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Exploration of Boron and Boron-Based Materials

Edited by Metin Aydin



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Aims and Scope of the Series

Materials science has always occupied an extremely high position in the human development process. As we explore the oceans of stars, various industries have put forward more stringent requirements for the performance of materials, forcing us to pay more and more attention to the development of new materials. At the same time, the formation of a data-driven scientific paradigm is dramatically shortening the development cycle of new materials. The huge data generated by synergistically combining theories, high-throughput experiments, high-throughput computation, and artificial intelligence is greatly contributing to our ability to utilize materials science to solve real-world problems. The three topics of this book series - Metals and Nonmetals; Composite Materials; and Surface Science - will address important areas of advancement in materials science. There will be a range of interesting works published under these topics.

Meet the Series Editor



Prof. Chonghe Li received his Ph.D. from the Chinese Academy of Sciences in 1995. From 1995 to 2000, he worked as a researcher at the Shanghai Institute of Metallurgy, Chinese Academy of Sciences, where he also served as director of the research laboratory. In 2000, he was appointed Professor at the Institute of High-Performance Computing in Singapore, where he worked on computation and simulation in materials science until 2004. Since then, he has been a professor at the School of Materials Science and Engineering, Shanghai University, China, as well as the director of the Shanghai Specialty Casting Engineering and Technology Research Center. Prof. Li's research focuses on titanium alloys, titanium-aluminum single crystals, intermetallic compounds, theoretical calculations, alloy design, and special refractory materials. His broad scientific expertise is well recognized by the scientific community around the world. He is a member of the editorial board of the journal *Metals*. As an author, he has published more than 200 peer-reviewed papers, 2 books, and over 40 patents.

Meet the Volume Editor



Dr. Aydin earned his BS (1989) and MS (1993) degrees in Engineering Physics from Ankara University. He subsequently completed his Ph.D. (2001) in Physical Chemistry at the City University of New York (CUNY). Following the award of his doctorate, Dr. Aydin was appointed Postdoctoral Associate at the Center for Analysis of Structures and Interfaces in the Department of Chemistry at The City College of CUNY, a position he held from 2001 to 2005. In 2008, he joined the Department of Chemistry at Ondokuz Mayıs University. Dr. Aydin's research has been published in numerous peer-reviewed journals, and he has delivered oral presentations at a wide range of international scientific conferences.

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Preface

In recent years, boron and its related compounds have garnered increasing attention due to their remarkable chemical versatility, electronic properties, and mechanical resilience. From nanoscale phenomena to macroscale engineering systems, boron-based materials enable significant advancements across a broad spectrum of scientific and technological domains. This book, *Exploration of Boron and Boron-Based Materials*, brings together selected studies that reflect the diverse roles and emerging significance of boron-containing systems. Intended for researchers, engineers, and professionals working in materials science, nanotechnology, energy systems, and environmental applications, this volume offers an integrated perspective on current developments and future directions.

The content has been structured into two parts to facilitate focused exploration, each capturing a distinct thematic orientation. Section I: “Functional and Quantum Applications of Boron-Based Nanomaterials”, centers on fundamental research into the physical properties and nanoscale functionalities of boron-based materials. The opening chapter introduces a novel methodology for manipulating light emission from atomic defects in hexagonal boron nitride (h-BN) via surface acoustic waves. This dynamic modulation technique reveals the promise of h-BN as a platform for room-temperature single-photon sources, with implications for quantum information processing and photonic communication systems.

Following this, the book explores the unique superconducting properties of magnesium diboride (MgB_2), a material recognized for its relatively high critical temperature and cost-effective production methods. The discussion highlights progress in wire synthesis and scalability, as well as its prospects in magnetic field generation and sensor applications. The final chapter in Section I examines the synthesis and structural analysis of boron nitride nanomaterials for gas sensing, illustrating how controlled crystallinity and nanostructuring can improve sensitivity and performance in environmental detection.

Section II: “Industrial Performance and Environmental Sustainability of Boron-Based Materials” shifts focus to engineering applications and environmentally responsive technologies involving boron-based materials. The initial chapter in this section assesses the performance of cubic boron nitride (CBN) tools in machining operations, particularly for materials known to be difficult to process. It examines how variables such as tool composition and operational parameters affect cutting efficiency, wear resistance, and surface integrity, providing insights into optimizing the use of CBN tools across various industrial scenarios.

Subsequent chapters explore the development and application of boron-doped diamond (BDD) electrodes in electrochemical systems. One chapter presents a sequential approach to electrodepositing platinum and nickel onto BDD substrates for ammonia oxidation reactions, a process relevant to wastewater treatment

technologies. The synergy between the catalyst composition and the boron-doped substrate yields improved electrocatalytic performance, signalling the potential for enhanced environmental remediation strategies.

The closing chapter offers a broader perspective on the role of BDD electrodes in sustainable water treatment, highlighting their chemical stability, wide electrochemical window, and environmental compatibility. These materials demonstrate how advanced functional surfaces can be utilized to address global challenges in water purification while supporting long-term ecological objectives.

This collection captures the multifaceted nature of boron-based materials, from their role in frontier physics and chemistry to their real-world deployment in industrial and environmental contexts. We hope this book will serve as both a reference and a source of inspiration for ongoing and future research, encouraging collaborative innovation in the pursuit of high-performance, sustainable materials solutions.

We are grateful to the authors and researchers who contributed to this volume, as well as to the broader scientific community, for their ongoing contributions to this exciting and evolving field.

Finally, I would like to express my sincere gratitude to my beloved wife, Oya Aydın, for her unflinching understanding and patience, as well as to the IntechOpen team for their constant support throughout the planning and compilation of this book.

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Section 1

Functional and Quantum
Applications of Boron-Based
Nanomaterials

Dynamic Sound Tuning of Quantum Light Emission from Atomic Defects in Hexagonal Boron Nitride

Sanja Đurđić Mijin and Snežana Lazić

Abstract

A key advancement and the main focus of this chapter is the dynamic *in situ* control of quantum emitters using traveling sound waves generated by acoustic phonons in hexagonal boron nitride. As they travel through the material, these phonons interact with radiative defect states through deformation potential coupling, leading to tunable optical, electronic, and mechanical properties, thereby facilitating control over the spectral position and timing of single-photon emission. The spectral tuning is achieved within a bandwidth of approximately 2.5 meV, which is then combined with spectral detection filtering to accomplish temporal control of the emitted quantum light. The impact of the acousto-mechanical effect in hexagonal boron nitride further demonstrates an exceptionally strong elasto-optic coupling in this material system, measured at approximately 50 meV/%, which facilitates the production of scalable strain-engineered quantum photonic devices. Altogether, by leveraging acoustic modulation techniques in hexagonal boron nitride semiconductor, this work establishes a foundation to enhance the functionality of quantum emitters that are relevant for applications in nanophotonic and quantum information technologies.

