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Preparation and Characterization of Hexagonal Boron Nitride and its Applications.

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ABSTRACT:

Hexagonal boron nitride (h-BN) is an advanced ceramic material with considerable potential for use as a dielectric and electrically insulating layer. Its crystal structure consists of alternating boron and nitrogen atoms arranged in a two-dimensional hexagonal network resembling that of graphite. The material exhibits several desirable characteristics, including excellent thermal resistance, chemical inertness, low density, and a large electronic band gap. These properties make it suitable for applications in electronic insulation, thermal regulation, protective surface coatings, and ceramic-based composite materials. In this investigation, boron nitride powder is obtained using boric acid (H_3BO_3) and urea [$CO(NH_2)_2$] as precursor materials, followed by heat treatment in a nitrogen-rich environment. The prepared samples are examined using different characterization methods, namely X-ray diffraction (XRD), Scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR), and Differential thermal analysis (DTA).

The present study also describes the fabrication of hexagonal boron nitride thin films through atmospheric-pressure chemical vapour deposition (APCVD), employing copper foil as the growth substrate. The copper layer is removed by chemical etching, and the resulting h-BN film is transferred onto the required substrate. The thickness of the deposited films can be varied by regulating the deposition time and modifying the synthesis parameters. Parallel-capacitance measurements indicate that the dielectric constant of the prepared h-BN film is approximately 2.4. Furthermore, the CVD-fabricated h-BN layers are incorporated into top-gated graphene devices, where they serve as dielectric materials.

KEYWORDS: Hexagonal boron nitride, chemical vapour deposition, X-ray diffraction, Scanning electron microscopy, Differential thermal analysis.

1. INTRODUCTION

Hexagonal boron nitride (h-BN) is an important III–V compound material that has gained substantial research interest because of its structural resemblance to graphene and its distinctive physical, chemical, and functional properties. Its notable characteristics include ultraviolet emission [1–2], outstanding mechanical performance, high thermal stability, promising optoelectronic behaviour, and excellent resistance to chemical degradation [3–5]. Structurally, h-BN consists of stacked atomic layers similar to graphene, in which boron and nitrogen atoms are alternately connected through sp^2 covalent bonding, resulting in a well-defined hexagonal arrangement within each layer.

Because of its superior lubricating behaviour, boron nitride is considered an effective replacement for conventional lubricating materials employed in metal-working and processing operations. In addition, its

non-toxic nature, chemical stability, and electrically insulating characteristics make it suitable for several technological applications. Boron nitride can crystallize in different structural forms that are electronically comparable to the various forms of carbon. For example, cubic boron nitride possesses a diamond-like arrangement, although its mechanical hardness is comparatively lower than that of diamond, while it offers excellent thermal stability. Among the different crystalline modifications, the hexagonal structure is the most widely studied and commonly occurring form. Due to its favorable physical and chemical characteristics, h-BN has found applications in solid lubricants, cosmetic formulations, and various industrial materials.

Single-layer hexagonal boron nitride is also an important two-dimensional material that has attracted attention because of its interesting predicted physical effects, including flexoelectric behaviour [6]. An especially significant application of h-BN is its use as a supporting substrate for graphene-based electronic structures. Research findings indicate that graphene exhibits considerably improved electronic transport characteristics when deposited on h-BN rather than on conventional silicon dioxide (SiO_2) supported by silicon substrates. In particular, for comparable materials, including those produced through chemical vapour deposition (CVD), the carrier mobility measured using h-BN as the supporting substrate has been reported to be approximately ten times greater than that obtained with SiO_2 on Si substrates [7–8]. Therefore, the availability of high-quality hexagonal boron nitride is an important requirement for the development of efficient graphene-based electronic devices, creating a need for improved and reliable synthesis procedures.

Several experimental approaches have been developed for preparing hexagonal boron nitride, including carbothermal nitridation [9–11], chemical vapour deposition [12], thermal decomposition of precursor compounds [13–14], microwave-assisted preparation, and controlled heat-treatment processes [15–18]. Different film-transfer procedures have also been investigated to facilitate the deposition of h-BN onto various functional substrates [19]. In recent years, particular attention has been given to the fabrication of h-BN films by chemical vapour deposition using copper and nickel substrates [20]. In these synthesis processes, ammonia borane ($H_3B–NH_3$) and borazine ($B_3N_3H_6$) are frequently selected as the source materials. By appropriately adjusting the deposition temperature, precursor concentration, and other growth parameters, it is possible to obtain either single-layer or multiple-layer h-BN films. These deposited films can subsequently be transferred to different substrates according to the requirements of the intended application.

