and ultimate tensile strength, the toughness is also improved [33]. The investigation of epoxy composites with hybrid boron nitride nanotubes and boron nitride nanosheets shows that the Young's modulus is improved with a low content of nanofillers [34]. Polyvinylpyrrolidone modified hBN nanoplatelets can improve the flexural properties and thermal stability of epoxy [35]. A block copolymer polyethylene oxide-polypropylene oxide-polyethylene oxide was used to modified hBN nanosheets, the flexural modulus and strength increase 3.6% and 5.7% with 0.08 wt% nanosheets in epoxy matrix and it can help to facilitate the dampening of cracks for flexural properties enhancement [36].

In this paper, by preparing BNNS decorated nano-silver (Ag-BN) and adding them into epoxy matrix, we successfully obtain the nanocomposite (EP-AB) with high thermal conductivity and good mechanical properties. Also, the effects of Ag-BN on thermal conductivity, thermal stability and mechanical properties is discussed. It's expected that such a nanocomposites is suitable for mechanical equipment under high heat density.

2 Samples preparation

2.1 Preparation of Ag-BN

Firstly, the h-BN was dissolved in a sufficient amount of N,N-methylformamide and then put them in the ultrasonic pool for 72 h. In this process, the h-BN was exfoliated into

BNNS. Next, slowly adding a solution of silver nitrate and N,N-methylformamide into the mixture that was stirring and sonication synchronously, this process would last for 2 h. After that, the mixture was kept at the room temperature for a whole day and night to ensure uniform distribution of the nano-silver on the surface of the BNNS. Finally, the solution was filtered, repeatedly washed with alcohol and acetone, and dried to obtain Ag-BN.

2.2 Preparation of EP-AB

The Ag-BN was added into acetone and homogenized by ultrasonic. Then, E-51 epoxy was added into the solution and continuous stirred at 70 °C for 7 h until the acetone was completely evaporated. After adding 593 curing agent, the mixture was kept stirring and sonication synchronously at room temperature for 1 h, which is followed by 1 h degassing in a vacuum box for 1 h. After that, the curing process was in a mold at 100 °C for 5 h to get composites of EP-AB. By this method, the nanocomposites containing 5 vol%, 10 vol%, 15 vol%, 20 vol% and 25 vol% of Ag-BN were prepared.

2.3 Morphology characterization and performances measurement

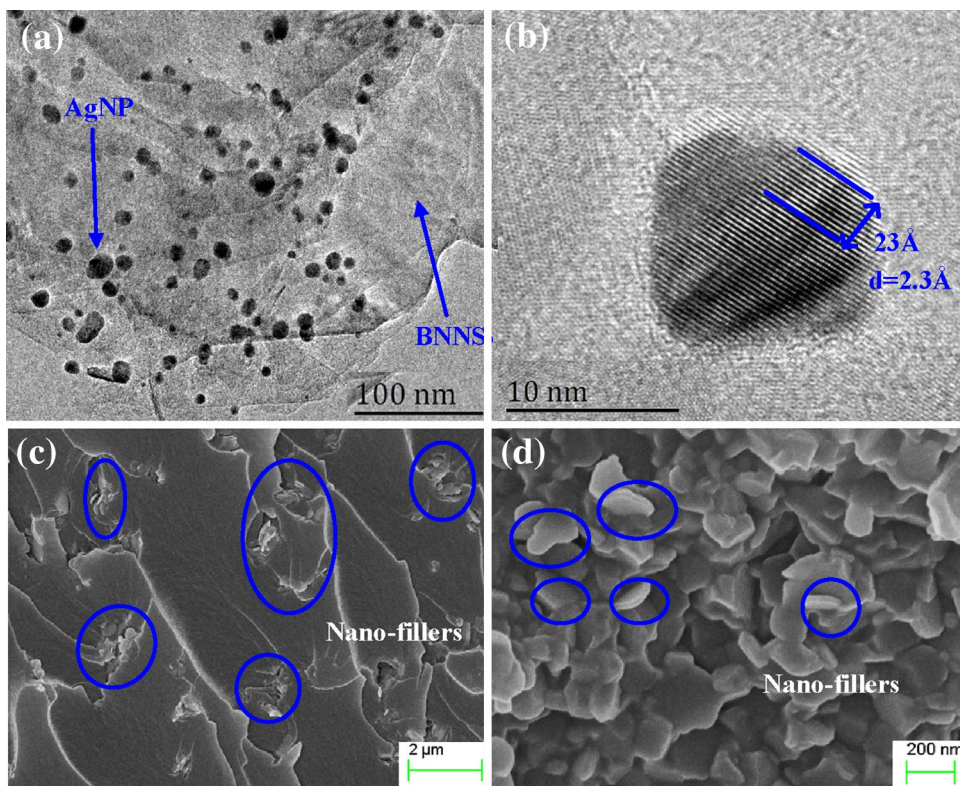
The morphology of Ag-BN was observed by transmission electron microscopy (TEM) and the acceleration voltage was 200 kV. The morphology of EP-AB was tested by scanning electron microscope (SEM) and the accelerating voltage is 30 kV. The thermal conductivity was measured by fully automatic thermal conductivity meter, which used the heat flow method according to the ASTM D5470-2006 standard. The thermogravimetric curves were measured by synchronous thermal analyzer, the gas atmosphere was nitrogen, the measurement temperature ranged from 40 to 600 °C, and the heating rate was 10 °C/min. The dynamic thermomechanical analysis was used to test the stiffness and mechanical loss of a temperature range from – 30 to 130 °C and a temperature rise rate of 3 °C/min. The measured forces and displacements were set to 5 N and 10 μm, respectively, and different measurement frequencies of 1 Hz, 5 Hz, 20 Hz, and 40 Hz were tested to study the effect of frequency on dynamic thermomechanical properties.