Keywords: single photons, surface acoustic waves, atomic defects, hexagonal boron nitride, elasto-optic coupling, nanophotonics

1. Introduction

The field of quantum science and related technologies is advancing rapidly, with profound implications for science, industry, and everyday life. Through continued research and development, we are approaching the realization of quantum devices and circuits that surpass the limitations of current digital systems. Among the various candidates for implementing quantum technologies, the quantum properties of light and light-matter interactions have emerged as key areas of exploration. At the forefront of this research is the deterministic generation and control of light at the single-photon level—a fundamental requirement for quantum applications. Single-photon sources play a crucial role in scalable quantum technologies, including quantum

computing, cryptography, communication, and sensing [1, 2]. Photons are particularly suitable for quantum information processing because they serve as robust, decoherence-resistant qubits that can propagate through fiber optics or free space with minimal interaction. Furthermore, they offer multiple degrees of freedom that can be used as qubits for quantum information encoding, such as polarization, time-binning, orbital angular momentum, frequency, phase, etc. [3]. Another important advantage of photon-based quantum technologies is the use of remarkably stable (two-level) condensed matter systems for deterministic production of quantum light states.

Despite significant progress, achieving stable and scalable single-photon sources remains a major challenge. Efficient generation and control of single photons are essential for the widespread application of quantum photonics. Among various available non-classical light sources, defect states in insulating hexagonal boron nitride (h-BN) semiconductor have emerged as a promising platform for quantum photonics.

In this layered material with a honeycomb lattice structure and a wide bandgap (~ 6 eV [4]), the emission of antibunched light arises from radiative atomic defects, which introduce localized states deep within the bandgap, enabling single-photon emission [5–15]. The quantum light emission from h-BN defects excels in high single-photon purity, robustness against external perturbations, and stable operation under elevated temperatures and pressures. Such solid-state quantum light devices operating in excess of room temperature are vital for large-scale on-chip integration of single-photon emitters (SPEs) due to the inherent system heating.

To enable integrated quantum photonic chips, it is essential to develop quantum emitters that can be easily fabricated, replicated, and deterministically positioned at the sub- μm scale. This is particularly relevant given that a crucial challenge in the development of large-scale quantum photonic circuits is the scalability of single-photon emitters. Due to its planar structure and weak interlayer interactions, the h-BN material can be easily exfoliated and integrated into diverse platforms, enabling compatibility with existing technologies, thereby facilitating the development of compact and scalable advanced on-chip nanophotonic and optoelectronic devices.

Consistency in the optical properties of these emitters is also crucial, as uniformity across an array enhances the efficiency and functionality of chip-scale quantum photonic systems. Traditional bulk and three-dimensional single-photon sources face significant limitations in terms of photon extraction efficiency and seamless integration into photonic circuits. In contrast, quantum emitters based on layered materials, such as h-BN, offer an exciting alternative.

h-BN hosts a variety of atomic-scale defects that demonstrate stable and bright single-photon emission. While the precise nature of these defects remains under investigation, they have emerged as promising candidates for room-temperature quantum emitters. Nevertheless, a major obstacle in leveraging h-BN for quantum technologies lies in the spectral variability between emitters, which gives rise to a wide dispersion of emission wavelengths. Several theoretical and experimental analyses reported in Refs. [8, 9, 11] suggest that this broad range of emission wavelengths may stem from inhomogeneous broadening associated with multiple defect types, or more likely, from defects of an identical atomistic configuration subjected to fluctuating local lattice strain and/or varying dielectric environment. Regardless of its specific origin, such significant spectral inhomogeneity poses a challenge to the realization of identical quantum light sources, which are essential for scalable photonic systems that require precisely defined emission energy. One approach to overcoming this issue involves post-fabrication tuning quantum emitters, allowing for *in situ* modulation of

their emission properties. Typically, manipulation of light emitted from h-BN defect states has only been experimentally demonstrated by tuning the photon emission energy using either static elastic strain [8, 9] or an electric field [16]. However, a limitation of these approaches is their inability to *achieve dynamic in situ* control of radiative h-BN defect states. In contrast, the present work demonstrates that utilizing acousto-mechanical and acousto-electric effects driven by radio-frequency (RF) surface acoustic waves (SAWs) offers the advantage of tracking the temporal evolution of defect emission down to a (sub-) nanosecond timescale.

As previously observed in other material systems, the oscillating strain and piezoelectric fields generated by propagating acoustic waves can effectively interact with defect centers [17–19] or nanostructures such as semiconductor quantum dots (QDs) [20–25] positioned near the surface of the wave-supporting medium. In this study, we demonstrate real-time manipulation of the optical emission from atom-like defects in h-BN *via* SAWs. We find that the acousto-mechanical coupling induced by the SAW causes a periodic modulation of the emission energy from individual, linearly polarized single-photon emitters. The extent of this modulation is proportional to the mechanical coupling strength between the emitting defect and the acoustic substrate. Based on the optical response of these emitters to the moving mechanical vibrations, we estimate the deformation potential to be approximately 5 eV, with a strain-induced tuning coefficient near 50 meV/% for h-BN. These values surpass previous experimental results obtained under static external strain by an order of magnitude [8], highlighting the strong elasto-optic coupling in h-BN. Additionally, our method combines strain-mediated, real-time control of the defect emission energy with a spectral filtering technique for temporal modulation of the emitted photons. Overall, our findings illustrate that SAWs serve as a highly effective, contactless, and nondestructive external tool for dynamic, real-time control of non-classical light sources — enabling precise spectral tuning and time-gated emission of single photons. Such capabilities are promising for integrating h-BN defect centers into advanced quantum photonic devices.

2. Experimental section

2.1 Sample fabrication

To manipulate the light emitted from our h-BN defects, we employed surface acoustic waves (SAWs). These waves are elastic vibrations that travel along the surface of a material, with the majority of their energy confined to a thin region near the surface, typically with a depth approximately equal to one acoustic wavelength. In piezoelectric materials, the propagating mechanical wave is accompanied by an oscillating piezoelectric potential. Both the strain and piezoelectric fields generated by the SAW provide a nondestructive method to dynamically modulate the mechanical, electronic, and optical properties not only of the underlying substrate material, but also of semiconductor structures deposited on its surface. Moreover, the tuning rate enabled by these fields can span a broad frequency range, from a few megahertz (MHz) up to several gigahertz (GHz).

