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From Sheets to Tubes: A Review of Boron Nitride Nanostructure Synthesis

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Abstract

Boron nitride (BN), along with its one-dimensional (1D) and two-dimensional (2D) nanostructures, has seen a surge in interest in both academic research and practical applications. These nanomaterials have emerged as highly promising fillers for polymer-based nanocomposites, significantly enhancing their properties. Most of the studies reviewed in this paper begin with hexagonal boron nitride (h-BN) as the base material, which is then used to produce its 2D form, boron nitride nanosheets (BNNS), and its 1D counterpart, boron nitride nanotubes (BNNTs). This review aims to explore the fundamental properties of h-BN, followed by a focused discussion on the synthesis and characteristics of BNNS and BNNTs. The paper concludes by summarizing the most effective synthesis approaches for each material, highlighting their potential while considering current challenges in the field.

Keywords: *Boron nitride nanotubes, Boron nitride nanosheets, h-Boron nitride(h-BN), Synthesis.*

1. Introduction

Nanoscience focuses on modifying materials at the nanoscale to unlock enhanced properties at the macroscopic level. These nanoscale structures have shown remarkable potential across a range of properties mechanical, electrical, optical, and thermal making them suitable for a variety of high-performance applications [1]. Among the wide range of nanomaterials being studied, boron nitride (BN) stands out as one of the most promising, both in research and industry. Structurally like graphite, BN is made up of equal numbers of boron (B) and nitrogen (N) atoms. However, its bonding characteristics are quite distinct: The B and N atoms form strong sigma bonds [2,3] through sp^2 hybrid orbitals, resulting in a partially ionic character. This leads to weak van der Waals forces between layers and gives BN its unique anisotropic properties. BN nanomaterials can also be classified based on their dimensional structure: zero-dimensional (0D) forms like nanospheres, one-dimensional (1D) structures such as nanotubes, nanoribbons, and nanofibers, and two dimensional (2D) formats including thin films and nanosheets. Hexagonal boron nitride (h-BN) is well known for its exceptional thermal stability, maintaining its structural integrity even at elevated temperatures. In addition to its thermal resilience, h-BN is an outstanding electrical insulator even under extreme conditions and high temperatures [4,5,6] Hexagonal Boron Nitride Various nanoscale forms can be derived from bulk BN, including boron nitride ribbons, nanotubes, nano powders, and nanosheets. This review focuses specifically on the integration of boron nitride nanosheets (BNNS) and boron nitride nanotubes (BNNT) to develop transparent nanocomposites.

2. Boron Nitride Nano Sheets (BNNS)

BNNS are the two-dimensional (2D) nanostructures derived from hexagonal boron nitride (h-BN), They are often considered analogues to graphene due to their layered structure and exceptional properties [6]. BNNS can be synthesized in bulk using two primary approaches:

- Top-down methods such as micro milling of h-BN followed by sonication-assisted exfoliation.
- Bottom-up methods like chemical vapor deposition (CVD), which can be performed with or without a substrate.

Key parameters to control during BNNS synthesis include the size, shape, thickness, density, and alignment of the sheets all of which directly affect the performance of the final nanocomposite. (Figure 1)

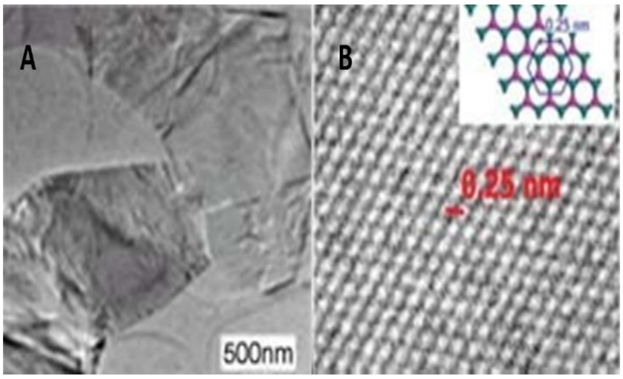

Figure 1 BNNS Sample Under SEM

3. Boron Nitride Nanotubes (BNNT)

Boron nitride nanotubes (BNNTs) as a more stable alternative [7]. Structurally, BNNTs are like CNTs, consisting of a hexagonal network where boron and nitrogen atoms replace carbon atoms, forming an sp^2-hybridized trigonal planar structure.