3 Results and discussion

3.1 Morphology characterization of Ag-BN and EP-AB

Figure 1a, b show the TEM graph of Ag-BN at different scales. The transparent sheet in Fig. 1a is BNNS, and the

Fig. 1 **a, b** TEM graphs of Ag-BN at different scales; **c, d** SEM graphs of EP-BN at different scales

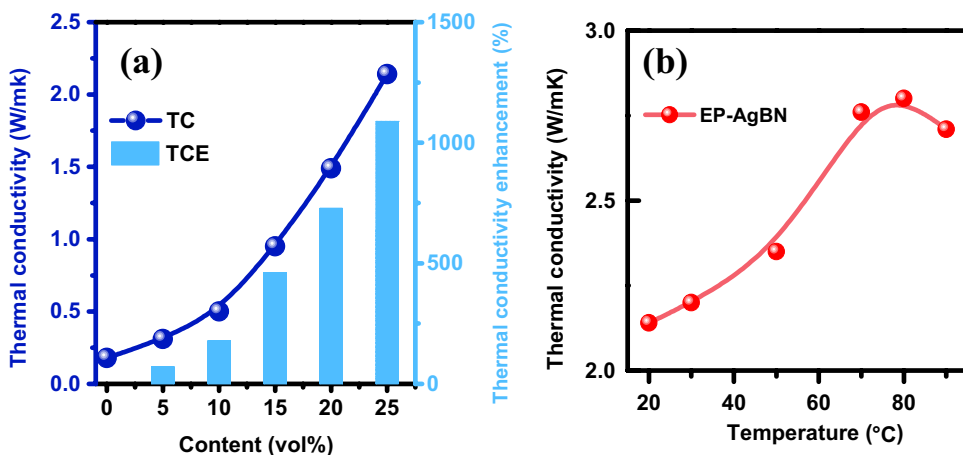


black dots with a size of 5–15 nm on the surface of BNNS are silver nano-particles. In the image, there are no AgNP in other regions except on the BNNS, which indicates that the AgNP generated by reduction are fixed on the BNNS. Figure 1b shows the lattice diffraction fringes of black dots with a stripe width of 2.3 Å, which corresponds to the plane (111) of silver [35] prove that the prepared product is nano-silver decorated boron nitride nano-sheets.

Figure 1c, d is the SEM graphs of EP-AB at different scales. In Fig. 2, nanomaterial with several hundred

nanometers are distributed in the epoxy matrix and before they are uniformly dispersed, there is a trend of aggregating into small groups which may because of the surface effect with small size. The nanomaterial are closely connected with the matrix due to high temperature and pressure during the preparation process. However, it is difficult to distinguish BNNS and silver from the surface topography because of the non-transparent epoxy matrix.