Our SAW experiments were performed using an acoustic delay line, illustrated in **Figure 1a**, consisting of two interdigital transducers (IDTs), each containing 35 finger pairs, which were patterned by photolithography and completed through a lift-off metallization process involving a 10 nm Ti/30 nm Al/10 nm Ti layer stack on the

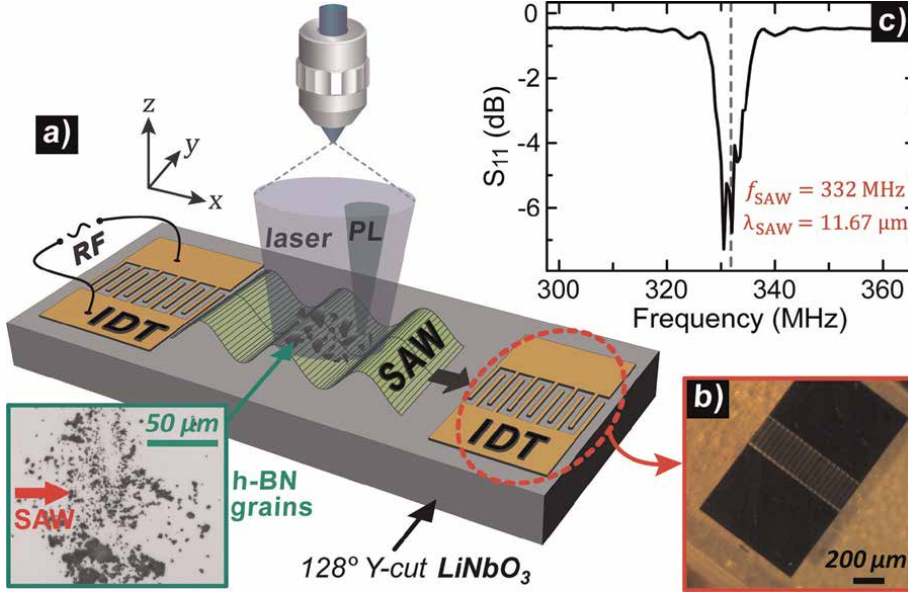


Figure 1.

(a) Acoustic delay line consisting of two interdigital transducers (IDTs) photo-lithographically patterned on the surface of a LiNbO_3 crystal. The inset shows an optical microscope image of the thermally expanded h-BN flakes mechanically transferred onto the area between the two IDTs. (b) Image of the IDT electrode captured using an optical microscope. (c) Dependence of the RF power reflection coefficient (S_{11}) on the frequency of the RF signal applied to one of the IDTs. The plot displays the region around the resonant frequency used in our experiments ($f_{\text{SAW}} = 332 \text{ MHz}$).

surface of a commercially available 128°Y -cut lithium niobate (LiNbO_3) crystal. The optical microscopy image showing the unidirectional IDT layout with floating electrodes is displayed in **Figure 1b**. The IDTs are designed to generate multiple harmonics of a fundamental wavelength $\lambda_{\text{SAW}} = 35 \mu\text{m}$. The experiments reported here were carried out using the third harmonic with $\lambda_{\text{SAW}} = 11.67 \mu\text{m}$, which corresponds to a resonance band centered at frequency $f_{\text{SAW}} = 332 \text{ MHz}$ (cf. **Figure 1c** displaying the IDT RF reflection spectrum measured with a vector network analyzer at room temperature) and a temporal period of $T_{\text{SAW}} = 2.96 \text{ ns}$ at room temperature. Full characterization of the RF scattering parameters (see Ref. [20]) gives an estimate of the electric-to-acoustic power conversion efficiency of approximately 30% for both transducers, indicating that only 30% of the nominal RF power supplied to the IDT is converted into acoustic power. This conversion efficiency remains notably stable across the entire RF power range employed during the experiments.

To distinguish SAW-induced effects from those potentially caused by thermal heating at high RF powers, both the laser excitation and RF signal were chopped at the same frequency of 400 Hz [22]. To isolate and eliminate temperature-related effects, for each acoustic power, the PL spectrum recorded under out-of-phase conditions between the optical and RF excitations (representing only thermal effects) was subtracted from the spectrum obtained under in-phase conditions (representing both SAW and thermal effects). This approach is especially crucial for identifying SAW-induced spectral shifts that are smaller than the typical PL linewidths. No significant RF-induced thermal contribution to the position or intensity of the emission lines was observed.

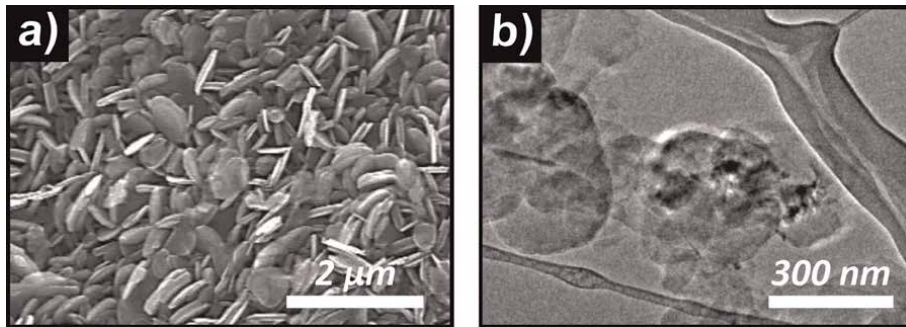


Figure 2.
(a) Scanning electron microscopy (SEM) and (b) transmission electron microscopy (TEM) images of thermally expanded h-BN flakes before their mechanical dispersion onto the SAW delay line shown in **Figure 1a**.

The use of a highly piezoelectric substrate enables the generation of strong strain and piezoelectric fields associated with the propagating surface acoustic wave. These SAWs are electrically generated by applying an RF voltage to one of the IDTs (cf. **Figure 1a**).

We subsequently utilized commercially available h-BN powder (Sigma Aldrich supplier) consisting of grains with an average diameter of approximately 1 μm and a purity of 98%. To generate quantum light emitters in the grains, this powder was thermally expanded at 1000°C for 10 minutes in an argon atmosphere using a tubular oven. This process yielded flakes composed of few-layer h-BN, with lateral dimensions of roughly 200–300 nm and thicknesses in the range of 3–5 nm, as estimated from the SEM and TEM images in **Figure 2a** and **b**, respectively. These flakes were then mechanically dispersed along the acoustic propagation path between the two IDTs. As seen in the optical microscopy image in the inset of **Figure 1a**, the flakes are randomly dispersed on the substrate, and we obtained regions with high and low areal flake density, the latter being favorable for optical spectroscopy on a single h-BN flake. We then analyzed the optical properties of these flakes under direct optical illumination by a focused laser beam, as described in the following subsection.

2.2 Optical characterization

Photoluminescence. The micro-photoluminescence (μ-PL) characterization was conducted within a customized experimental setup. The sample was mounted inside a CryoVac Helium-flow cryostat for the entire duration of the experiment and subjected to measurements at a temperature of 10 K. Utilizing a linearly polarized continuous-wave (cw) 442 nm laser as the excitation source, the laser beam was focused onto a 1.5 μm spot on the sample surface using a ×50 microscope objective lens with a numerical aperture of $NA = 0.73$. The emitted light was collected using the same objective lens, directed through a 442-nm ultra-steep long-pass edge filter (Semrock RazorEdge filter), and dispersed *via* a single-grating monochromator (with a spectral resolution of approximately 250 μeV, Jobin Yvon Horiba 750 M spectrometer). The dispersed light was detected with a nitrogen-cooled CCD camera Teledyne Vision Solutions (**Figure 3**).

