

## Synthesis of Nanoscale Hexagonal Boron Nitride Sheets by Molten Salt Method

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### Abstract

Hexagonal boron nitride possesses a layered structure composed of six-membered rings similar to graphene, thereby exhibiting excellent mechanical properties and thermal conductivity, which has attracted widespread attention in numerous application fields. In response to the high energy consumption issues associated with hexagonal boron nitride preparation processes, this study aims to propose a greener, low-energy-consumption synthesis route. This work employs the molten salt medium method as the preparation approach. This method utilizes KCl-NaCl as the molten salt system, melamine (C<sub>3</sub>N<sub>6</sub>H<sub>6</sub>) and boric acid (H<sub>3</sub>BO<sub>3</sub>) as reactants, and introduces nitrogen gas (N<sub>2</sub>) as the reaction atmosphere. This research systematically investigated the influence of various conditions on the preparation of hexagonal boron nitride products by altering parameters such as the mass ratio of molten salt to reactants, the nitrogen-to-boron molar ratio in the reactants, the holding temperature, and the holding time. Through characterization and analysis of the obtained samples, the optimal preparation conditions were determined. Under the systematic investigation of this work, a mass ratio of molten salt to reactants of 2:1, a nitrogen-to-boron molar ratio of 1:2, and holding at 1000°C for 10 hours were found to be the optimal preparation conditions. Under these conditions, hexagonal boron nitride nanosheets with an average size of approximately 50 nm can be prepared. This demonstrates that under optimal process conditions, hexagonal boron nitride nanosheets with controllable particle size, uniform morphology, and good crystallinity can be synthesized. This work provides a novel pathway for the green and low-carbon synthesis of hexagonal boron nitride nanosheets.

## Full Text

### Preamble

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### Abstract

Hexagonal boron nitride (h-BN) possesses a lamellar structure composed of six-membered rings similar to graphene, thus exhibiting excellent mechanical properties and thermal conductivity that have attracted widespread attention across numerous applications. Addressing the high energy consumption issue in conventional h-BN preparation processes, this study proposes a greener, lower-energy synthesis route. The molten salt media method was employed using a KCl-NaCl eutectic system with melamine ( $C_3N_{6H}6$ ) and boric acid ( $H_3BO_3$ ) as reactants under a nitrogen ( $N_2$ ) atmosphere. Systematic investigation of various parameters—including the mass ratio of molten salt to reactants, the nitrogen-to-boron molar ratio in reactants, holding temperature, and holding time—revealed their effects on h-BN product formation. Characterization and analysis of the resulting samples identified optimal preparation conditions: a molten salt-to-reactant mass ratio of 2:1, a nitrogen-to-boron molar ratio of 1:2, and holding at 1000°C for 10 h. Under these conditions, hexagonal boron nitride nanosheets with an average size of approximately 50 nm were successfully prepared, demonstrating controllable particle size, uniform morphology, and good crystallinity. This work provides a novel green and low-carbon pathway for synthesizing h-BN nanosheets.

**Keywords:** Hexagonal boron nitride, Graphene, Molten salt, Low-carbon, Nanosheets

## Introduction

Hexagonal boron nitride features a hexagonal layered structure similar to graphene, with lattice constants of  $a = 0.2504$  nm and  $c = 0.6661$  nm, a theoretical density of  $2.27 \text{ g} \cdot \text{cm}^{-3}$ , and a melting point of  $3000^\circ\text{C}$ , commonly known as “white graphite” [1]. In its structure, each layer consists of nitrogen (N) and boron (B) atoms interconnected to form six-membered rings. The N and B atoms within each layer are tightly bound by strong  $\text{sp}^2$  covalent bonds, while interlayer connections rely on weaker van der Waals forces [2-6]. This unique hexagonal ring structure endows h-BN with exceptional properties including high lubricity, thermal conductivity, heat resistance, oxidation resistance, corrosion resistance, machinability, and chemical stability [7], leading to widespread applications in thermal management, metallurgy, catalysis, lubrication, insulation, hydrogen storage, electronics, high-temperature anti-oxidation coatings, and aerospace [8-12].

