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# Preparation of few layers hexagonal boron nitride nanosheets via high-pressure homogenization



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## ABSTRACT

We demonstrated a facile and efficient approach to fabricate well-dispersed boron nitride nanosheets (BNNs) by liquid phase exfoliation of hexagonal boron nitride (h-BN) bulk using a high-pressure homogenizer. The h-BN bulk was mixed with isopropyl alcohol for several hours, and then homogenized under 100 MPa for 10 min, followed by centrifugation to obtain the BNNs. The resulted few-layer BNNs are confirmed by Raman spectra and transmission electron microscope. The concentration of BNNs dispersion is accurately measured by Inductive Coupled Plasma, up to  $0.083 \text{ mg mL}^{-1}$  and the yield is 3.02%. These results provide a physical perspective applied to the other layered materials by liquid phase exfoliation. The above findings may pave the way for the scalable production of BNNs.

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## 1. Introduction

Hexagonal boron nitrides (h-BN), the so-called “white graphite”, have recently received intensive attentions due to their extraordinary physical and chemical properties in the past few years [1,2]. The h-BN nanosheets (BNNs) are structurally analogous to graphene nanosheets, but it has been demonstrated an improved constant wide band gap, more thermal stability and distinguished chemical stability than graphene [2,3]. It offers opportunities for opening up new fundamental and technological possibilities in fields like catalyst, thermal conductors, and high-performance composites [4,5]. Various methods are available to prepare few-layer BNNs. Among them, chemical vapor deposition (CVD) and liquid phase exfoliation (LPE) are the most popular routes. CVD has been demonstrated for the synthesis of low-defect, single atomic sheet BNNs [6], but the high cost and complicated procedure makes it difficult to scale up. LPE is a relatively simple and manageable approach to produce BNNs. Generally, researchers mixed the h-BN power with the solvent such as isopropyl alcohol (IPA) [9], N, N-dimethylformamide (DMF), dimethyl sulfoxide (DMSO) [10] and N-methylpyrrolidone (NMP) [7] with the assistance of ultrasonic [7] or ball-milling [8]. Surfactants like polyvinyl pyrrolidone (PVP) [11], sodium cholate (SC) [12] and methanesulfonic acid [13] could also be used to prepare high-

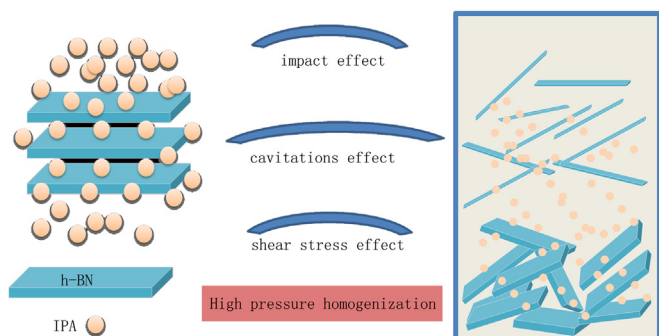
concentration BNNs, but it's difficult to get rid of the surfactants completely. The scalability of the product by the approach of ultrasonic and ball-milling is also limited for the drawback of high-energy consumption and time-consuming. Nowadays, considerable attentions have focused on other mechano-chemical reactions [14] to produce BNNs, for example, Coleman used high-shear mixing method to exfoliate h-BN [15].

The high pressure homogenization (HPH) could bring about three consequent effects: cavitations effect, impact effect and high shear stress effect [16,17]. It has been widely applied in the treatment of liquid samples for reducing particle size and modifying mechano-chemical properties. The strong force can overcome the van der Waals force between molecules and it can delaminate the layered materials. Peukert and co-workers [18] reported this method for exfoliating graphene in the solvent of NMP and NMP-additives. Our group had used this technology for delaminating graphene and  $\text{MoS}_2$  nanosheets in the pure solvent DMF [19,20].

Herein we developed a versatile and effective method for LPE of h-BN in IPA. We had investigated the exfoliation of layered h-BN using a high pressure homogenizer under 100 MPa for 10 min to give stable dispersions of BNNs. The dispersion exhibited obvious Tyndall phenomenon and showed characteristic absorption in UV-vis spectrum. Using techniques from the Raman spectrum, transmission electron microscope (TEM) and high resolution TEM (HRTEM), we revealed that the BNNs dispersion had only few layers.

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**Fig. 1.** Schematic illustration of the exfoliation procedures to prepare BNNs in IPA solvent using high pressure homogenization.