For polarization-dependent measurements, an achromatic half-wave plate and a beam-displacement prism, providing two orthogonally polarized and spatially separated output beams, were inserted into the optical detection path. The use of a fixed

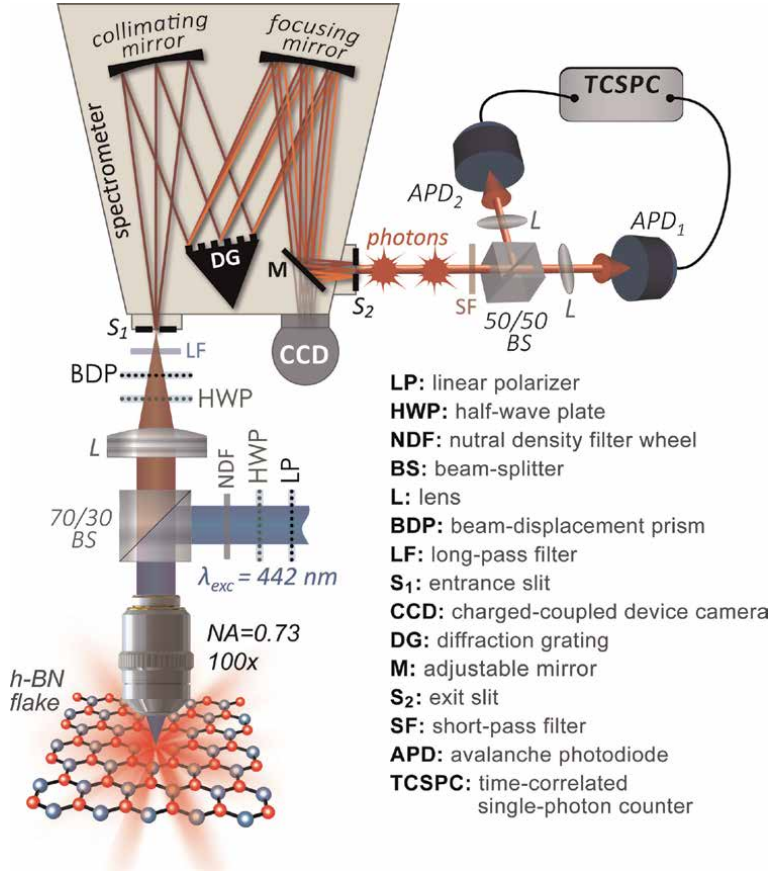


Figure 3. Experimental setup used for time-averaged, spatially, polarization- and time-resolved micro-photoluminescence as well as photon correlation spectroscopy measurements.

polarization analyzer in combination with a quarter-wave plate positioned in front of the monochromator's entrance slit effectively eliminates the influence of polarization anisotropy caused by the diffraction grating. The linear polarization of the laser excitation could also be rotated (using a half-wave plate inserted into the incident path) with respect to the direction of SAW propagation, which was aligned with both the entrance slit of the spectrometer and the extraordinary transmission axis of the beam-displacing prism. The projection of the emission dipole onto the sample plane and the degree of polarization were determined by rotating the half-wave plate in the detection path between 0 and 180° in steps of 5°, with 0° pointing out the SAW propagation direction (i.e., x-axis in **Figure 1a**).

Power-dependent measurements were carried out by adding a DC motor-controlled continuously variable reflective filter wheel to the incident optical path.

Photon correlation spectroscopy. These experiments were performed using a Hanbury-Brown and Twiss (HBT) interferometer placed at an additional monochromator exit port provided with a slit for spectral filtering of the PL emitted by the selected quantum light source, thus allowing the isolation of a single emission line. The HBT setup consists of two single-photon counting avalanche photo-diodes (APDs from Perkin Elmer), one in each path of a 50/50 non-polarizing beam splitter.

A 650 nm short-pass filter is inserted in front of one of the APDs to suppress the optical cross-talk between the two APD detectors. The signal from the detectors is sent to a time-correlated single-photon counting module (PicoHarp, Picoquant), where histograms of the correlation events as a function of the time intervals between photon detection at both detectors are obtained. The same setup with only one APD was used for time-resolved PL experiments carried out under pulsed laser excitation with a 442 nm wavelength, <100 ps pulse width, and 40 MHz repetition rate.

3. Results and discussion

As seen in **Figure 4a**, spectral analysis of thermally expanded h-BN flakes at low temperature ($T \approx 10$ K) reveals the presence of multiple narrow emission lines associated with atomic-scale defects. As previously reported in h-BN flakes [8, 9, 13], powder [14], and epitaxially grown films [26], these zero-phonon lines (ZPLs) covering a broad emission range (of ~ 900 meV) correspond to electronic states localized deep within the bandgap, which serve as single-photon emitters. In fact, the emission frequencies of SPEs found in h-BN can spread over a considerable spectral range from infrared to ultraviolet, as seen in Refs. [27, 28]. The spectral position of these peaks changes for different locations on the sample, suggesting that defects in h-BN can indeed couple to local lattice strain and/or dielectric environment, inducing large variations in their emission wavelength. Evidence for environment-dependent line

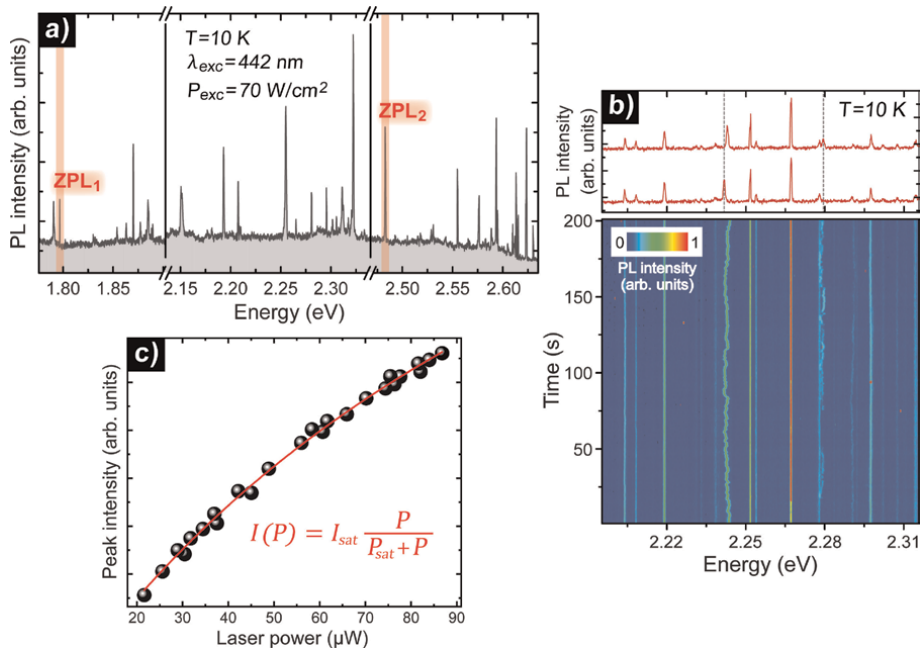


Figure 4. (a) A representative μ -PL spectrum recorded at 10 K under cw 442 nm laser excitation at power density of 70 W/cm², displaying sharp emission lines extending over a broad energy range. (b) Lower panel: Temporal-spectral map showing PL intensity spectra acquired sequentially on a set of ZPLs optically probed by the focused laser spot. Upper panel: Horizontal line profiles taken at two different times from the PL map in the lower panel. The integration time per trace is 1 sec. (c) Excitation power-dependent integrated intensities of the ZPL₂ in (a). The red line is a power-law fit given by Eq. (1).