High-temperature solid-state sintering and “top-down” approaches represent the primary methods for preparing h-BN nanosheets [13-14]. Traditional high-temperature solid-state sintering is an important industrial route, most commonly using borax and urea (or ammonium chloride) as raw materials reacted in ammonia or nitrogen atmospheres at high temperatures [15-16]. Although this method is technologically mature, it requires prolonged high-temperature sintering, resulting in high energy consumption, low efficiency, and products that are typically large crystalline flakes with non-uniform particle size distributions. Li et al. [17] prepared h-BN particles approximately  $5 \mu\text{m}$  in diameter using the boric acid-urea method, while Zhang et al. [18] synthesized highly crystalline h-BN by mixing borax with urea, ammonium chloride, and melamine followed by high-temperature sintering.

“Top-down” methods involve exfoliating or breaking down bulk BN into nanosheets through external forces, including mechanical, chemical, and gas exfoliation, as well as ball milling and ultrasonication. While effective for preparing h-BN nanosheets, these approaches introduce impurities through added chemical reagents or grinding media during the breakdown process, limiting practical applications. Jiang et al. [19] exfoliated  $200 \text{ nm}$  BN nanosheets with thickness less than  $5 \text{ nm}$  from h-BN using a radiation-induced reduction-exfoliation method in aqueous alcohol solution. Goto et al. [20] exfoliated larger h-BN nanosheets using cavitation-bubble plasma in water, while Ghosh et al. [21] obtained h-BN nanosheets ranging from  $50\text{-}270 \text{ nm}$  in thickness and  $1\text{-}3 \mu\text{m}$  in lateral dimensions via liquid-phase exfoliation. Yan et al. [22] used starch as a ball-milling aid to exfoliate h-BN nanosheets with an average size of  $5 \mu\text{m}$ .

The molten salt method has attracted considerable attention in recent years as it enables reactions in molten inorganic eutectic salt media at relatively low temperatures. The homogeneous reaction environment provided by the molten salt’s solubility for reactants at high temperatures significantly enhances contact

efficiency, improves reaction kinetics, and maintains process stability. Metin et al. [23] synthesized ~50 nm nanosheets using  $H_3BO_3$  and  $NH_4Cl$  as reactants with a KCl-NaCl molten salt system. Ye et al. [24] prepared highly crystalline h-BN nanosheets via molten-salt-mediated magnesiothermic reduction of  $Na_2B_4O_7$  and Mg powder at 1200°C under  $N_2$  protection. Tian et al. [25] synthesized h-BN nanosheets at 900–1200°C using a NaCl-KCl molten salt medium with borax and melamine as raw materials through molten salt nitridation.

This work employs a nitridation method in molten salt media to prepare nano-hexagonal boron nitride. Systematic investigation of the effects of molten salt-to-reactant mass ratio, nitrogen-to-boron molar ratio in reactants, holding temperature, and holding time on h-BN synthesis enabled the preparation of nanoscale h-BN with controllable particle size and uniform morphology under optimal conditions.

## Experimental

### Reagents and Materials

Boric acid (AR, \$ 99.5%), melamine (CP, \$ 99.0%), potassium chloride (CP, \$ 99.5%), and sodium chloride (CP, \$ 99.5%) were purchased from Sinopharm Chemical Reagent Co., Ltd. Alumina crucibles (HERBED, 99% alumina square crucibles) were used as reaction containers.

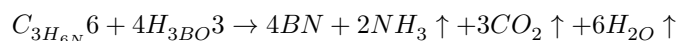
### Equipment

A horizontal tube furnace (HWGL-1200, Suzhou Hateng Technology Co., Ltd.), forced-air drying oven, X-ray diffractometer (X' Pert Powder, Malvern Panalytical), field-emission scanning electron microscope (FIB-SEM, Zeiss Crossbeam 540, Carl Zeiss), and simultaneous thermal analyzer (STA 449 F3) were employed for synthesis and characterization.

