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# Few-layer boron nitride nanosheets: Preparation, characterization and application in epoxy resin

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## ABSTRACT

Large-scale exfoliated boron nitride nanosheets were achieved via a liquid exfoliation sonication method by using sodium fluoride and ammonium hydroxide as Lewis base compounds. The crystal structure, surface functional groups, morphology and thickness of the as-prepared samples were characterized by X-ray diffraction, fourier transform infrared spectroscopy, transmission electron microscopy and atomic force microscopy, respectively. The as-prepared samples were introduced into epoxy resin to fabricate the polymer-based composites. Experimental results showed that the layer thickness of the as-prepared nanosheets was in the range of 1 to 3 layers. Moreover, it could improve the tensile properties of the matrix. When the loading of the as-prepared nanoparticles was 0.4 wt%, the tensile strength and elongation at break of the composites reached to their maximum values 65.6 MPa and 25.9%, which were increased by 118% and 192% more than that of pure resin. In addition, the as-prepared boron nitride samples could improve the thermostability and promote the curing of the matrix.

## 1. Introduction

Boron nitride (BN), so-called “white graphite”, is a layered structure material which is analogous to graphite. As reported, boron nitride has equally good mechanical, thermal properties and chemical stability [1–4], which can be widely used in the fields of mechanical parts, electronic devices, multifunctional composite fillers and so on [5–10]. Recent advances have been focused on making BN into monolayer or few-layer boron nitride nanosheets (BNNS) due to their better performance than that of the bulk counterpart [11–13]. Up to now, the bottom-up [14] and top-down methods [15] have been used to prepare BNNS. Compared to other methods, the top-down methods usually show some advantages, such as simple process, high output, high efficiency and low cost [16]. Among the large number of reported top-down methods, liquid exfoliation sonication is a promising way for large-scale industrialization [17].

Recently, attaching amino functional groups to the B atoms in preparation of BNNS has been reported. The locally polarized B–N bonds allow convenient targeting of either B or N atoms for the Lewis acid–base interaction [18–20] indicating that B atoms possess Lewis acid characteristics and are susceptible to the attack by Lewis base. For example, stable Lewis acid–base complexes can be formed based on the

amine group, which makes BN dispersed easily and exfoliated. Therefore, the exfoliation of BNNS should be benefit from the Lewis base characteristics [21].

In this work, in a hydrothermal process, large-scale exfoliated BNNS were achieved via a liquid exfoliation sonication method by using commercially available BN as raw materials, sodium fluoride and ammonium hydroxide as Lewis base compounds. The possible exfoliation mechanism was also proposed. Moreover, in order to evaluate the as-prepared BNNS, it was introduced into epoxy (EP) resin, and the tensile and thermal properties of the EP/BNNS composites were discussed. Experimental results showed that adopting hydrothermal strategy and using commercially available boron nitride could efficiently prepare BNNS. The as-prepared BNNS could obviously improve the tensile strength and elongation at break of the matrix. Furthermore, it could also enhance the thermostability and promote the curing of the matrix.

## 2. Experimental

### 2.1. Materials and methods

Hexagonal BN and sodium fluoride were purchased from

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Sinopharm Chemical Reagent Co., Ltd, China. Bisphenol-A EP (DQ200E) and curing agent (DQ204A) were supplied by Dong Fang Turbine Co., Ltd, China. Ammonium hydroxide and absolute ethanol were obtained from Shijiazhuang Chemical reagents Co., Ltd, China. All reagents were AR grade.

## 2.2. Preparation of BNNS

Typically, 1.0 g BN powder and 11.34 g sodium fluoride were fully dispersed in 30 mL deionized water by ultrasonic wave (500 W). Then, 30 mL ammonium hydroxide was added into the above solution, and the mixture was sealed in a 150 mL Teflon-lined stainless-steel autoclave. Subsequently, the autoclave was kept in a drum wind drying oven at 180 °C for 24 h and naturally cooled down to the room temperature. The white precipitate was washed by using deionized water and absolute ethanol until the pH of the filter liquor was close to seven. The obtained samples were re-dispersed in ethanol by sonicating (500 W) for 30 min. Finally, the as-prepared samples were dried at 60 °C for 12 h.

## 2.3. Preparation of the EP/BNNS composites

A certain amount (0, 0.16, 0.32, 0.48 and 0.64 g) of the as-prepared BNNS was fully scattered in 20 g curing agent by ultrasonic wave (500 W). Then, 60 g EP resin was mixed with it and then vigorously stirred at 55 °C for 30 min. The fillers loading were 0, 0.2, 0.4, 0.6 and 0.8 wt% based on the total weight of the EP resin and curing agent. Subsequently, the mixture was degassed in vacuum to get rid of bubbles. Finally, the obtained mixture was poured into a mould for pre-curing at 80 °C for 2 h and post-curing at 100 °C for 2 h. A control sample, pure EP, was also prepared via the same route without using the as-prepared BNNS.