shifts can be found in the literature [8, 9]. However, this wide span of the emission wavelengths in h-BN may also result from the presence of several defect species [11, 29]. Understanding these spectral variations is crucial for designing engineered quantum emitters with tailored properties. To better comprehend the origin of the wavelength occurrence of ZPLs, we have studied here the photophysics properties and stability of quantum light sources emitting at different energies.

The temporal stability of the PL signal is also a critical factor for the prospective use of emitting centers in h-BN. Monitoring the temporal evolution of the low-temperature optical response of representative defects by consecutively recording their PL spectra (cf. **Figure 4b**), we observed that most ZPLs demonstrate stable emissions—in terms of both intensity and emission energy—over the acquisition time with minimal line broadening, indicating high-quality quantum emitters. However, a small number of emitting centers exhibit spectral diffusion of their transition energy. Similar effects have been observed in other systems, such as semiconductor QDs, and are typically related to the local variation of the electric field induced by charge fluctuations in the vicinity of the emitting centers [30]. Such spectral jumps are also expected from emitters with a permanent dipole moment such as the anti-site nitrogen vacancy defect ($N_B V_N$, comprised of a missing nitrogen atom and a substitution of an adjacent boron atom by a nitrogen), which has been suggested as the potential atomic structure responsible for single-photon emission in h-BN SPEs (see, e.g., Refs. [8-10, 16, 29, 31, 32]).

Furthermore, the optical transition between the two electronic states that produce a ZPL observed near 2.28 eV in **Figure 4b** may exhibit additional emission corresponding to charge recombination between the excited and an additional electronic state that can be intrinsic to the defect (e.g., multilevel defect state inherent to some h-BN vacancy types [33]) or may originate from a nearby defect. We also measured the PL intensity of the ZPL₂ in **Figure 4a** as a function of the laser excitation power, and we found that it follows a saturated-emitter power dependence model described by:

$$I(P) = I_{sat} \frac{P}{P + P_{sat}}, \quad (1)$$

where $I(P)$ is the integrated peak intensity, P is the excitation power, and I_{sat} and P_{sat} are the saturation intensity and power, respectively. From the fit of Eq. (1) to the experimental data presented in **Figure 4d**, we extract $P_{sat} = 140 \mu W$.

Representative narrow ZPLs labeled ZPL₁ and ZPL₂ in **Figure 4a** are further characterized to gain a deeper understanding of the photophysical properties of the emitting quantum states. **Figure 5** displays polarization-resolved micro-PL measurements showing that these peaks are highly linearly polarized with the average polarization degrees equal to or above 70%. Values of the linear polarization degree were estimated by fitting the experimental data with Malu's law:

$$I(\theta) = a + b \cos^2(\theta - \theta_0), \quad (2)$$

where $I(\theta)$ is the signal intensity, θ_0 is the linear polarization direction of the studied ZPL line and a and $a + b$ are the minimum (I_{min}) and maximum (I_{max}) PL intensities. The degree of linear polarization is estimated from the fit using [34]:

$$DOLP = \frac{I_{max} - I_{min}}{I_{max} + I_{min}} = \frac{b}{2a + b}. \quad (3)$$

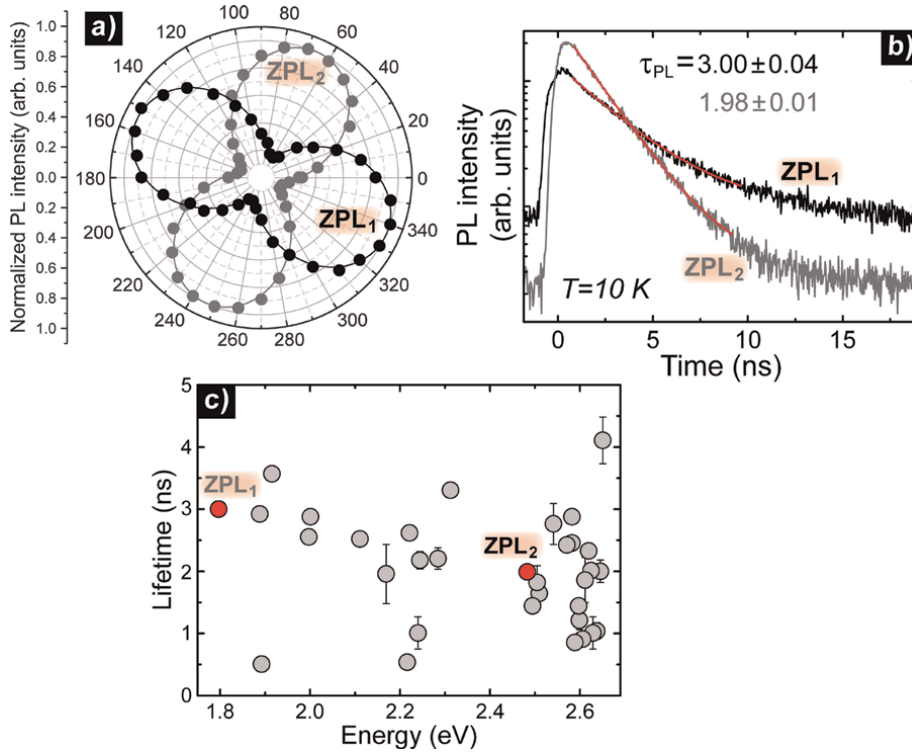


Figure 5. (a) Normalized and background-corrected low-temperature polarization-dependent intensities of peaks ZPL₁ (red circles) and ZPL₂ (blue circles) from **Figure 4a**, showing SPEs with a high linear polarization degree of 0.70 and 0.76, respectively. Solid lines are fits of the experimental data with Eq. (2). (b) Photoluminescence lifetime measurements exhibiting excited-state time constants (τ_{PL}) indicated in the figure, measured at low temperature for ZPLs appearing in three different spectral regions outlined in **Figure 4a**. Red traces are single exponential fits yielding τ_{PL} in the nanosecond time scale. (c) Lifetime data estimated for 34 different ZPLs.

