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Research Letter

An Affordable and Simple Synthesis of Boron Nitride Nanosheets for Terahertz Radiation Shielding

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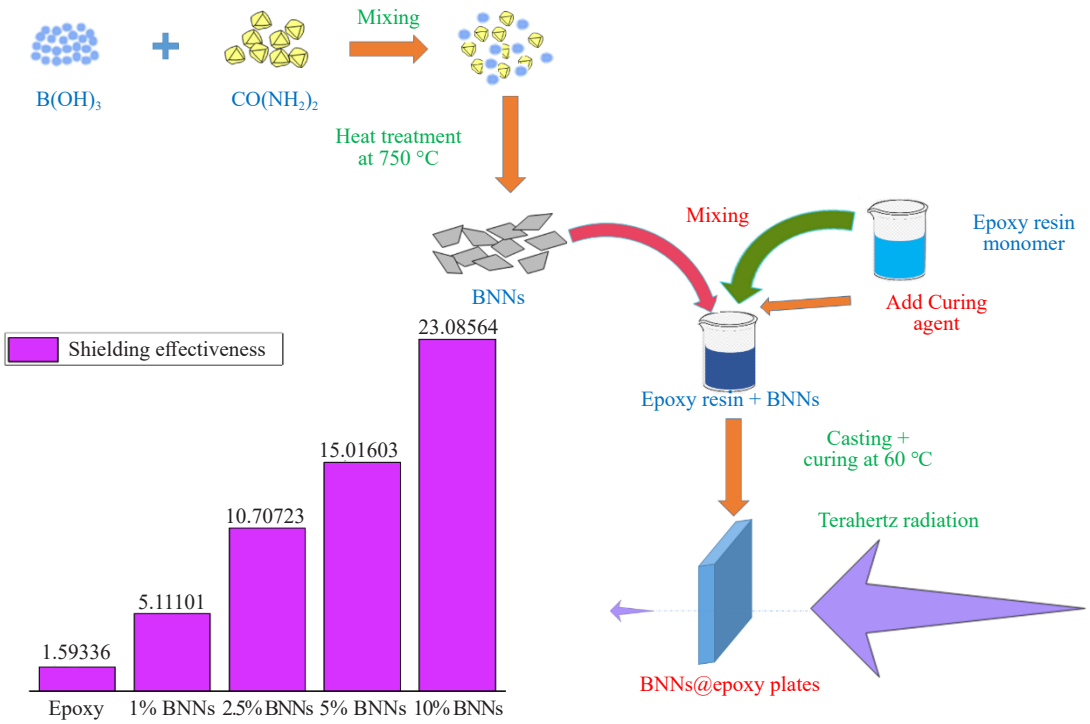
Graphical Abstract:



Abstract: Two-dimensional hexagonal boron nitride (h-BN) is an outstanding material characterized by its low dielectric constant, making it an excellent candidate for absorbing radiation in the high-frequency range, including the terahertz band. This study is dedicated to the preparation of BN nanosheets (BNNs) using a facile and straightforward

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bottom-up pyrolysis method with inexpensive urea and boric acid. The morphology, crystalline structure, and elemental composition of the as-prepared BNNs were analyzed using powder X-ray diffraction (XRD), Raman spectroscopy, energy-dispersive X-ray spectroscopy (EDS), and scanning electron microscopy (SEM). The results confirmed the successful synthesis of BNNs. The terahertz radiation shielding (TRS) effectiveness of BNNs was evaluated by incorporating them into epoxy resin matrices with varying fractions. The results demonstrate that the 3 mm thick BNN@epoxy resin plate loaded with 10% BNNs exhibits an extraordinary TRS effectiveness of 23.1 dB, making it a highly effective shield against terahertz radiation.