Fig. 2 **a** Thermal conductivity and thermal conductivity enhancement of composites; **b** thermal conductivity as a function of temperature



3.2 Thermal conductivity

Figure 2a shows the thermal conductivity and thermal conductivity enhancement of EP-AB. Pure epoxy has a low thermal conductivity of 0.18 W/mK, but the thermal conductivity of EP-AB increase gradually with the addition of Ag-BN. When the content of Ag-BN is 10 vol%, the thermal conductivity of EP-AB is 0.5 W/mK. The improvement is not obvious because of the low nano-fillers loading, the Ag-NP are completely surrounded by the epoxy and no contact exists among fillers. However, with the content of Ag-BN exceeding 15 vol%, the thermal conductivity of nanocomposites improves significantly, and the thermal conductivity of EP-AB reaches 2.14 W/mK at a nano-filler loading of 25 vol%. This is attributed to the bridge connection of silver among BNNS and thermal conduction channels formation in the composite [34, 37]. As for the thermal conductivity enhancement, it increases with the content of Ag-BN. When the content is 15 vol%, the thermal conductivity enhancement is nearly 500%, and it is as high as 1089% with a Ag-BN loading of 25 vol%. All of these indicate that the prepared nanomaterial can significantly improve the thermal conductivity of epoxy.

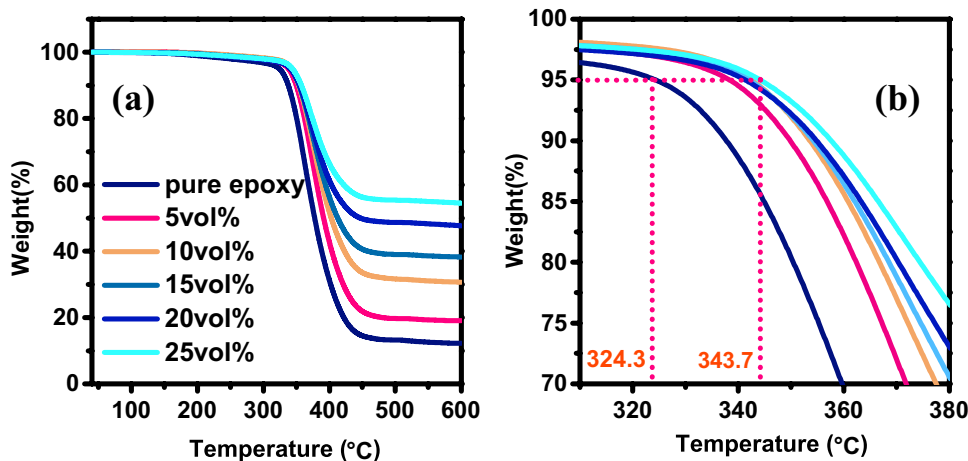
Temperature dependence of thermal conductivity is also studied for EP-AgBN. The results are presented in Fig. 2b. The thermal conductivity increases with temperature up to 80 °C, which is likely due to the enhanced phonon transmission at interface and reduction in the interfacial thermal resistance between nanoparticles and h-BN sheets. The thermal conductivity decreases slightly beyond 80 °C which could be associated with dominant Umklapp phonon–phonon scattering at high temperatures [38].

3.3 Thermal stability

As for materials used in mechanical industries, the thermal stability has a significant impact on their service life. Figure 3 shows the thermal weight loss curves of pure epoxy and EP-AB under the nitrogen atmosphere. In Fig. 3a, the thermal decomposition of the composites can be basically divided into three stages. The first stage is the temperature below 300 °C. During this period, there is a small mass loss due to the dehydration of the secondary alcoholic groups and the evaporation of the water with a weak physical connection to the surface of composites [39]. The second stage is the temperature from 300 to 450 °C, which is the main stage of epoxy decomposition and occupies the majority of weight loss. In this phase, the mass loss is caused by the gas products generated by the pyrolysis of aromatic groups in epoxy and amine groups in curing agent [40]. When the temperature is future higher, the weight loss of the composites hardly changes in the third stage. The remainders of this stage are composed of carbonized materials and nanomaterial that cannot be decomposed under a nitrogen atmosphere. With the addition of Ag-BN, the decomposition temperature and thermal stability of the composites are all improved.