As seen in **Figure 5**, ZPL₁ and ZPL₂ as well as other lines appearing in the emission spectra at different energies, have their own intrinsic direction of the polarization axes. This indicates that the linear polarization of the PL signal is most likely a result of the specific anisotropic structure of the particular emission center. Such high *DOLP* is crucial for various polarization-encoded quantum information applications, such as quantum key distribution and linear optical quantum computing using photonic qubits [35, 36].

We have also performed the time-resolved stroboscopic μ -PL experiments under pulsed optical excitation, as displayed in **Figure 5b**. The experimental data were fitted using a single-exponential decay, yielding a lifetime of ~ 2.1 ns for most of the investigated emitters, but the excited state lifetime can be as low as 0.5 ns and as high as 5 ns. The obtained lifetime spread outlined in **Figure 5c** is consistent with the characteristic decay times found in other monolayer and multilayer h-BN samples [9, 10, 12, 15, 37, 38].

To prove the presence of non-classical light emission from our h-BN emitters, we studied the statistics of emitted photons using a standard HBT interferometer delivering coincidence histograms that are proportional to the second-order correlation function:

$$g^{(2)}(\tau) = \frac{\langle I(t) \cdot I(t + I(t + \tau)) \rangle}{\langle I(t) \rangle^2}, \quad (4)$$

which describes the correlation between two temporally separated (by time difference τ) signal intensities from an individual light source. For a truly single-photon emitter we expect to observe no coincidences at zero time delay $\tau = 0$ between photons detected at the two HBT detectors. Indeed, the photon correlation histogram of a representative ZPL, depicted in the left-hand panels of **Figure 6a** and **b** recorded under cw laser excitation at $T = 10$ K (upper panels) and at room temperature (lower panels), respectively, shows a reduction of the coincidence rate at zero-time delay, therefore suggesting reduced probability of simultaneous multiple photon emission. To quantify the single-photon performance, we use a model for a three-level system, which is typically used to describe SPEs in h-BN [10, 39]:

$$g_{ideal}^{(2)}(\tau) = 1 - A \cdot e^{-\left|\frac{\tau}{\tau_1}\right|} + B \cdot e^{-\left|\frac{\tau}{\tau_2}\right|} \quad (5)$$

According to this model, in addition to antibunching of the excited quantum state on a short timescale (tens of nanoseconds, cf. **Figure 6a** and **b**, middle panels), our measurements also show photon bunching on much longer timescales (microsecond range, cf. **Figure 6a** and **b**, right-hand panels), thereby indicating the presence of long-living, site-specific metastable states [38]. In Eq. (5), τ_1 and τ_2 are the excited and metastable state lifetimes, whereas A and B are the antibunching and bunching amplitudes. The actual $g^{(2)}(0)$ values specified in the middle panels of **Figure 6** are obtained after considering the finite time resolution of the HBT setup and without

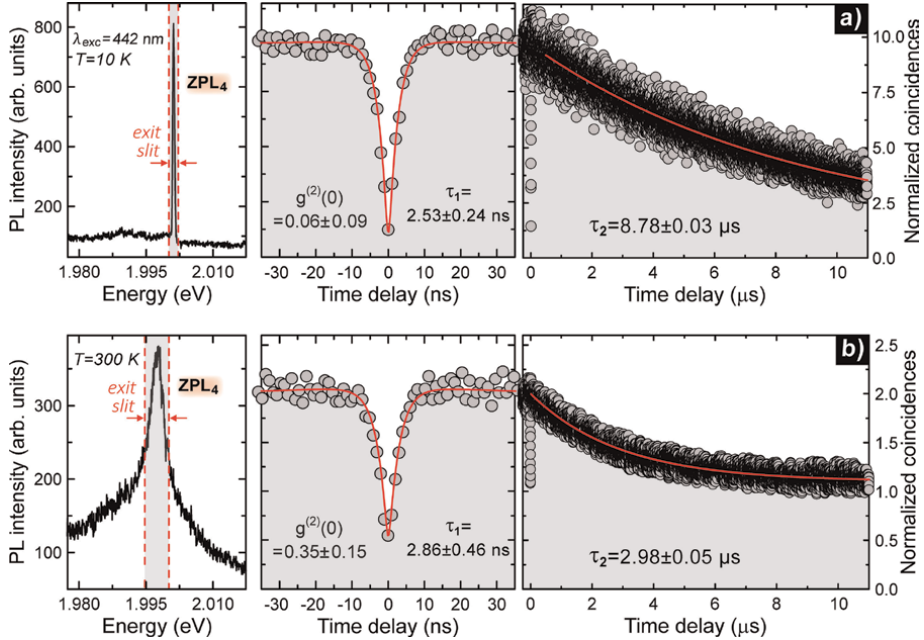


Figure 6.

(a), (b) Left-hand panels: The emission spectrum of a single h-BN defect excited with a 442 nm cw laser and recorded at an excitation power density of 70 W/cm^2 at 10 and 300 K, respectively. The dashed vertical lines represent the spectral bandwidth sent to the HBT interferometer. Middle panels: Normalized second-order autocorrelation function measured under the same excitation conditions as the corresponding PL spectra. The short timescale (tens of nanoseconds) shows photon antibunching. Right-hand panels: Photon bunching due to the presence of a long-living (tens of microseconds) metastable state. The experimental data are normalized so that $g^{(2)}(\tau \rightarrow \infty) = 1$.

applying any background correction. The latter is because the dark counts of the HBT detectors are very low compared to the measured single-photon count rate. The correction for temporal resolution was carried out by fitting our experimental results

with $g_{ideal}^{(2)}(\tau)$ convolved with the IRF of the form of $h(\tau) = C \cdot e^{-\left|\frac{\tau}{\tau_{IRF}}\right|}$. The resulting fitting function is as follows:

$$g_{meas}^{(2)}(\tau) = \int_{-\infty}^{\infty} h(\tau - \tau') g_{ideal}^{(2)}(\tau') d\tau' = 1 - A \cdot \frac{\tau_1^2 \cdot \tau_{IRF}}{\tau_{IRF}^2 - \tau_1^2} \left(\frac{e^{-\left|\frac{\tau}{\tau_{IRF}}\right|}}{\tau_1} - \frac{e^{-\left|\frac{\tau}{\tau_1}\right|}}{\tau_{IRF}} \right) + B \cdot \frac{\tau_2^2 \cdot \tau_{IRF}}{\tau_{IRF}^2 - \tau_2^2} \left(\frac{e^{-\left|\frac{\tau}{\tau_{IRF}}\right|}}{\tau_2} - \frac{e^{-\left|\frac{\tau}{\tau_2}\right|}}{\tau_{IRF}} \right), \quad (6)$$

where C is the normalization factor and $\tau_{IRF} \sim 350$ ps is the temporal resolution of the HBT interferometer, which is mainly determined by the time jitter of the employed APDs. At $\tau = 0$, the measured photon correlation is thus determined by:

$$g_{meas}^{(2)}(0) = 1 - A \cdot \frac{\tau_1}{\tau_1 + \tau_{IRF}} + B \cdot \frac{\tau_2}{\tau_2 + \tau_{IRF}}. \quad (7)$$

The actual quality of single-photon emitters in **Figure 6**, after correction for the experimental temporal jitter, with $g^{(2)}(0)$ dipping to below 0.5 values for both cryogenic (0.06 ± 0.09) and room (0.35 ± 0.15) temperature measurements, is then given by the antibunching value:

$$g^{(2)}(0) = 1 - A + B, \quad (8)$$

where A and B are extracted from fitting Eq. (6) to the measured data. As expected, the antibunching dip decreases at higher temperatures as a consequence of the increased background luminescence [40].

