



# In-situ deformation mechanism and orientation effects in sintered 2D boron nitride nanosheets



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## ABSTRACT

Two-dimensional hexagonal Boron nitride nanosheets (BNNS) are consolidated as a monolithic pellet by spark plasma sintering at 1650 °C with a pressure of 50 MPa without any sintering aid. X-ray diffraction study confirmed the h-BN structure after sintering of BNNS along with B<sub>2</sub>O<sub>3</sub> and B<sub>4</sub>C as trace impurities. The preferred orientation of the basal plane (0002) perpendicular to the direction of pressure is observed on the top surface of the pellet after consolidation. High load (several hundred N) in-situ indentation studies inside SEM are carried out in the cross-section and on the top surface of the sintered BNNS pellet to understand the orientation effect on the deformation behavior. Total energy dissipation during indentation along the top surface was 50% greater than the cross-section. Predominant deformation mechanisms observed on the top surface indentation are compression of the layered sheets, delamination and BNNS pile-up. For the cross-section direction, the key deformation mechanisms are cracking and fracturing with an insignificant delamination. The dominant deformation mechanisms in sintered BNNS pellet are directly related to the preferred orientation of the crystallographic planes which was validated with TEM and crystallographic texture studies. Along the top surface, there is no active slip system. However, twinning was observed. In the cross-section, active pyramidal slip system resulted in a plastic deformation. Additionally, the weak van der Waals forces between the layers caused crack propagation along the cross-section.

## 1. Introduction

Boron nitride (BN) occurs in several polymorphic forms such as cubic, hexagonal, rhombohedral and wurtzite [1]. Cubic boron nitride (c-BN) and hexagonal boron nitride (h-BN) are the most widely used structures due to their structural similarity to diamond and graphite, respectively [1, 2]. Hexagonal boron nitride is a 2D layered material like graphite, where the carbon atoms in the graphite are replaced by boron and nitrogen atoms alternate to each other. But, the stacking sequences are different from graphite for h-BN where the boron is above the nitrogen atom with AAA stacking sequence. The sp<sup>2</sup> hybridized B-N hexagonal unit forms a layer with the periodic arrangement, and stacked layers are held by weak van der Waals forces [3]. Hexagonal boron nitride exhibit excellent mechanical, thermal, electrical, dielectric and chemical properties, which makes it a potential candidate for various applications such as in lubrication, refractory materials, as a coating for electronic parts, and as fillers for polymers [4–7].

Ever since the emergence of graphene, other 2D layered materials are more deliberately explored due to their impressive properties [8]. As such, two-dimensional h-BN sheets, available in the form of boron nitride nanosheets (BNNS), have been investigated as a structural analog to graphene, (often referred to as white graphene) [9]. They are found in stable form as a single monolayer or few layers of thin sheets of h-BN. BNNS are isolectric, the boron and nitrogen atoms within the plane have strong covalent bonding, and thus have superior thermal, chemical and mechanical properties. The B-N atoms also exhibit partial ionic bonding both in-plane and out-of-plane which makes them an electrical insulator [10]. The BNNS edges are tunable to form either a zigzag B/N edge or a BN paired armchair edge [11].

BNNS are useful for many applications, the weak van der Waals bond between BN layers facilitates gliding of layers against each other which enables them to be used as an excellent solid lubricant. It has been previously reported that functionalized BN nanosheets dispersed in various oils exhibited enhanced lubrication compared to h-BN

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powder [12]. BNNS have also been used as a reinforcement in composite materials for improved mechanical properties. BNNS are reported to be stable in air up to 850 °C, thereby showing an increased resistance to oxidation over graphene, enabling their use in higher temperature fabrication techniques [13]. As examples, composites composed of amorphous borosilicate glass and BNNS have been synthesized as a composite using spark plasma sintering (SPS). BNNS reinforced composites showed about ~ 45% enhancement in the fracture toughness and flexural strength compared to amorphous borosilicate glass [14]. Ceramic composites of  $\text{Si}_3\text{N}_4$  with boron nitride nanosheets were prepared by hot isostatic pressing. Compared to monolithic  $\text{Si}_3\text{N}_4$ , ceramic composites ( $\text{Si}_3\text{N}_4/\text{BNNS}$ ) displayed an increase in fracture toughness especially with an addition of 5 wt% of BNNS [15]. Ceramic composite comprising of h-BN and magnesium aluminum silicate (MAS) have also been fabricated by hot pressing. The ceramic composite of 50 wt% of each resulted in an enhanced mechanical properties with bending strength of  $213 \pm 24$  MPa and fracture toughness of  $2.49 \pm 0.35$  MPa  $\text{m}^{1/2}$  [16]. BN nanosheets were also employed in polymers as nanofillers in polyvinyl butyral coatings, which showed better corrosion resistant properties [17]. BNNS have a wide range of additional applications including deep ultraviolet emitter, functional composites, self-cleaning coatings, optoelectronic and nanoelectronic devices based on their bonding and layer count [12, 18–20]. Although 2D-BNNS are finding application as reinforcement in composites, there is a poor understanding of the effect of their orientation on the mechanical properties. Previously, conventional h-BN were sintered by pressureless sintering, hot-press sintering or SPS method using a sintering aid or an additional process like slip casting [21–26]. The focus of those studies was largely to understand the effect of sintering conditions on the crystallographic texture of conventional h-BN [21–26]. Duan et al. [21, 22, 25] reported the fracture mechanisms of the textured conventional h-BN pellets as a function of orientation using flexure tests. Flexural strength was 3–4 times greater in the loading along the top surface as compared to the cross-section [22]. The zig-zag fracture was observed when loaded along the top surface as compared to crack propagation without any deflection in the samples loaded along the cross-section. It must be noted that all observations were made post-fracture [22, 25] and did not provide an understanding of the intermediate deformation behavior from loading until fracture.