In Fig. 3b, the temperature corresponding to 5% of the weight loss increased from 324.3 °C of pure epoxy to 343.7 °C of EP-BN with a 25 vol% Ag-BN loading, nearly 20 °C. The highly thermally conductive nanomaterial promote the formation of carbon and improve the heat resistance of the polymer, which can act as a barrier to gases produced by polymer degradation and reduce the mass release of volatiles [41]. Moreover, nanomaterial limit the mobility of epoxy segments and enhance the thermal stability of EP-AB.

Fig. 3 The weight loss curve of pure epoxy and EP-AB **a** from 40 to 600 °C; **b** from 310 to 380 °C



3.4 Dynamic thermo-mechanical properties

The dynamic thermos-mechanical analysis yields a complex modulus $E^* = E' + iE''$, where the real part E' is storage modulus and represents the energy storage capacity in the material under strain. The imaginary part E'' is loss modulus and represents the loss of energy. The ratio of these two $\tan \delta = E''/E'$ is the loss tangent, which represents the mechanical loss [42].

3.4.1 Storage modulus (E')

Storage modulus is dependent on the stiffness of materials. Figure 4a shows the storage modulus changes with temperature. The E' of pure epoxy and EP-AB is not big, mainly because of the low stiffness of epoxy [43]. However, the storage modulus of EP-AB increases obviously with the addition of nanomaterial. When the content of Ag-BN is 20 vol% or less, the E' of EP-AB increases gradually due to the stiffness of BNNS is higher than that of the epoxy matrix and the nanofillers can effectively inhibit the mobility of epoxy segments. Also, tougher material has an efficient mechanism of internal energy absorption, which restricts the crack propagation and results in increase of stiffness [44]. But with the nanomaterial adding more (25 vol%), the E' of EP-AB decreases slightly, which may attribute to more space for segments movement provided by the stacking of nanomaterial and the gaps between nanomaterial and epoxy matrix [45]. Moreover, the E' of all the samples shows a decrease trend with increasing temperature because the movement of epoxy segments accelerated with the increase temperature. Also, the E' of all the samples approaches 0 as the temperature above 100 °C. As because in this state, with the temperature reaches the glass transition temperature of the sample, they are converted from a glassy state to a highly elastic state and

the matrix is not practically in contact with nanomaterial, resulting in no shear force between them.

Figure 4b shows the E' of pure epoxy and EP-AB with a content of 20 vol% varies with frequency. For the pure epoxy, the E' drops sharply with the frequency increase from 1 to 5 Hz, which indicates that the mobility of the epoxy segments is significantly increased in this frequency band. On the other hand, as the frequency further rises to 50 Hz, the storage modulus is nearly no longer changed, which means that the mobility of epoxy chains keeps at a high level and fluctuated slightly. As for the EP-AB with a loading of 20 vol%, the effect of frequency on the storage modulus is not significant. It proves that the Ag-BN can not only limit the motion of epoxy segments, but also maintain stability of the movement under a changing frequency.

3.4.2 Mechanical loss ($\tan \delta$)

The tangent of the mechanical loss angle indicates the internal damping of the material, which reflects the viscosity of the material. The peak value is related to the energy dissipation under stress and the temperature corresponding to the peak value of $\tan \delta$ is usually used to characterize the glass transition temperature (T_g) of the sample [46]. Figure 5b shows the $\tan \delta$ of pure epoxy and EP-AB vary with temperature. It is obvious that the pure epoxy has the highest peak value, which means a higher degree of segments mobility [47]. The peak value of $\tan \delta$ decreases marginally with the presence of Ag-BN because of the nanomaterials can reduce the viscoelastic damping coefficient of epoxy, thereby slowing down the energy dissipation. The height peak of $\tan \delta$ gets the minimum value at a nanomaterial content of 20 vol%, and it increases slightly when the Ag-BN loading is 25 vol%. The aggregation of Ag-BN creates more gaps or voids in the epoxy matrix, thus providing the space for molecular motion and resulting in an enhancement of damping coefficient. However, seen

Fig. 4 Storage modulus of samples **a** varies with temperature; **b** varies with frequency