In total, we measured 34 SPEs (the same ZPLs as in **Figure 5c**), all showing the single-photon purity below the 0.5 two-photon threshold (not shown), which is a characteristic signature of single-photon quantum emitters [1].

In addition to high single-photon purity, one of the critical challenges in employing h-BN for scalable quantum technologies is controlling the spectral properties of single-photon emitters. Studies published in our work [41] and in [42] have demonstrated the use of SAWs to dynamically modulate defect-related emissions in h-BN. When the acoustic wave is applied, some of the optically probed quantum emitters undergo an apparent splitting of their emission lines, as seen in the case of the higher-energy ZPL₆ in **Figure 7a**. Since the magnitude of this SAW-induced splitting depends, in general, on the mechanical coupling strength between the probed defect and the SAW substrate, the fact that not all emission lines are affected by the propagating acoustic wave indicates that they most likely originate from different grains with different coupling strengths to the LiNbO₃ substrate. This assumption is supported by the experimental results in **Figure 5**, which demonstrate that different ZPLs display distinct photophysical characteristics and temporal behaviors. In fact, only a small fraction of radiative defects—just a few percent—are efficiently coupled to the SAW. This likely stems from the irregular shapes and sizes of the dispersed h-BN flakes, along with their random orientations, which result in varying mechanical contact between the flakes (and the embedded emitters) and the underlying SAW-carrying substrate. More specifically, effective acousto-mechanical modulation is expected only for those

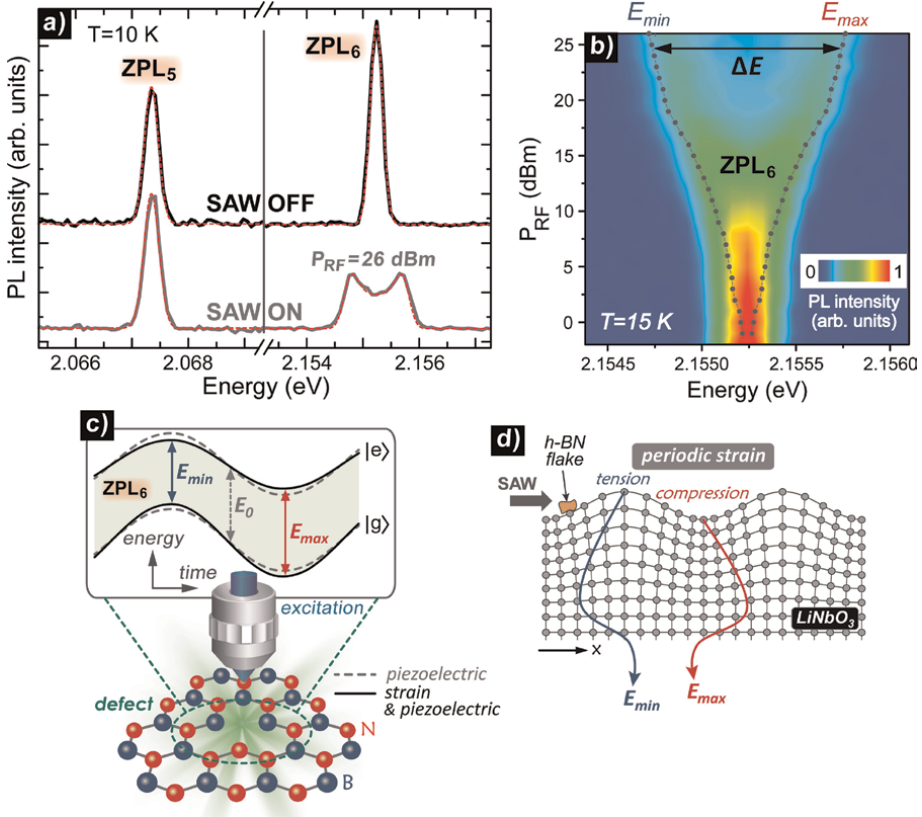


Figure 7.

(a) Low-temperature time-integrated PL spectrum from h-BN defects showing two different emission lines (ZPL₅ and ZPL₆) in the absence of the SAW (upper trace) and in the presence of the SAW generated by applying 26 dBm to one of the IDTs in **Figure 1a** (lower trace). Spectra are vertically offset for clarity. (b) False color plot of the PL emission from a single h-BN defect corresponding to ZPL₆ in (a) as a function of the driving SAW amplitude ($\propto \sqrt{P_{RF}}$), showing splitting of the associated optical transition into a doublet consisting of the lowest (E_{min}) and the highest (E_{max}) energy values separated by ΔE . The E_{min} and E_{max} values, extracted from fitting Eq. (9) to the experimental results and recorded at different RF levels at the surface of the LiNbO₃ substrate, are indicated by black dots. (c) Simplified energy level diagram of the optically probed h-BN defect (denoted by the dashed green circle), depicting the effect of the oscillating SAW strain on the defect's electronic structure. The optical transition energy is dynamically modulated around the ZPL energy E_0 with the amplitude of $\frac{\Delta E}{2} = \frac{E_{max} - E_{min}}{2}$. The SAW strain superimposes a small change in the modulation of the ground $|g\rangle$ and excited $|e\rangle$ energy levels induced by the oscillating piezoelectric field. (d) Strain field generated in the LiNbO₃ substrate by the SAW. The regions of maximum tension and compression are marked with E_{min} and E_{max} , respectively.

defects that are either in direct mechanical contact with the substrate (requiring a specific flake orientation), or are embedded in heavier flakes (providing better defect-to-SAW coupling). This observation is in good agreement with earlier studies which indicate that strain transfer from the bottom to the top layers in multilayer h-BN flakes [8], as well as in other two-dimensional structures [43], can be significantly restricted due to weak interlayer van der Waals interactions. The hypothesis that acoustic coupling strength is influenced by the quality of mechanical contact between the emitter and the vibrating substrate is further supported by our recent SAW experiments conducted on a comparable LiNbO₃ SAW chip with few-layer (i.e., lighter) h-BN flakes deposited along the SAW propagation path. In that case, no influence of the acoustic wave on the embedded defect centers was detected. A similar

phenomenon was previously observed in experiments involving randomly dispersed dot-in-a-nanowire semiconductor heterostructures exposed to acoustic waves on the surface of a LiNbO₃ SAW chip [22, 45]. In those samples, the strongest acoustic coupling to quantum dots embedded in the tapered, “pencil-like” tip region of the nanowires took place in cases where the pyramidal tip was in direct mechanical contact with the substrate [22]. Furthermore, in the same sample, depositing a thin SiO₂ layer over the randomly dispersed nanowires was shown to enhance mechanical coupling between the acoustic substrate and the embedded QDs. As a result, all nanowire-based QDs investigated under optical excitation and subjected to propagating SAWs exhibited pronounced SAW-induced spectral tuning, irrespective of the nanowire orientation (unpublished result). A similar enhancement in coupling strength is expected for h-BN flakes under comparable conditions.