In the present work, hexagonal boron nitride thin films having an approximate thickness of 20 nm are fabricated on copper foil through atmospheric-pressure chemical vapour deposition, with borazine ($B_3N_3H_6$) serving as the precursor. After completion of the deposition process, the copper substrate is removed through chemical etching, and the resulting h-BN films are transferred onto the desired supporting substrates. The prepared films are subsequently utilized as dielectric layers in top-gated graphene-based electronic devices. The investigation primarily focuses on controlling the thickness of h-BN films by varying the deposition duration and adjusting the corresponding growth conditions. The combination of atmospheric-pressure CVD and a suitable transfer process offers a convenient and effective method for incorporating h-BN thin films into graphene-based electronic devices.

2. Materials and Methods

High-purity analytical-grade boric acid (H_3BO_3) and urea [$CO(NH_2)_2$] were used as starting materials, along with distilled water and high-purity nitrogen gas. The molar proportions of the precursors were carefully determined and maintained throughout the preparation process. A boric acid-to-urea molar ratio of 1:5 was considered, depending on the furnace conditions, nitrogen flow rate, quantity of starting materials, and required phase purity. The corresponding quantities of both precursors were calculated according to the selected molar ratio.

Initially, boric acid and urea were dissolved in distilled water under continuous stirring. The solution was heated to approximately 70°C and maintained under stirring for about 2 hours to ensure proper mixing and uniformity. Subsequently, the solvent was removed through evaporation to obtain a homogeneous precursor mixture, which was further dried until a constant weight was achieved.

The dried precursor was then placed in an appropriate covered alumina or boron-nitride-compatible crucible and subjected to thermal treatment in a tube furnace under a continuous flow of nitrogen gas. According to a previously reported preparation method, the initial heat-treatment temperature was approximately 650°C. The heating rate, holding duration, nitrogen flow rate, and any additional annealing conditions were selected according to the experimental requirements. After completion of the thermal treatment, the furnace was allowed to cool under a nitrogen atmosphere before the synthesized powder was collected, the obtained product was subjected to washing and subsequent drying using an appropriate and validated purification procedure. Finally, the product yield was determined, and the prepared boron nitride powder was preserved in a dry, airtight container for further characterization.

3. Characterization Techniques

3.1. Scanning Electron Microscopy (SEM) Analysis:

Nanoparticles synthesized through nanoprecipitation or solvent-evaporation techniques generally exhibit spherical morphology because surface tension tends to minimize their surface area. Such preparations may also display a particle-size distribution. In contrast, nanoparticles synthesized from inorganic materials can develop different morphological structures, including cubic, rod-like, disc-shaped, and star-like forms. The final particle morphology is often influenced by the nature of the inorganic constituents and the auxiliary materials employed during synthesis.

The surface morphology and structural features of the synthesized boron nitride (BN) powder and its nanoparticles were examined using scanning electron microscopy (SEM). The corresponding SEM micrographs of boron nitride and nanoparticles prepared in the present investigation are presented in Figure (1).