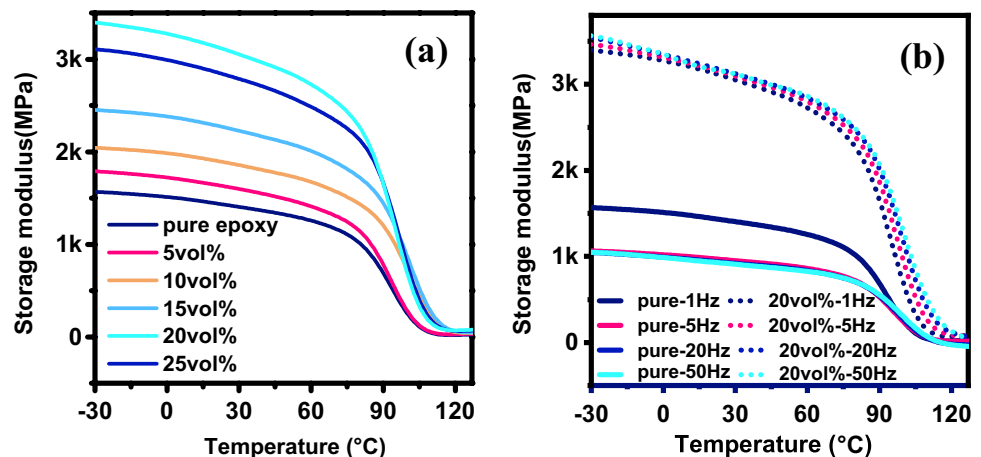
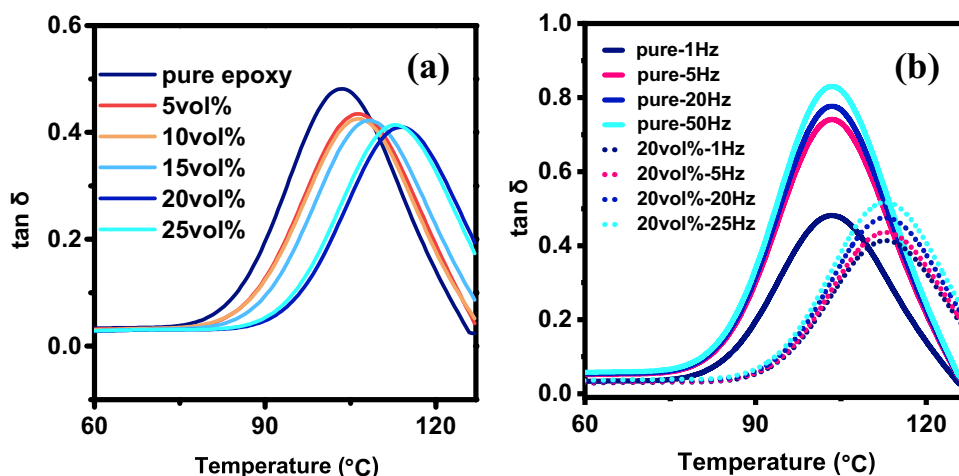


Fig. 5 Mechanical loss of samples **a** varies with temperature; **b** varies with frequency



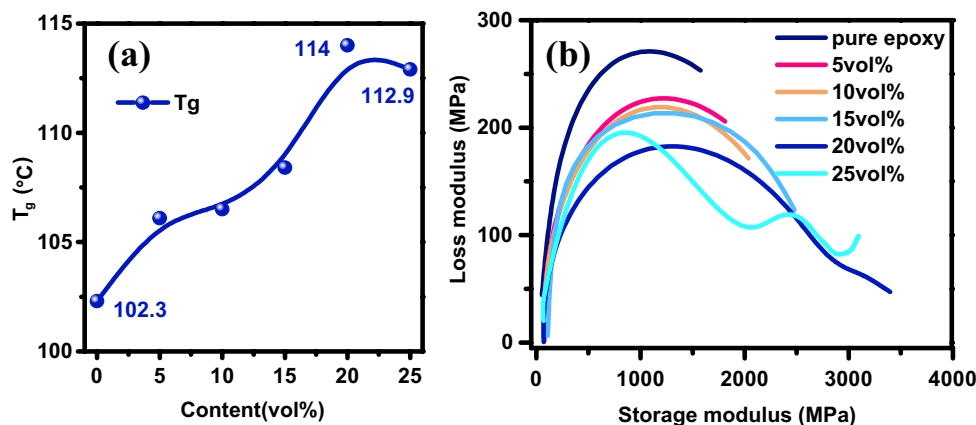
from the perspective of severity of impact, the effect of nanomaterial prepared in this paper on the storage modulus is much greater than that on the mechanical loss.