***Keywords*:** boron nitride nanosheets, electron microscopy, BNNs@epoxy, terahertz radiation shielding

1. Introduction

Electromagnetic radiations (ERs), including the terahertz band, may create malfunctions in electronic defensive systems, communication, military, and medical, but more importantly, they may damage biological organisms and the environment.[1,2] In order to avert this impending danger, researchers seek to design effective electromagnetic radiation shielding materials (ERSMs), which have the capacity to counteract or reduce ERs by absorption or reflection mechanisms. Among ERSMs, 2-D-materials *i.e*. graphene and derivatives, MXens, transition metal, dichalcogenides, and black phosphorus have received attention as effective ERSMs because of their alluring mechanical, chemical, electrical, and optoelectronic properties.[3,4]

Due to their low electrical resistivity, high thermal conductivity, extensive surface area, lightweight nature, and tunable properties,[5,6] graphene and its derivatives have emerged as the most extensively studied 2D ERSMs for low-frequency applications. Nevertheless, graphene and its derivatives are weak absorbers and strong reflectors of ERs, resulting in serious secondary pollution; therefore, their use in high-frequency radiation shielding is not preferred.[7,8]

On the other hand, the rapidly expanding uses for terahertz radiations have created an urgent need for ERSMs with superior absorption capabilities to limit the interference of terahertz radiations, enhance their transmission environment, and maintain the proper operation of sensitive electronic devices.[9] Furthermore, materials that can conceal terahertz waves are extremely important for national security and safeguarding information. These materials can help prevent significant losses for individuals, businesses, and even entire countries.[10]

Regarding literature, several materials may fulfill the requisite absorption performance, however, the majority of them suffer from excessive density and poor chemical stability. In terms of absorption/stealth capacity, hexagonal boron nitride (h-BN) may satisfy the requisite criteria, notably exhibiting a large electrical band gap (5-6 eV) and a low dielectric constant (3-4).[11,12] Additionally, h-BN boasts the highest thermal conductivity among all electrical insulators (280 $W \cdot m^{-1} \cdot K^{-1}$), positioning it as an exceptional material for thermal management.[13] Its strong absorption capability further minimizes the risk of secondary radiation pollution, a common limitation of graphene-based materials.[14]

Structurally, h-BN is an isostructural analog of graphite, composed of layers that are bonded by van der Waals forces. The distance between each layer is 0.33 nanometers, so it can effortlessly be exfoliated into 2D sheets by several existent techniques.[15,16] The bottom-up synthesis method offers notable advantages over traditional exfoliation techniques for producing boron nitride nanosheets (BNNs). Liquid-phase exfoliation methods often yield only 1% to 10%, while requiring significant energy input, prolonged processing times, and reliance on organic solvents, which increase costs and environmental concerns. Furthermore, BNNs produced through techniques like mechanical ball milling,[17] electrochemical exfoliation,[18] and supercritical CO_2 exfoliation[19] are prone to high defect levels, compromising their structural integrity and degrading their inherent properties. In contrast, the bottom-up approach, which employs cost-effective precursors like boric acid and urea, achieves higher efficiency with a reduced environmental footprint.[20,21] Moreover, BNNs synthesized through this method demonstrate superior structural uniformity and minimal defects compared to their exfoliated counterparts, resulting in enhanced mechanical strength and thermal stability.[22]

In this study, BNNs were synthesized using a straightforward and cost-effective bottom-up pyrolysis method from inexpensive precursors, namely boric acid and urea. The quality and morphology of the synthesized BNNs were characterized using advanced analytical techniques. To assess TRS performance, the BNNs were incorporated into epoxy resin matrices at various loadings and compared with commercial boron nitride microparticles (BNMs). The

results revealed that the as-prepared BNNs exhibited superior shielding effectiveness, achieving 23.1 dB attenuation with a plate thickness of just 3 mm. This demonstrates the remarkable potential of the as-obtained BNNs as an efficient terahertz radiation shield.

2. Materials and methods

2.1 *Materials*

Boric acid and urea have been acquired from SIGALD. ALADDIN was the supplier of Boron nitride microparticles (BNMs) (China). From Wuhan Biet Co., Ltd., epoxy resin and the curing agent N-(3-aminopropyl) cyclohexylamine were acquired.

2.2 *Synthesis of BNN*

BNNs were produced using inexpensive starting materials, namely boric acid and urea. A mixture of boric acid and urea was prepared with a molar ratio of 1:5 and then stirred in water for 2 hours using a magnetic stirrer. The mixture was then dehydrated at 70 °C. The as-obtained solid was heat-treated at 750 °C for 4 hours in an inert environment (N_2) (Figure 1).