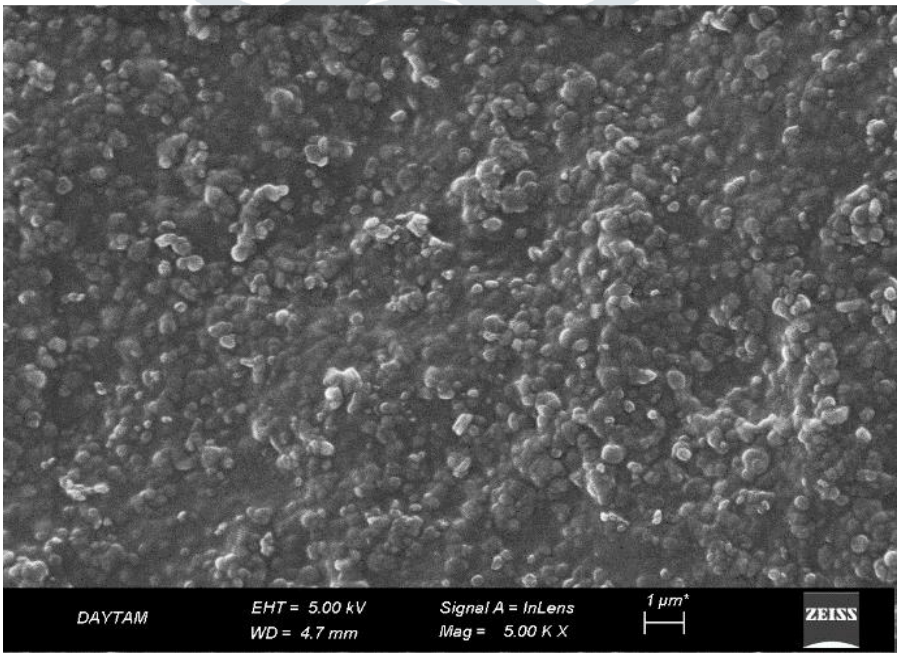

Figure (1) shows the SEM image of Hexagonal Boron Nitride.

The SEM images revealed that the bulk boron nitride sample consisted of irregularly shaped, amorphous solid particles. In contrast, the boron nitride nanoparticles exhibited a uniform three-dimensional morphology with a circular appearance rather than a spherical shape. Furthermore, comparison of the particle dimensions observed in the images with the corresponding measurements indicated good agreement in terms of particle size.

3.2. X-ray Diffraction (XRD) Analysis:

Powder X-ray diffraction (XRD) measurements were carried out using Cu Kα radiation with a wavelength of approximately 1.5406 Å over a suitable 2θ range of 10°–80°. The scanning rate and step size should be specified according to the experimental conditions. The crystalline phases were identified by comparing the recorded diffraction patterns with standard crystallographic database references. For hexagonal boron nitride (h-BN), the characteristic diffraction reflections generally appear at approximately 26.7° (002), 41.6° (100), 43.9° (101), 50.2° (102), and 55.2° (004). Minor variations in peak positions, intensities, and widths may occur depending on the degree of crystallinity, lattice strain, and stacking defects. The observed diffraction peaks are consistent with the characteristic reflections reported for h-BN in the literature.

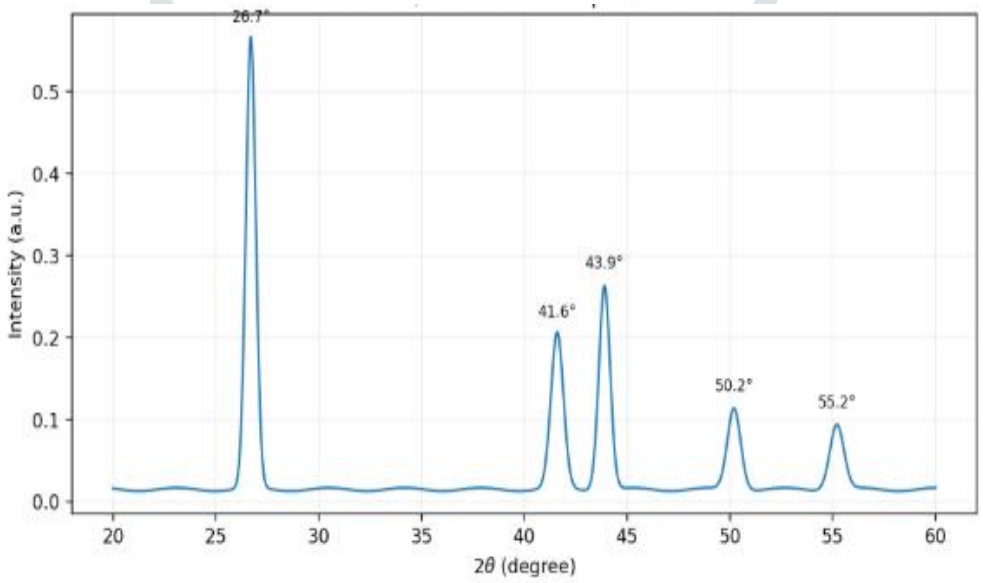

Figure (2). Shows XRD pattern of hexagonal boron nitride (h-BN).