Figure 5b shows the $\tan \delta$ of pure epoxy and EP-AB with Ag-BN content of 20 vol% varies with frequency. It's notable that the frequency only affects the peak value of both pure epoxy and EP-AB, but the effect on temperature corresponding to the peak value is indiscernible. For pure epoxy, the peak value of $\tan \delta$ rises sharply with the frequency increases from 1 to 5 Hz for that the increasing frequency causes an abrupt decrease of E' . And the acceleration of molecular movement at a higher frequency also increase the E'' . As the frequency is further high, the peak value of $\tan \delta$ increases slightly because that the E' hardly changes in this frequency band and the E'' increases with the increasing frequency. On the other hand, the $\tan \delta$ peak value of EP-AB goes up marginally with frequency, which indicates that the frequency stability in mechanical loss of the prepared nanocomposite is higher than that of the pure epoxy.

3.4.3 Glass transition temperature

The content and dispersion of nanoparticles can influence the glass transition temperature of epoxy [48]. Figure 6a shows the glass transition temperature of pure epoxy and EP-AB. Compared with pure epoxy, the nano-composite has a higher T_g and the T_g increases first and decreases slightly with the content of nanomaterial. The T_g gets the maximum value of 114 °C with a Ag-BN content of 20 vol%, which is nearly 12 °C higher than that of pure epoxy (102.3 °C). The incorporation of Ag-BN to the matrix acts as a hindrance and increases the heterogeneity of the cross-linked structure of nanocomposites. Moreover, increased heterogeneity minimizes the free space and subsequently restricts the polymer mobility within EP-AB and the interaction between the functional group of BNNS and epoxy also causes the increase in T_g [44, 48]. In Fig. 5d, the frequency has little effect on the temperature corresponding to the peak value of $\tan \delta$ means that the frequency would not affect the T_g of the sample.

Fig. 6 **a** The glass transition temperature of pure epoxy and EP-AB; **b** the Cole–Cole plot of pure epoxy and EP-AB



3.4.4 Cole–Cole plot

The Cole–Cole plot can be used to interpret the relationship between E' and E'' , which can effectively obtain the homogeneity of samples [45]. Studies show that the curve with smooth semi-arc shape indicates that the material is homogenous. Conversely, the curve deviates from the semicircular shape or irregularity indicates that the material is nonhomogeneous due to phase segregation or aggregation of nanomaterial [49]. Figure 6b shows a Cole–Cole plot of the E' as the horizontal axis and E'' as the vertical axis. Obviously, the curves of epoxy and EP-AB with a low nanomaterial content (below 15 vol%) are smooth and almost similar semicircular means perfect dispersion of Ag-BN in the epoxy matrix. The nanocomposite with 20 vol% of nanomaterial basically shows a similar semicircular, but tend to deviate from the semicircular in the high E' region, indicating that the homogeneity is worse than that of pure epoxy and EP-AB with a low nanomaterial content. However, the curve of EP-AB with 25 vol% of Ag-BN is more irregularity and represents further nonhomogeneous.

4 Conclusion

In this work, aiming at the problem of poor thermal conductivity of epoxy, by preparing nano-silver decorated boron nitride nano-sheets first and incorporated into epoxy matrix as a filler, we successfully obtained the nanocomposites with ultra-high thermal conductivity. The conclusions are as follow:

1. The EP-AB behaves a significant enhancement of 1089% at an Ag-BN loading of 25 vol% compared to the pure epoxy.
2. An increase of 20 °C in the temperature corresponding to 5% of the weight loss means that the thermal stability is improved and it can be used in higher temperature environments.
3. The dynamic thermomechanical properties reveal the effects of nanomaterial on the stiffness, mechanical loss, and glass transition temperature of the composites. The nanomaterial content has the best value for improving the dynamic thermomechanical properties. When the content of nanomaterial is 20 vol%, the prepared nanocomposite has the highest stiffness, the smallest mechanical loss, and the highest glass transition temperature, which has an increase of 12 °C.
4. The frequency stability in mechanical properties of composite materials also behaves better than that of pure epoxy. Moreover, the Cole–Cole plot can effectively represent the homogeneous of the composites.

All of these demonstrated that the nanocomposites prepared in this paper could be a potential materials in mechanical equipment with high mechanical stress and heat density.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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