2.3 *Preparation of BN@epoxy plates*

The BN@epoxy plates were produced by in-situ polymerization. The first step consisted of mixing 10 g of the monomer (epoxy resin) with the filler (BNNs or BNMs), with different mass proportions (1%, 2.5%, 5%, 6.5%, 10%), under a mechanical stirrer equipped with a propeller and in an ultrasonic bath for 8 minutes. Then, 5 g of the curing agent was added under the same circumstances and mixed for 5 minutes, followed by 60 minutes of baking at T = 60 °C (Figure 1). The thickness of BN@epoxy plates is 3 mm.

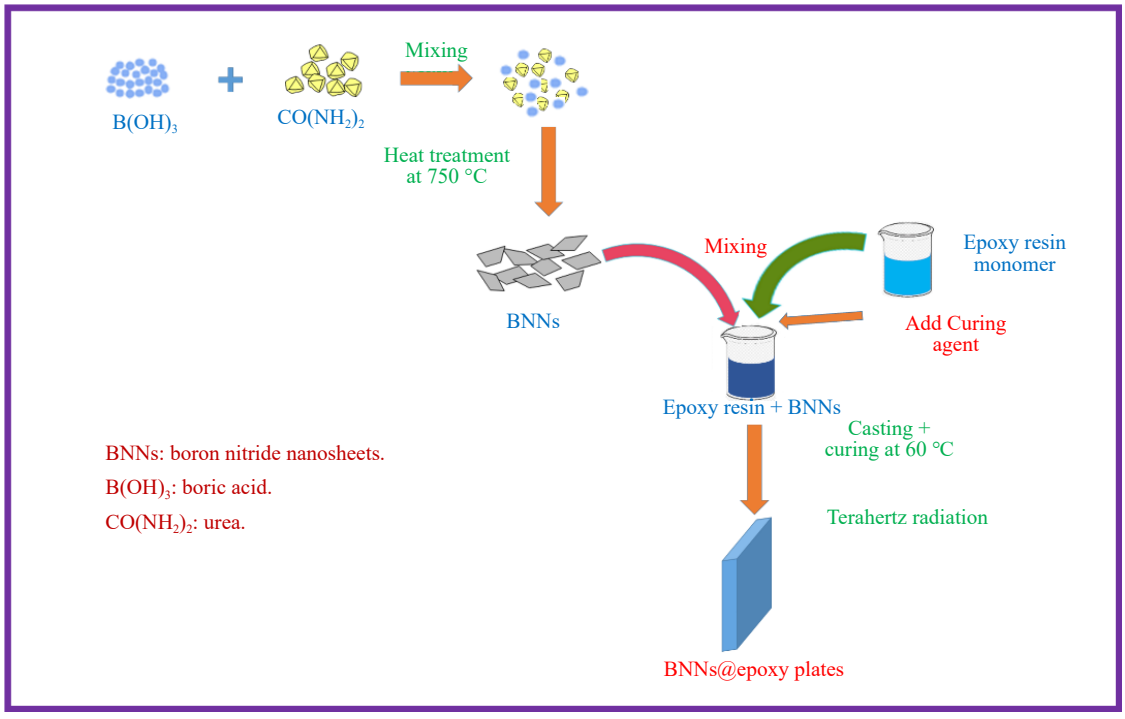

Figure 1. Schematic illustration of the experimental procedure

3. Results and discussion

3.1 *Morphological and structural characterizations of the as-obtained BNNs*

The SEM analysis of the as-obtained BNNs reveals their distinct morphology. At the micrometer scale (Figure 2a), the BNNs exhibit a layered, flower-like structure, characterized by uniform and well-defined agglomerates.