BNNTs were first observed with inner diameters ranging from 1 to 3 nm, lengths up to 200 nm, and an energy band gap of approximately 5.5 eV. Unlike CNTs, the properties of BNNTs are largely independent of tube diameter, chirality, or the number of concentric layers. They also offer excellent resistance to oxidation, even at elevated temperatures.

4. Synthesis of BNNTs

One of the earliest methods developed for synthesizing boron nitride nanotubes (BNNTs) was the arc discharge technique, where BNNTs were grown using a setup that employed a cooled copper substrate, and a tungsten electrode packed with boron nitride.

4.1. Thermal Annealing and Chemical Vapor Deposition

In BOCVD, a vertical induction furnace is used. A boron nitride crucible containing a mixture of boron and magnesium oxide is placed at the base and heated to around 1300°C. Under these conditions, boron oxide and magnesium vapours are generated [8,9]. These vapours then react with ammonia gas in an argon-controlled atmosphere at relatively lower temperatures, promoting the formation of high-purity BNNTs by separating boron from boron oxide during the reaction.

4.2. Catalytic Chemical Vapor Deposition (CCVD) and Growth Vapor Trapping (GVT)

Lee et al. developed a successful method for synthesizing high-purity BNNTs using a conventional resistive tube furnace at 1200 °C. This technique uses catalytic chemical vapor deposition (CCVD) and introduces a novel concept known as the growth vapor trapping (GVT) approach. In this method, a closed-end quartz test tube is employed to trap the growth vapours, enhancing the conditions for BNNT formation [10]. In addition to producing high-quality BNNTs at predefined locations, the GVT method is noted for its simplicity and reproducibility, making it accessible to many research laboratories.

Moreover, the BNNT films produced using this technique exhibit superhydrophobic properties.

4.3. Laser Vaporization and Pressurized Vapor/Condenser (PVC) Method

This catalytic PLD approach enabled the synthesis of BNNTs with diameters around 10–20 nm, even at relatively low substrate temperatures (~600 °C). [11,12] A breakthrough came in 2009, with the development of the high temperature/high-pressure (HTP) method, often referred to as the pressurized vapor/condenser (PVC) technique.

As these droplets rise and interact with nitrogen, BNNTs rapidly form.

The typical production rate for this process ranges from 20 to 120 mg/h, and the resulting BNNTs are often found as bundled tubes or entangled fibril networks.

4.4. Large-Scale Synthesis Using Inductive Thermal Plasma

In this process, solid h-BN powder is introduced along with nitrogen and hydrogen gases into a high-temperature induction plasma operating above 8000 K at atmospheric pressure. The extreme temperatures decompose all precursors into elemental forms (B, N, and H), and as the stream cools rapidly at a gradient of 105 K/s, nano-sized boron droplets form downstream. These droplets act as nucleation sites for BNNT growth [13]. Hydrogen plays a critical catalytic role by forming intermediate H–B–N species, which help prevent the recombination of nitrogen radicals, promoting efficient BNNT formation.

5. Synthesis of Boron Nitride Nano Sheets (BNNS)

BNNS can be synthesized through both mechanical and chemical exfoliation methods, and to a lesser extent, through chemical vapor growth. One early approach involved the use of borazine to grow a BNNS-like structure directly on a metal substrate [14].

5.1.Mechanical Exfoliation

Mechanical exfoliation relies on applying physical forces to separate h-BN layers, leveraging the weak van der Waals interactions between them.

- Scotch tape exfoliation, one of the earliest techniques, involves peeling thin layers from bulk BN crystals [15].
- Ultrasonication-assisted exfoliation is another common approach, where BN powder is dispersed in a solvent and ultrasonicated to produce thin BNNS layers [16].
- Ball milling is a particularly effective method for exfoliating h-BN into few-layered nanosheets. A commonly used strategy is combining h-BN with urea, whose small molecules penetrate interlayer spaces, improving exfoliation efficiency [17].
- The thickness of the resulting BNNS directly impacts their properties and is heavily influenced by both the method and duration of the exfoliation process.
- Despite these limitations, mechanical exfoliation remains a cost-effective and scalable method, particularly when optimizing milling conditions and choosing appropriate dispersing agents.