### Experimental Procedure

Boric acid and melamine were thoroughly mixed at a specified molar ratio to prepare the reactants. The KCl-NaCl molten salt system was prepared at a 1:1 molar ratio. The reactants and molten salt were uniformly mixed at a predetermined mass ratio and placed in an alumina crucible. The crucible was inserted into a horizontal tube furnace, the tube was sealed, and  $N_2$  gas was introduced. The mixture was heated at a controlled rate to the target temperature, held for the specified duration, and then cooled. Ammonia in the exhaust gas was absorbed by a dilute sulfuric acid scrubber. After cooling, the product was washed multiple times with deionized water to remove the molten salt. The filtered cake was dried in a forced-air oven at 120°C for 20 h prior to characterization. Experimental parameters including the B-source to N-source molar ratio, reactant-to-molten-salt mass ratio, holding temperature, and holding time

were systematically varied to characterize the resulting products. The reaction proceeds according to the following equation:



### Reaction Mechanism

The KCl-NaCl molten salt system forms a low-melting-point eutectic mixture (melting point 657°C), providing a liquid-phase reaction environment that reduces mass transfer resistance, promotes reactant diffusion and contact, and suppresses high-temperature agglomeration. Boric acid ( $H_{3BO}3$ ) dehydrates upon heating to form boron oxide ( $B_{2O}3$ ), while melamine ( $C_{3H_6N}6$ ) decomposes at high temperatures to generate nitrogen-containing active gases (such as  $NH_3$  and HCN) that supply nitrogen atoms for nitridation. During the initial heating stage (<300°C), boric acid and melamine react to form melamine borate ( $C_{3H_6N}6 \cdot 2H_{3BO}3$ ), a stable precursor that ensures uniform distribution of boron and nitrogen atoms. In the molten salt medium,  $B_{2O}3$  reacts with nitrogen atoms to generate intermediate boron nitride (BN) nuclei. The formed BN dissolves and precipitates in the molten salt, growing preferentially along the (002) crystal plane to develop a hexagonal layered structure. The key functions of the molten salt include: (1) providing a homogeneous liquid environment for reaction, avoiding the incompleteness of solid-phase reactions; (2) molten salt ions adsorbing on BN crystal nuclei surfaces to guide two-dimensional growth and form nanosheet structures, acting as a template; and (3) encapsulating reaction byproducts (such as  $H_2O$  and CO) that are subsequently removed by water washing after cooling, thereby improving product purity.

### Sample Characterization

An X' Pert Powder X-ray diffractometer (XRD) was used to analyze the crystal structure of samples, with characteristic diffraction peaks compared against standard h-BN patterns. A Zeiss Crossbeam 540 field-emission scanning electron microscope (SEM) was employed to observe sample morphology and measure particle sizes. Size measurements were performed using the SEM scale function on at least 100 particles per sample for statistical analysis to determine average particle diameter. A STA 449 F3 simultaneous thermal analyzer (TG) was used to evaluate chemical stability, with stability assessed based on heating curves up to 900°C to identify weight loss/gain points and determine whether oxidation temperatures matched those of h-BN and to quantify weight loss percentages.

## Results and Discussion

### Effect of Molten Salt-to-Reactant Ratio

Reactants were prepared with a melamine-to-boric acid molar ratio of 1:3 (corresponding to a nitrogen-to-boron elemental molar ratio of 2:1). The KCl-NaCl

molten salt was mixed with reactants at mass ratios of 4:1, 3:1, 2:1, 1:1, and without molten salt. The mixtures were placed in alumina crucibles and reacted in a horizontal tube furnace at 500°C for 1 h, followed by holding at 1000°C for 4 h. Figure 1 [Figure 1: see original paper] shows the X-ray diffraction patterns of the BN products prepared under different molten salt ratios, while Figure 2 [Figure 2: see original paper] presents the corresponding SEM morphological analysis.

As shown in Figure 1, all five molten salt ratio conditions produced a prominent diffraction peak at 25°–28° and a relatively weak peak at 41°–44°. The former corresponds to the characteristic (002) crystal plane of h-BN, while the latter represents the underdeveloped (100) and (101) planes. A characteristic (110) plane diffraction peak appears at 75°–77°. This pattern is consistent with turbostratic BN (t-BN) reported in literature [26], an intermediate state between amorphous BN and h-BN, analogous to the transformation of amorphous carbon to graphite at high temperatures [27]. The XRD patterns reveal that when the molten salt-to-reactant mass ratio is 2:1, the diffraction peaks exhibit the highest intensity, with a relatively distinct (004) plane characteristic peak appearing between 50°–60° and no impurity peaks, indicating the highest crystallinity.