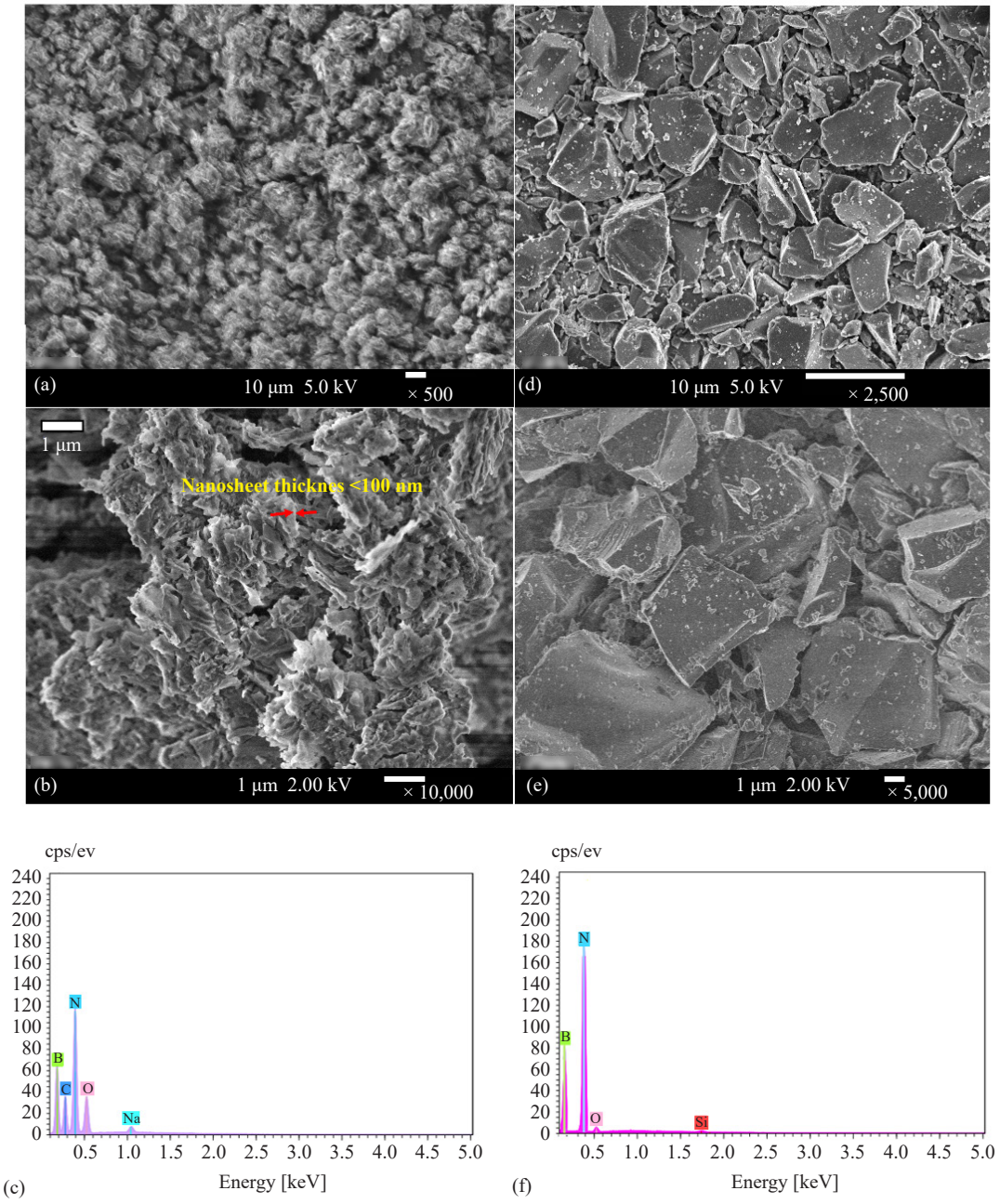

Figure 2. SEM images and EDS analysis of boron nitride nanosheets (BNNs): (a) low-magnification SEM image of the as-synthesized BNNs, (b) high-magnification SEM image of the as-synthesized BNNs, (c) EDS analysis of the as-synthesized BNNs; (d) low-magnification SEM image of commercial BNMs, (e) high-magnification SEM image of commercial BNMs, and (f) EDS analysis of commercial BNMs