## 2.4. Characterization

The crystal structure of pristine BN and the as-prepared BNNS was characterized by using x-ray diffraction (XRD, Model: D8ADVANCE, Bruker Co., Germany) with Cu-K $\alpha$  radiation at a scanning rate of 4 °/min. The functional groups of pristine BN and the as-prepared BNNS were determined by a Fourier transform infrared spectrometer (FT-IR, Model: 8400S, Shimadzu Co., Japan). The surface morphology of the samples particle size of the products was observed by a transmission electrical microscope (TEM, Model: JEM-2100, JEOL Co., Japan) with an accelerating voltage of 120 kV. The layer thickness of the as-prepared BNNS was conducted by an atomic force microscope (AFM, Model: MFP-3d, Asylum Research Co., American) under tapping mode. Moreover, the tensile properties and elongation at break of pure EP and the EP/BNNS composites were carried out on a universal testing machine (Model: GOTTECH AI-7000M, Taiwan high speed rail testing instrument co., Ltd., China). The thermostability of pure EP and the as-prepared EP/BNNS composites were examined on a thermogravimetric analyzer (TGA, Model: TGA/SDTA 851 $^{\circ}$ , Mettler-Toledo apparatus, Switzerland) at the heating rate of 10 °C/min under the protection of argon. In addition, calorimetric studies of the samples were carried out by using a Differential scanning calorimeter (DSC, Model: DSC822 $^{\circ}$ , Mettler-Toledo apparatus, Switzerland) at the heating rate of 10, 20 and 40 °C/min, respectively.

## 3. Results and discussion

### 3.1. XRD analysis

Fig. 1 shows XRD pattern of pristine BN and the as-prepared BNNS. It can be seen in Fig. 1 that the characteristic diffraction peaks of the as-prepared BNNS match well with that of pristine BN. However, the peak intensity of the as-prepared BNNS is weaker than that of

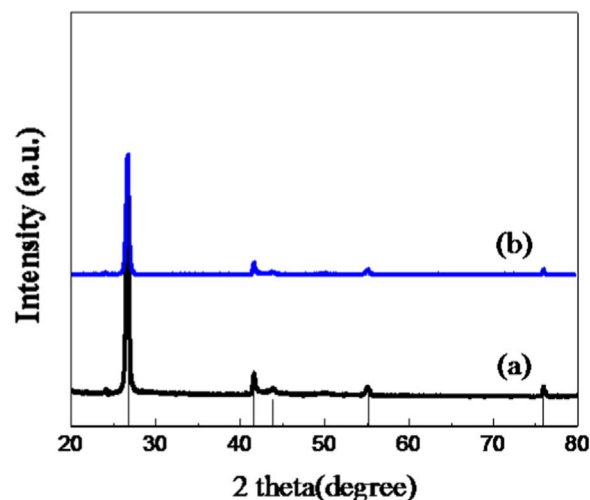


Fig. 1. XRD spectra of pristine BN (a) and the as-prepared BNNS (b) Xiangfeng Wu, et al.

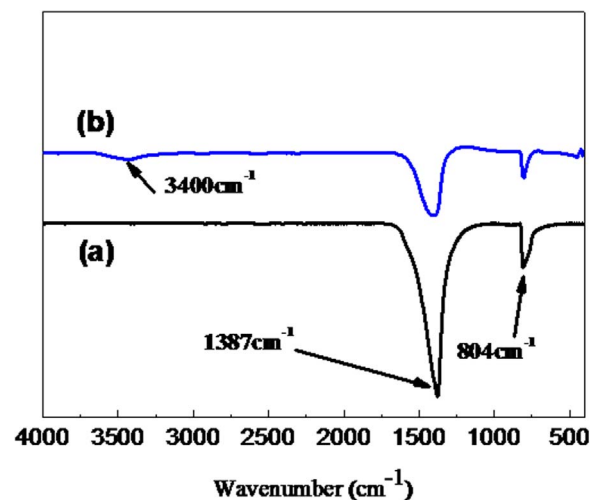


Fig. 2. FT-IR spectra of pristine BN (a) and the as-prepared BNNS (b) Xiangfeng Wu, et al.

pristine BN. It may be attributed to decreasing density of the as-prepared BNNS and caused by the exfoliation of BN into BNNS.

### 3.2. FT-IR analysis

Fig. 2 shows FT-IR spectra of pristine BN and the as-prepared BNNS. It can be seen that pristine BN and the as-prepared BNNS exhibit two sharp absorption peaks at 1387 and 804  $\text{cm}^{-1}$ , which are attributed to in-plane BN stretching vibration of  $\text{sp}^2$ -bonded and out of plane B-N-B bending vibration [22]. Moreover, there is a broad peak at 3400  $\text{cm}^{-1}$  shown in Fig. 2(a), which corresponds to hydroxyl groups.

### 3.3. TEM analysis

TEM images of pristine BN (a) and the as-prepared BNNS (b) are shown in Fig. 3. It can be observed in Fig. 3(a) that pristine BN is flakes with the size range of 100–250 nm. Moreover, in Fig. 3(b), the as-prepared BNNS is thinner than that of pristine BN. This indicates that using sodium fluoride and ammonium hydroxide as the Lewis base compounds can efficiently exfoliate pristine BN into BNNS during the hydrothermal process.

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