SEM analysis in Figure 2 shows that without molten salt (Figure 2a), the sample obtained at 1000°C for 4 h consists of agglomerated large BN particles, similar to the t-BN morphology reported in literature [26]. When the molten salt-to-reactant mass ratio increases to 1:1 (Figure 2b), numerous sheet-like BN structures appear with diameters of 200–700 nm and thicknesses under 20 nm, demonstrating improved BN crystal growth. At a mass ratio of 2:1 (Figure 2c), the sample exhibits the best morphology with sheet-like BN diameters of 200–400 nm, thicknesses under 10 nm, and a higher quantity of more uniformly sized sheets, representing the most favorable conditions for BN growth. Further increases in the molten salt-to-reactant mass ratio did not effectively improve sample morphology.

Comprehensive analysis of XRD patterns and SEM images indicates that a molten salt-to-reactant mass ratio of 2:1 yields sheet-like BN with the most uniform size and optimal crystallinity.

### Effect of Holding Temperature and Time

Based on the above results, a molten salt-to-reactant mass ratio of 2:1 produced large sheet-like structures rather than smaller nanoscale h-BN. Therefore, holding temperature and duration were varied to investigate their effects on h-BN formation. For comparison, experiments fixed the nitrogen-to-boron molar ratio at 2:1 and the molten salt-to-reactant mass ratio at 2:1, with holding temperatures of 800°C and 1000°C for durations of 4 h and 10 h. The XRD patterns and SEM images of the resulting samples are shown in Figures 3 and 4 [Figure 3: see original paper][Figure 4: see original paper].

The XRD patterns in Figure 3 show that increasing the holding temperature

from 800°C to 1000°C for 4 h slightly enhanced the intensity of the (002) peak and the mixed (100) and (101) peaks. Comparing Figures 3b and 3d reveals that extending the holding time at lower temperature effectively improves BN growth, with the 800°C/10 h sample showing significantly enhanced characteristic peak intensity, indicating that longer holding times can compensate for lower temperatures. The highest intensity characteristic diffraction peaks with resolved (100) and (101) peaks were obtained at 1000°C for 10 h, approaching the characteristic pattern of h-BN.

SEM analysis in Figure 4 shows that 800°C for 4 h produced no sheet-like structures, maintaining the t-BN morphology. At 1000°C for 4 h, larger sheet-like structures of 200–600 nm with non-uniform sizes appeared. Extending the holding time to 10 h at 800°C yielded results similar to the 1000°C/4 h sample. However, holding at 1000°C for 10 h produced finer, more uniform sheet-like structures with sizes uniformly controlled below 100 nm, consistent with nanoscale h-BN morphology. These results demonstrate that extended holding times can compensate for lower reaction temperatures, though the time cycle becomes excessively long. Longer holding times at higher temperatures more effectively enhance BN growth, yielding uniform nanoscale h-BN products.

### Effect of Nitrogen-to-Boron Ratio

Based on previous results, h-BN nanosheets with uniform size were prepared at a molten salt-to-reactant mass ratio of 2:1, 1000°C, and 10 h. Therefore, these conditions were fixed while varying the nitrogen-to-boron molar ratio to 1:2, 1:1, 2:1, 3:1, and 4:1 to investigate its effect on h-BN formation. The XRD and SEM results are presented in Figures 5 and 6 [Figure 5: see original paper][Figure 6: see original paper].

Figure 5 shows that a nitrogen-to-boron molar ratio of 2:1 (Figure 5c) yields the lowest characteristic diffraction peak intensity, indicating the poorest crystallinity. As the nitrogen-to-boron ratio decreases, the characteristic peak intensity progressively increases. At a ratio of 1:2 (Figure 5a), the peaks exhibit the highest intensity, sharp shape, and smallest half-peak width, with the most pronounced separation of (100) and (101) peaks, indicating optimal crystallinity. At higher ratios of 3:1 and 4:1 (Figures 5d and 5e), peak intensity increases but remains lower than that at 1:2, with broader peak shapes. The XRD patterns in Figures 5d and 5e show a distinct leftward shift of the main diffraction peak at 20°–30°, attributed to the t-BN to h-BN transition, where all t-BN diffraction peaks shift slightly leftward. This suggests that increasing nitrogen-to-boron ratios are detrimental to h-BN formation. XRD analysis indicates that a nitrogen-to-boron molar ratio of 1:2 is optimal.