At higher magnifications (Figure 2b), the flower-like formations are shown to consist of aligned and flexible

nanosheets with random rotations and translations around the normal layer. These nanosheets exhibit an ultrathin thickness of less than 100 nm. This morphology confirms the successful synthesis of boron nitride nanosheets, showcasing their characteristic 2D structure and uniformity. This remarkable tendency may be attributable to the coupling of a huge flat surface with an ultrathin thickness, as is the case with graphene. In agreement with the material's stoichiometry, the matching EDS spectrum (Figure 2c) reveals the presence of boron and nitrogen in a proportion of B: N = 1:0.9.[23,24] On the other hand, Figures 2d and 2e present SEM images illustrating the morphology of the commercial BNMs. The purchased BNMs display a morphology characterized by microsized particles, each measuring a few micrometers in size. The EDS analysis further confirms the presence of both boron (B) and nitrogen (N) within the structure, validating its composition.

XRD analysis was performed on both the purchased BNMs and synthesized BNNs to examine their crystalline structures. The diffraction pattern of the as-obtained BNNs indicates two major peaks with diffraction angles (2θ) of 26.7° and 43.6°, corresponding to the (002) and (101) diffraction planes, respectively (Figure 3a). These peaks are characteristic of hexagonal boron nitride, with the larger peak (2θ = 26.7°) indicating that the BN sheets possess partial crystallinity, with layers that are stacked almost parallel to each other but with random rotations and translations around the normal layer.[25] Additionally, the synthesized BNNs exhibit structural differences compared to the purchased boron nitride materials (BNMs), as evidenced by the broader peaks. This broadening confirms the successful formation of nanometric sheets.[15,26]

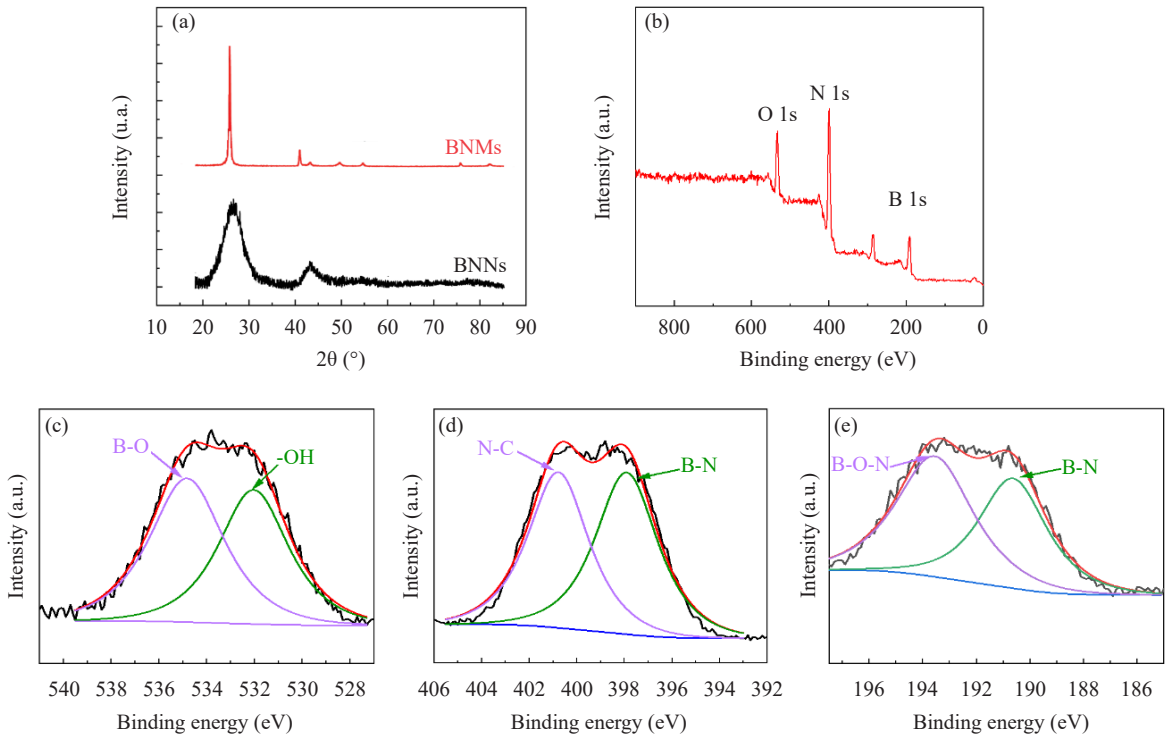

Figure 3. (a) XRD pattern of the purchased BnNMs and the as-obtained BNNs; (b) XPS survey of the as-obtained BNNs; (c) deconvolution of the XPS O 1s peak; (d) deconvolution of the XPS N 1s peak and (e) deconvolution of the XPS B 1s peak