The XRD pattern obtained for boron nitride nanoparticles exhibits diffraction peaks corresponding to the characteristic crystalline structure of boron nitride. The peak positions remain nearly unchanged, indicating that the fundamental crystal structure has been preserved during nanoparticle preparation. However, differences in diffraction intensity are observed between the bulk boron nitride and the nanoparticle samples. Such variations may be associated with the presence of surfactants used during nanoparticle formulation, which can adsorb onto the particle surfaces and influence the measured diffraction intensities. Overall, the diffraction results indicate that the preparation process has not resulted in the formation of any detectable undesirable crystalline phase, and the characteristic diffraction features of boron nitride are retained in the formulated nanoparticles.

3.3. Fourier Transform Infrared (FTIR) Analysis

The FTIR spectra recorded for bulk boron nitride and boron nitride nanoparticles are presented in Figure (3). The characteristic absorption bands observed in pure boron nitride at approximately 715 cm^{-1} and 1413 cm^{-1} are also present in the FTIR spectrum of the synthesized nanoparticles. These absorption bands correspond

to the characteristic vibrational modes of boron nitride. The absorption band appearing near 1413 cm^{-1} is attributed to the B–N stretching vibration, whereas the band observed around 715 cm^{-1} is associated with the B–N–B vibrational mode. The presence of these characteristic bands in both samples indicates that the fundamental bonding characteristics of boron nitride remain essentially unchanged after nanoparticle formation.

The FTIR spectra of bulk boron nitride and its nanoparticles exhibit comparable vibrational features. These similarities suggest that the nanoparticle preparation process has not introduced any significant undesirable chemical interactions affecting the basic boron nitride structure.

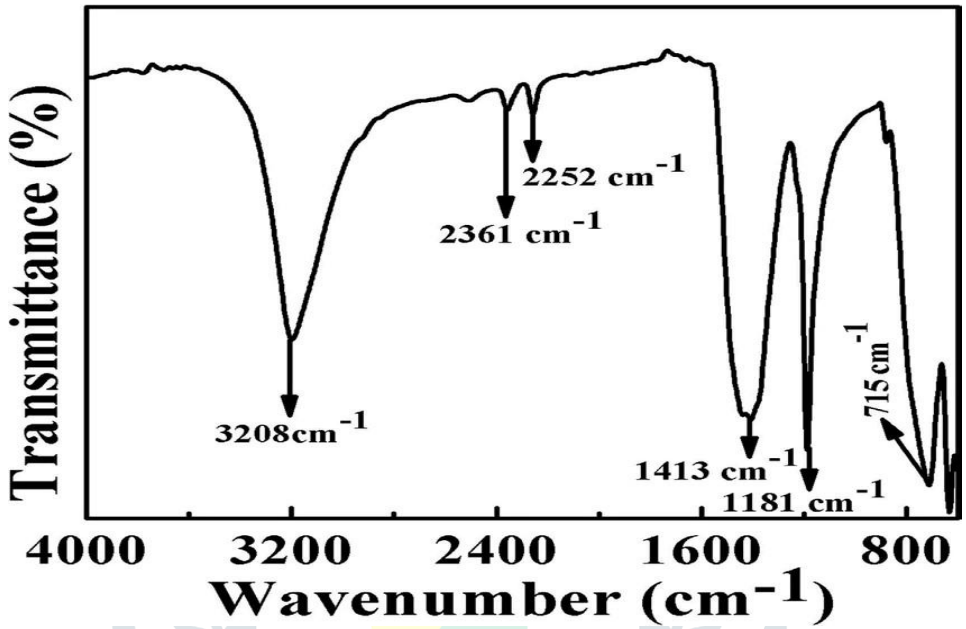

Figure (3). FTIR spectra of bulk boron nitride and nanoparticles.

Furthermore, the absorption features observed in the higher-wavenumber region, approximately 4000 –3500 cm^{-1} in the boron nitride sample, may undergo a shift towards the 3500 –3000 cm^{-1} region in the nanoparticle formulation. This variation may be attributed to the influence of surfactants or differences in the preparation conditions. A reduction in absorption intensity may also result from the adsorption of surfactant molecules onto the nanoparticle surfaces, which can modify the corresponding infrared absorption characteristics.