In the present work, the 2D boron nitride nanosheets were consolidated by spark plasma sintering (SPS) at 1650 °C under a pressure of 50 MPa *without any sintering aids*. The major *novelty* of this work lies in

following facts: (i) using high aspect ratio 2D BNNS as starting powder instead of conventional h-BN which will lead to stronger texture structure and hence, altered mechanical properties and (ii) high load *in-situ indentation* study inside scanning electron microscope to understand the deformation behavior as a function of orientation in *real time*. None of the previous studies [21–26] has addressed deformation behavior in *real time*. Therefore, the major focus of this study is to understand deformation behavior of highly oriented monolithic 2D BNNS with respect to parallel and perpendicular direction of loading in *real time*.

## 2. Experimental details

### 2.1. Sample preparation

Boron nitride nanosheets used in this study were purchased from pH Matter LLC, Columbus, Ohio. The BNNS has a surface area of ~ 60  $\text{m}^2/\text{g}$  [27]. As-received BNNS was consolidated by spark plasma sintering (SPS, Thermal Technologies model 10–4, Santa Rosa, CA, USA) using a cylindrical graphite die of diameter 20 mm. As-received BNNS were sintered at 1650 °C for 20 min at the heating rate of 100 °C/min and uniaxial pressure of 50 MPa. After sintering, the pellet was polished to remove the graphite foil. The pellet was 3 mm in thickness.

### 2.2. Characterization

The density of the as-received BNNS powders was measured by Helium gas pycnometer (Accupyc 1340, Micrometrics Instrument Corporation, Norcross, GA, USA). For the BNNS SPS pellet, the relative density was measured by Archimedes method. Also, true density of the pellet was measured by Helium gas pycnometer. The phases present in the as-received powder and sintered pellet were characterized by X-ray diffraction (XRD, Siemens D-5000 X-ray diffractometer) with  $\text{Cu K}\alpha$  ( $\lambda = 1.542$  Å). The data were collected in the  $2\theta$  range of 10–90°, with the scan rate of 0.02°/min. For the in-depth analysis of crystallographic texture on the top surface and cross-section of the pellet, pole figure measurements were performed using Bruker D8 Discover X-ray diffractometer with  $\text{Cu K}\alpha$  radiation in Bragg-Brentano geometry and four circle goniometer equipped with automated X-Y-Z movements. The data were collected for 0–360° with a step of 3° in Phi ( $\varphi$ ) and tilting 0–75° with a step of 3° in the Psi ( $\psi$ ) range. The measured data were plotted and analyzed using Labotex texture analysis software [28].

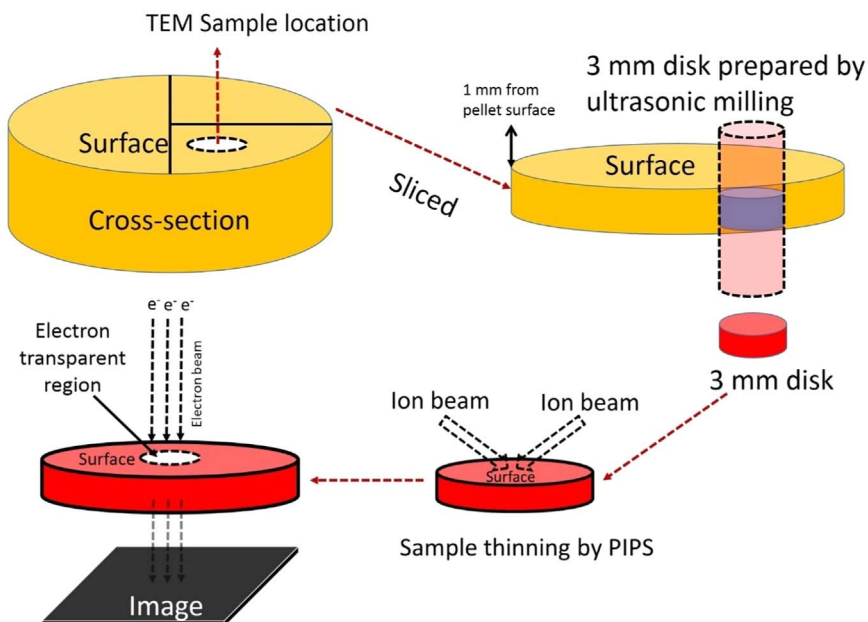


Fig. 1. Schematic showing the TEM sample preparation of sintered BNNS pellet.

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