XPS was also employed to evaluate the surface chemical composition and nature of the component bonds of the as-obtained BNNs. All spectra were normalized to aliphatic carbon at 285 eV binding energy. The resulting survey spectrum confirms the existence of the elements B, N, O, and C in the as-obtained BNNs (Figure 3b). The synthesis of boron nitride is shown by the presence of a localized peak at 190.7 eV, which corresponds to the distinctive B-N bond (Figure 3e) (BN). Observing a second reference signal at 193.7 eV for the B-O bond suggests that oxygen coupled

to boron atoms was added to the surface of BN. For the N 1s spectra (Figure 3d), two peaks at 397.9 and 399.0 eV correspond to the N-B and N-C bonds, respectively.[27] The existence of the N-C signal may be ascribed to urea residues employed as a nitrogen source during the production of BN. The first noticeable peak at 532.1 eV in the O 1s spectrum (Figure 3c) is due to -OH groups, whereas the second peak at 534.8 eV indicates the presence of O-B bonds. The latter may be attributable to oxygen deposited on the material's surface after exposure to ambient air and moisture.[28]

3.2 *Terahertz radiation shielding assessment for BNNs@epoxy composites plates*

Figure 4a portrays the as-obtained BNNs@epoxy composite plates with different BNNs fractions. Prior to doping with BNNs, the epoxy resin plate appears yellow and transparent. However, after doping, the color changes to white, and the transparency of the plates decreases as the BNNs concentration increases. It can also be inferred that the BNNs are evenly dispersed throughout the epoxy resin, leading to a homogeneous composite. Figure 4b illustrates the experimental setup utilized to assess the terahertz radiation shielding effectiveness of BNN@epoxy composite plates. The experimental setup consists of a Terasense source emitting a continuous wave at a frequency of 100 GHz with an output power of 80 mW, photoconductive antennas, and a THZ-B detector (GENTEC-EO). These components are seamlessly coordinated by LabVIEW software (GENTEC-EO) for efficient data acquisition and processing from the source. Notably, the emitted radiation passes through an attenuator made of BNNs@epoxy composite plates, meticulously designed to meet the specific requirements of the experiment.