## 2. Experimental

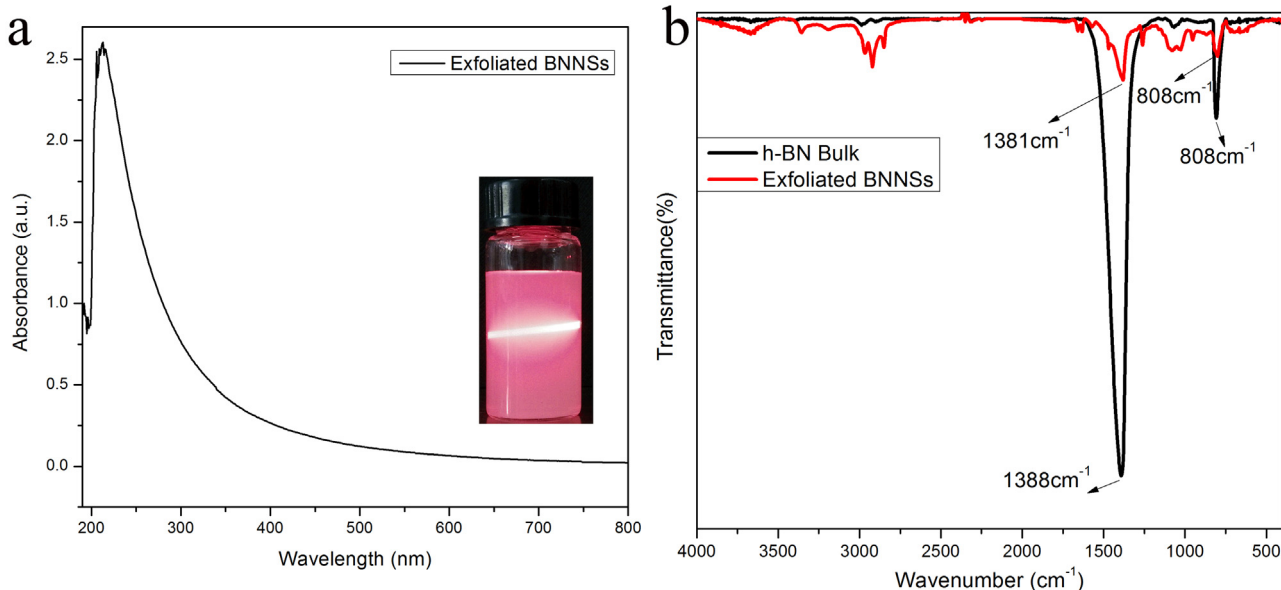
In a typical experiment, the pristine h-BN were provided by Aladdin ( $< 2 \mu\text{m}$ ) and other chemical reagents were of analytical grade and obtained commercially. The h-BN bulk (0.5 g) was dispersed in 200 mL IPA at an initial concentration of  $2.5 \text{ mg mL}^{-1}$ . Then, the mixture was subjected to the high pressure homogenizer under 100 MPa for 10 min keeping at  $25^\circ\text{C}$  through an external cooling apparatus, followed by centrifugation at 5000 rpm for 60 min. The supernatant was collected by pipette carefully and the milky supernatant was the BNNs. UV–vis spectra of BNNs were measured by UNICAN UV-500 instrument from 200 nm to 800 nm. Fourier Transform Infra-Red Spectroscopy (FTIR) was observed on a Thermo-Nicolet Magna-IR760. The samples were prepared by mixing the hBN or BNNs with KBr and were made into a pellet to collect the spectra which were used to identify the functionality of the exfoliated hBN. Raman spectroscopy was performed by Lab-RAM Aramis with He-Ne laser (633 nm) as the irradiation light source. The sample was prepared on a Si/SiO<sub>2</sub> wafer with an oxide layer of 300 nm. TEM (FEI-Tecna 12) and HRTEM (JEM-2010HR) were operated with an accelerating voltage of 200 KV and a micro-grid copper covered with carbon film was used to deposit the BNNs. AFM studies were carried out in a tapping mode on a NanoScope III (R) instrument after depositing a drop of the diluted dispersions on freshly cleaved mica. The concentration of the

BNNs was accurately calculated by ICP (Inductive Coupled Plasma) Emission Spectrometer. The flow rate of sample introduction and sample flow rate during the analyses were 50 rpm and the pump stabilization time was 5 s. The radio frequency applied power was 1150 w and the pressure of nebulizer gas was 0.2 MPa. The gas flow rate of auxiliary and plasma gas were  $0.5 \text{ L min}^{-1}$  and  $15 \text{ L min}^{-1}$ , respectively. In order to calculate the yield of BNNs, the amount of un-exfoliated bulk was measured quantitatively by vacuum filtration.

## 3. Results and discussion

**Fig. 1** illustrated the exfoliation process to prepare BNNs in the pure IPA solvent using the high pressure homogenizer. The starting h-BN bulk was exfoliated in the high pressure homogenizer under the pressure of 100 MPa for 10 min. A milky BNNs dispersion (**Fig. 2a** inset) was obtained by direct exfoliation of h-BN powder through HPH. The suspension was stable, and showed no precipitation after being stored for two weeks under ambient conditions. We used a 635 nm laser light to confirm the Tyndall effect to directly probe these colloidal dispersions. The light scattering effect was visible only when the colloidal nanoparticle size was below or near the wavelength of the exciting light. It's a representative phenomenon that certified the presence of nanometric structure. The obvious peak of the UV–vis absorption spectrum was observed at 209.5 nm (or 5.92 eV) (**Fig. 2a**) which was attributed to the intrinsic exciton absorption band of h-BN. However, there was spectral red shift effects compared with previous observations. It's owned to the size reduction of the nanosheets or the so called quantum confinement effect [6,21]. In the FTIR spectra (**Fig. 2b**), the characteristic B-N stretching mode was typically observed at  $1388 \text{ cm}^{-1}$  for the bulk hBN and  $1381 \text{ cm}^{-1}$  for the exfoliated BNNs. In addition, the out of plane bending mode of B-N-B were shown at  $808 \text{ cm}^{-1}$  for both of them [22]. Thus, there was no functionality for the BNNs.

Raman spectroscopy was employed to characterize the lattice vibration modes of two dimensional layered materials in the light of the peak shift, peak intensity and full width at half maximum (FWHM). The bulk exhibited a characteristic Raman peak (**Fig. 3a** and **b**) from  $E_{2g}$  phonon mode (B-N vibration mode) similar to  $E_{2g}$



**Fig. 2.** (a) UV–vis spectra of BNNs in IPA; Inset: photograph of exfoliated BNNs with the irradiation of a red laser beam. (b) FTIR spectra of bulk h-BN and exfoliated BNNs. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

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