SEM results in Figure 6 reveal that at a nitrogen-to-boron ratio of 1:2, the h-BN sheet structures are approximately 50 nm in size with the most uniform distribution. At a 1:1 ratio, the morphology is similar to the 1:2 ratio. As the nitrogen-to-boron ratio increases, the nanosheets gradually enlarge. At a 2:1

ratio, the sheet size is nearly double that of the 1:2 sample; at 3:1, sheet dimensions reach 100–200 nm with noticeably non-uniform sizes; at 4:1, the sheet structures increase further, with more numerous 200–400 nm sheets. Based on XRD and SEM analysis, uniform h-BN nanosheets are obtained at a molten salt-to-reactant mass ratio of 2:1, 1000°C for 10 h, and a nitrogen-to-boron molar ratio of 1:2. Particle size distribution was measured for 100 particles from the Figure 6a sample, with results shown in Figure 7 [Figure 7: see original paper]. Ninety-six particles were under 50 nm, with only four exceeding 50 nm, demonstrating a relatively uniform and controllable size distribution. Comparing all samples in Figure 6 reveals that during the transformation to h-BN, t-BN first forms a matrix with large 200–400 nm sheet structures. As the reaction proceeds, BN single nuclei in the t-BN separate to form smaller 100–200 nm sheets. Continued reaction transforms the crystal structure toward h-BN, ultimately yielding uniform, single-nucleus h-BN nanosheets.

### Thermogravimetric (TG) Analysis

TG analysis was performed to examine potential volatile impurities in the prepared BN nanosheets (Figure 8 [Figure 8: see original paper]). Curve a corresponds to the weight loss profile of BN powder prepared at a nitrogen-to-boron ratio of 2:1, molten salt-to-reactant mass ratio of 2:1, and 1000°C for 4 h. Curve b corresponds to h-BN powder prepared at a nitrogen-to-boron ratio of 1:2, molten salt-to-reactant mass ratio of 2:1, and 1000°C for 10 h. Both samples show weight loss within  $\pm 2\%$ , indicating BN purity exceeding 98%. Below 700°C, curve b exhibits more pronounced weight loss than curve a, suggesting higher volatile impurity content in product b, likely introduced during the extended 10 h high-temperature holding process. Both curves show an upward trend at 700°C with an inflection platform at 800°C, attributed to surface oxidation of a small amount of BN forming boron oxide. This oxidation temperature is consistent with h-BN oxidation temperatures [28] and higher than that of graphene [29], confirming the prepared samples as h-BN. Comparison of the two TG curves shows that h-BN exhibits a greater weight loss of approximately 2%.

### Conclusion

Using melamine as the nitrogen source, boric acid as the boron source, and KCl-NaCl as the molten salt system under  $N_2$  atmosphere, uniform and size-controllable nano-hexagonal boron nitride was successfully prepared. Systematic investigation of the molten salt-to-reactant mass ratio, holding temperature and time, and nitrogen-to-boron molar ratio revealed that optimal conditions of 2:1 mass ratio, 1:2 nitrogen-to-boron ratio, and 1000°C for 10 h produce highly uniform  $\sim 50$  nm h-BN nanosheets. By simply adjusting the nitrogen-to-boron ratio, h-BN nanosheets with controllable sizes of 100–200 nm and 200–400 nm can be prepared. Compared with conventional h-BN preparation processes, this method operates at temperatures not exceeding 1000°C, significantly lower than

industrial production temperatures of 1200–1800°C. The homogeneous reaction medium provided by the molten salt effectively reduces synthesis temperature [22–23], directly decreasing energy consumption. The prepared h-BN exhibits controllable particle size and uniform morphology, providing a reliable experimental basis for green, low-carbon, large-scale industrial production of BN nanosheets.

## Author Contributions

HUANG Yucan conducted the experiments and was responsible for drafting, writing, and revising the manuscript. SHEN Jiajie provided experimental guidance and manuscript review. GUAN Chengzhi provided financial support and experimental guidance. LONG Dewu designed the experimental framework, provided guidance, and reviewed and revised the manuscript. CHEN Bangfu and YANG Aohua contributed suggestions and assistance from their respective areas of expertise.

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