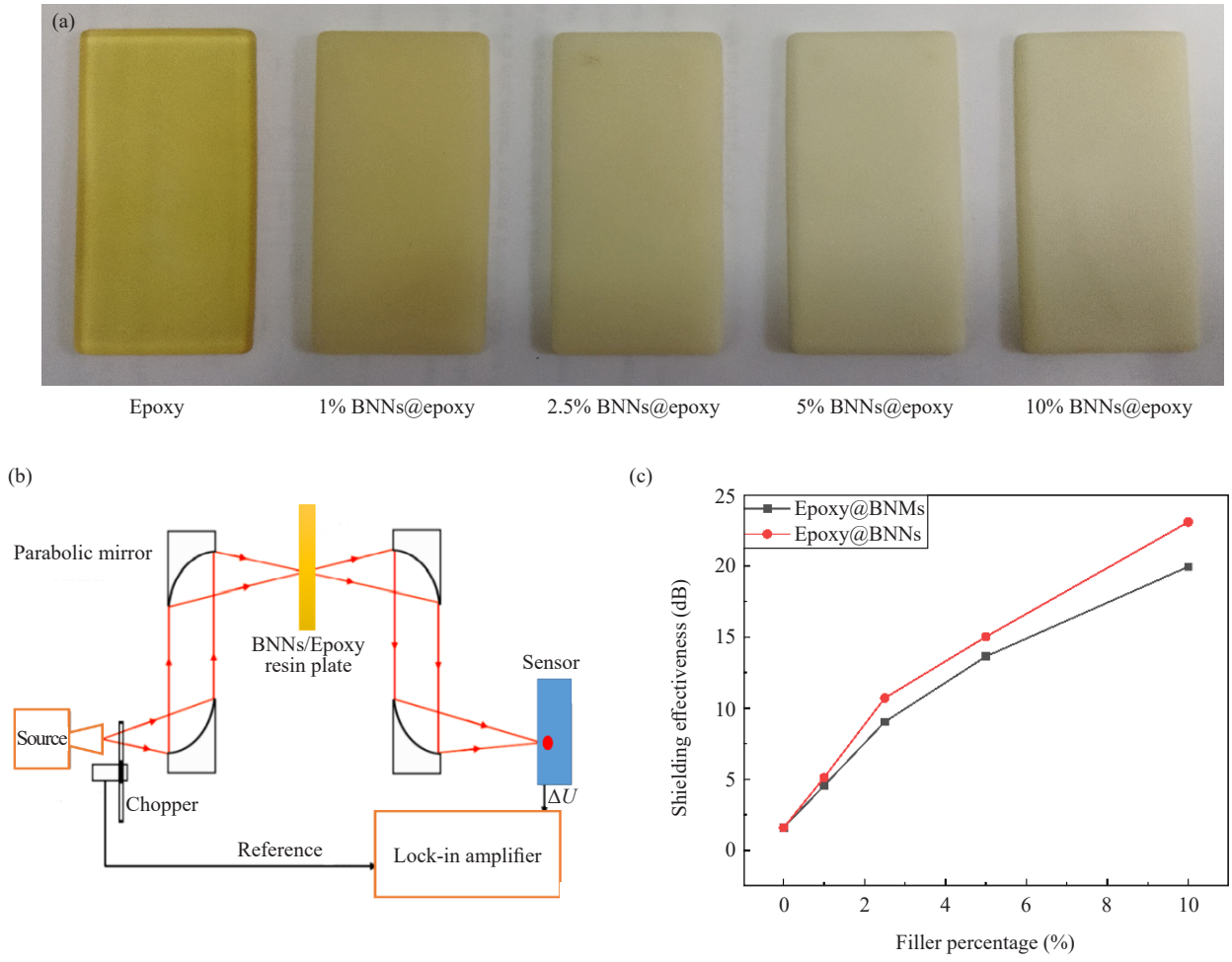

Figure 4. (a) Photographs of the as-obtained BNNs@epoxy resin composite plates with varying BNNs concentrations. (b) Schematic illustration of the experimental instrumentation of the shielding test. (c) Assessed shielding effectiveness values of BNNs@epoxy resin and BNMs@epoxy resin composites with varying filler percentages

As illustrated in Figure 4c, the as-synthesized BNNs demonstrate exceptional performance in attenuating terahertz radiation, significantly surpassing the purchased BNMs. At 0.1 THz, the TRS effectiveness of BNNs@epoxy and BNMs@epoxy increases markedly with higher filler percentages, rising from 5.1 dB to 23.1 dB and from 4.5 dB to 19 dB, respectively, as the filler content increases from 1% to 10%. Notably, the BNNs@epoxy material achieves a shielding effectiveness of 23.1 dB, making it approximately 1.21 times more effective at attenuating transmitted electromagnetic waves than the BNMs@epoxy material, which exhibits a shielding effectiveness of 19 dB. BNNs show higher effectiveness in attenuating terahertz radiation than purchased BNMs due to their high surface area, nanoscale thickness, and superior dielectric properties. They disperse uniformly in matrices, creating efficient wave absorption and scattering pathways. Additionally, their strong interfacial interactions and intrinsic stability enhance energy dissipation.[29]