3.4. Differential Thermal Analysis (DTA)

Differential Thermal Analysis (DTA) was employed to investigate the thermal behaviour of the synthesized boron nitride powder. For the measurement, a precisely weighed quantity of the sample was placed in a suitable crucible, along with an appropriate reference material. The analysis was performed under a controlled nitrogen atmosphere. The experimental parameters, including sample mass, heating rate, operating temperature range, nitrogen gas flow rate, and instrument calibration conditions, should be clearly specified. DTA is based on measuring the temperature difference between the sample and reference material during a controlled heating or cooling program. The resulting thermal curve provides information about endothermic and exothermic transformations occurring within the sample.

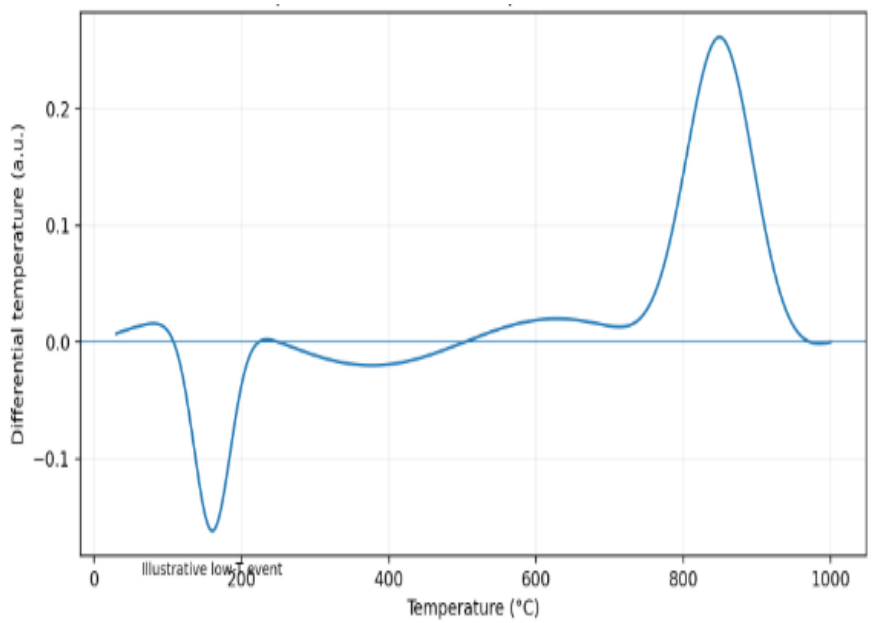

Figure (4). Shows DTA curve illustrating the thermal behaviour of boron nitride.

The thermal events observed in the DTA curve should be interpreted by considering the experimental atmosphere and, wherever possible, supported by complementary Thermogravimetric Analysis or Differential Scanning Calorimetry measurements. It is important to note that boron nitride may undergo oxidation at elevated temperatures when exposed to air, potentially resulting in boron oxide-containing residues. Therefore, thermal behaviour recorded under an oxidizing atmosphere may differ considerably from that observed under an inert nitrogen atmosphere.

4. CONCLUSION:

In the present investigation, an approach for synthesizing hexagonal boron nitride (h-BN) on copper foil and its potential application as a gate dielectric layer in graphene-based electronic devices has been discussed. The synthesis approach involving boric acid as a precursor, followed by thermal treatment under a nitrogen atmosphere, provides a practical method for preparing hexagonal boron nitride under laboratory conditions. The formation of the h-BN phase can be established through X-ray diffraction analysis, while Scanning electron microscopy (SEM) provides valuable information regarding particle morphology and surface characteristics.

The prepared h-BN films exhibit a polycrystalline nature, with an estimated crystallite size of approximately 20 nm. FTIR analysis confirms the presence of characteristic B–N vibrational bands, indicating the retention of the fundamental bonding structure of boron nitride after nanoparticle preparation. Furthermore, thermal investigations using DTA provide useful information regarding the thermal response of the synthesized material under a controlled atmosphere.

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