5.2. Liquid Exfoliation

Liquid-phase exfoliation is a widely adopted method for synthesizing BNNS by dispersing bulk h-BN in various solvents. Researchers have explored a range of solvent systems to achieve effective exfoliation, including 1,2-dichloroethane [19], deionized water [20], sodium dodecyl sulphate (SDS)–water mixtures [21], N,N′-dimethylformamide (DMF), sulfonic acid, and methane sulfonic acid [22].

The choice of solvent plays a crucial role in determining the exfoliation efficiency and yield of BNNS. Among the various solvents studied, isopropyl alcohol (IPA) has proven to be particularly effective. IPA's surface energy closely matches that of boron nitride nanosheets, which significantly reduces the van der Waals interactions between the h-BN layers. [23].

5.3.Solvent and Surfactant Considerations in BNNS Exfoliation

This method allows for the controlled synthesis of high-quality monolayer films. When considering solvent-based exfoliation methods, many potential solvents are excluded due to limitations such as high toxicity, high boiling points, or prohibitive cost. Among the various findings, it was observed that surfactants play a key role in stabilizing aqueous dispersions, although they contribute minimally to the actual exfoliation process itself [24]. Among all solvents studied, isopropyl alcohol (IPA) consistently shows the highest efficiency for BNNS exfoliation. This is largely attributed to its surface energy being well-matched to that of h-BN, which minimizes van der Waals forces and enables effective delamination [24]

Conclusions

Hexagonal boron nitride (h-BN) and its nanoscale derivatives boron nitride nanosheets (BNNS) and boron nitride nanotubes (BNNTs)—have emerged as highly promising nanomaterials due to their exceptional thermal stability, chemical inertness, mechanical strength, electrical insulation, and optical transparency. These properties make BNNS and BNNTs ideal candidates for applications across electronics, thermal management, aerospace, sensors, energy storage, water purification, and biomedical technologies. This review has explored and compared a wide range of synthesis techniques for BNNS and BNNTs, emphasizing the advantages and limitations of each. For BNNTs, methods such as arc discharge, ball milling, chemical vapor deposition (CVD), laser vaporization (HTP/PVC), and inductively coupled plasma (ICP) synthesis have been critically analyzed. While CVD and plasma-based approaches offer high purity and scalability, challenges remain in achieving consistent yield, structural control (diameter, chirality), and cost-effective mass production. Recent innovations, such as the GVT-CCVD approach and the use of EPIC systems, have improved process control and quality, yet industrial-scale production still faces technical and economic barriers. For BNNS, both mechanical and liquid-phase exfoliation methods have been investigated. Techniques such as ball milling with urea, ultrasonication, and shear-assisted exfoliation have shown significant promise in producing few-layered BNNS. Solvent selection is critical, with isopropyl alcohol (IPA) emerging as the most effective due to its surface energy compatibility with h-BN. The role of surfactants and polymers has been explored for dispersion stability, though difficulties in post-synthesis purification remain a significant drawback. Furthermore, chemical vapor deposition (CVD) has enabled the epitaxial growth of high-quality monolayer h-BN films on metal substrates such as Ni, Cu, Pt, and Ru, offering precision and scalability for electronic and optoelectronic applications. Among all techniques studied, CVD stands out for achieving high structural precision and purity for both BNNTs and BNNS, though it requires high temperatures, complex setups, and controlled atmospheres. On the other hand, exfoliation techniques offer simplicity and scalability, particularly for BNNS, but often at the expense of structural uniformity and purity. In conclusion, the choice of synthesis method must be guided by the specific application requirements, including morphology, purity, yield, cost, and scalability. While significant progress has been made over the last decade, further research is needed to overcome the challenges of large-scale, cost-effective production, especially for BNNTs. Optimization of precursors, catalysts, solvent systems, and reactor designs will be crucial in advancing both lab-scale research and industrial deployment of BN-based nanomaterials.

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