3.3 *Mechanisms of terahertz radiation attenuation in BNNs@epoxy*

The outstanding ability of the as-synthesized BNNs to attenuate terahertz radiation stems from their exceptional properties. Their inherent dielectric characteristics and wide bandgap enable effective interaction with terahertz radiation through reflection, absorption, and scattering mechanisms. The strong polar nature of boron-nitrogen bonds enhances dipole polarization under external terahertz radiation, effectively disrupting wave propagation.[30,31] Furthermore, defects and impurities, particularly carbon introduced during the synthesis stage, may contribute to additional loss mechanisms. These include the formation of localized electronic states and the creation of scattering centers.[32] In addition, their high thermal conductivity ensures efficient dissipation of absorbed energy, preventing overheating during shielding applications. Lastly, the layered structure of BNNs contributes to radiation attenuation through combined absorption and scattering effects, making them a highly efficient material for terahertz shielding. Figure 5 illustrates the mechanism of terahertz attenuation using BNNs@epoxy composite plates. In this composite, the epoxy matrix plays a pivotal role by ensuring uniform dispersion of the BNNs, creating a continuous medium that enhances wave propagation and attenuation mechanisms. This uniform distribution maximizes the nanosheets' dielectric properties and phonon scattering potential. Moreover, the epoxy's intrinsic dielectric behavior further strengthens the composite's overall shielding effectiveness by disrupting wave propagation.[33,34]

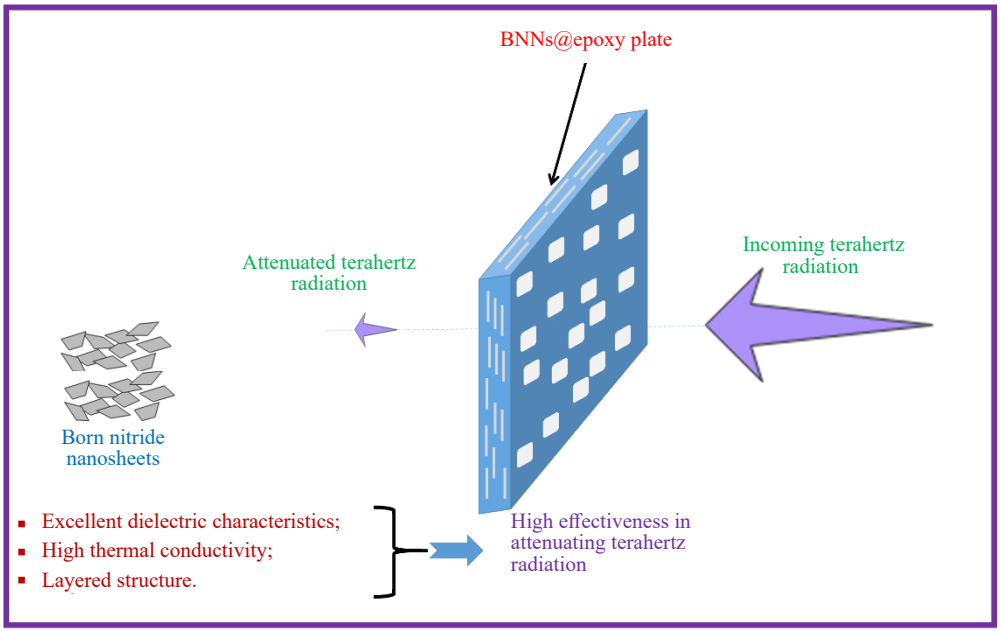

Figure 5. Schematic illustration of mechanism of terahearts radiation attenuation by using BNNs@epoxy plates

4. Conclusion

In conclusion, this study presents a cost-effective and straightforward bottom-up pyrolysis method for synthesizing boron nitride nanosheets (BNNs) using inexpensive urea and boric acid. The structural and morphological characterizations confirm the successful synthesis of BNNs with aligned and flexible nanosheets with random rotations and translations. The incorporation of BNNs into epoxy resin matrices at varying fractions results in a significant increase in the terahertz radiation shielding effectiveness, with a 3 mm thick BNN@epoxy resin plate loaded with 10% BNNs exhibiting an extraordinary TRS effectiveness of 23.1 dB. A comparison of the terahertz radiation attenuation performance between the synthesized BNNs and commercial BNMs highlights the superior effectiveness of the obtained BNNs. These results underscore the significant potential of BNNs for applications in terahertz radiation shielding. Furthermore, this work contributes to the existing literature by presenting a more efficient and scalable synthesis method for BNNs, providing an alternative to traditional, more complex approaches.

Conflict of interest

The authors declare that there is no conflict of interest.

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