To estimate the acousto-mechanical response of the optically probed and acoustically perturbed defect responsible for the observed ZPL₆ emission, we fitted the intensity $I(E)$ of this peak (dashed red traces in **Figure 7a**) with the Gaussian peak function of constant linewidth w and intensity A averaged over $T_{\text{SAW}} = \frac{1}{f_{\text{SAW}}}$:

$$I(E) = I_0 + \frac{1}{T_{\text{SAW}}} \cdot \frac{A}{w \cdot \sqrt{\frac{\pi}{2}}} \cdot \int_0^{T_{\text{SAW}}} e^{-2 \frac{(E-E_C(t))^2}{w^2}} dt, \quad (9)$$

where $E_C(t)$ is the time-varying emission energy which follows a sinusoidal temporal evolution with a modulation amplitude of $\frac{\Delta E}{2}$ around its unperturbed position E_0 [24, 45]:

$$E_C(t) = E_0 + \frac{\Delta E}{2} \cdot \sin\left(\frac{2\pi}{T_{\text{SAW}}} \cdot t\right). \quad (10)$$

The fitted line shape closely corresponds to the measured spectra, thus enabling a precise estimate of the SAW-governed modulation amplitude $\frac{\Delta E}{2}$. As seen in **Figure 7b**, the magnitude of this SAW-induced splitting $\Delta E = E_{\text{max}} - E_{\text{min}}$ increases with increasing the SAW amplitude, which is proportional to $\sqrt{P_{\text{RF}}}$. As seen in **Figure 7b**, the SAW-driven line splitting increases continuously with increasing the RF power applied to the transducer. These results indicate that SAW can be used to effectively fine-tune the emission energy from h-BN defects within a bandwidth up to ~ 2.5 meV for the highest acoustic amplitude. The same result is observed when using the SAW propagating in the opposite direction (cf. **Figure 1a**). We thus attribute the observed spectral changes to the periodic shifts of the electronic energy levels of the radiative defect induced by the deformation potential coupling between the emitting state and the oscillating SAW-driven mechanical vibration. In other words, as the SAW couples to the grain, the embedded emitter is dynamically strained and its optical transition is sinusoidally modulated in such a way that it oscillates between its minimum E_{max} and maximum E_{min} value with respect to its equilibrium energy E_0 in the absence of a SAW. The acousto-mechanical effect reported here is portrayed in **Figure 7c** for the anti-site nitrogen vacancy (N_BV_N) defect, which is one of the potential atomic configurations responsible for single-photon emission within the visible spectral range [8–10, 16, 31, 41]. The figure presents a schematic of the SAW-driven, time-dependent modulation of the ZPL transition energy, demonstrated for a simplified system involving an excited state $|e\rangle$ and a non-degenerate ground state $|g\rangle$. A comparable optical response to acousto-mechanical effects has been previously observed in quantum well [47, 48] and QD heterostructures [22, 23, 45, 46, 51] based on III–V semiconductors. In this scenario, the detected energy

splitting corresponds to the difference between the transition energies at the acoustic cycle phases representing maximal compressive and tensile strain, labeled E_{max} and E_{min} , respectively (cf. **Figure 7d**). Averaged over time, this modulation gives rise to an apparent splitting of the PL line ZPL₆.

In the case of SAWs propagating on a strongly piezoelectric substrate, such as LiNbO₃, the mechanical wave is accompanied by an oscillating piezoelectric field, which typically extends over a depth of roughly one acoustic wavelength (i.e., several micrometers) both above and below the surface of the SAW substrate [45, 46]. Unlike the SAW-induced strain field, which only affects defects with strong mechanical coupling to the substrate, the piezoelectric field is anticipated to influence all emission dipoles located within its reach. Given the random orientation of the h-BN flakes on the substrate, this field might also contribute to the modulation of the PL lines emitted from the embedded defects. Spectral shifts linked to the quantum-confined Stark effect driven by the SAW-induced piezoelectric field acting in the direction perpendicular to the defect plane can be estimated using the Stark tuning coefficients (as large as 5.4 nm per GV/m) that have been experimentally determined for atomic defects in h-BN flakes under externally applied out-of-plane static electric fields [16]. On the surface of a LiNbO₃ crystal, SAW-generated piezoelectric fields typically remain below a few tens of kV/cm [20, 22, 41, 45]. Consequently, the anticipated Stark shift resulting from the out-of-plane acousto-electric effect is less than 0.05 meV at the highest applied RF power, corresponding to the driving RF power of $P_{RF} = 26$ dBm. This shift is two orders of magnitude smaller than the values observed in our measurements. However, the Stark tuning coefficient for electric fields aligned within the emission dipole plane remains unknown and may result in larger spectral shifts. To gain more insight into the contribution of the in-plane acousto-electric effect on the observed spectral shifts of the ZPLs, we have performed a detailed analysis (plotted in **Figure 8a**) of the optical polarization behavior of various defect

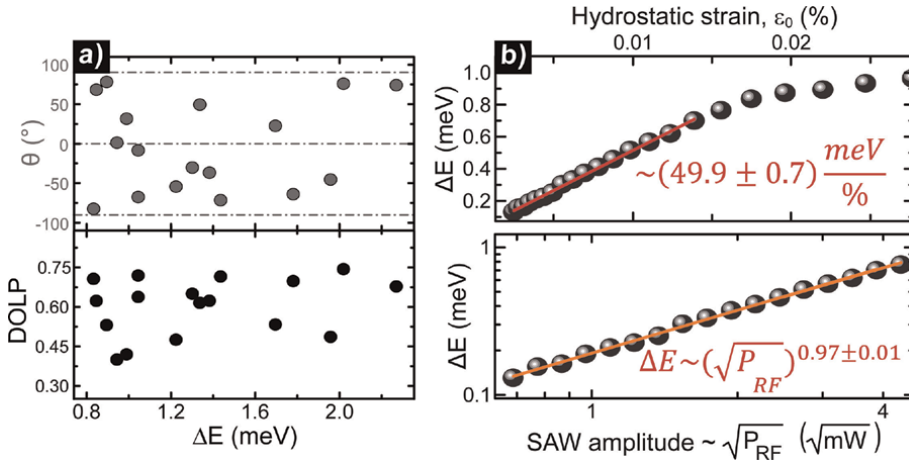


Figure 8.

(a) Degree of linear polarization (DOLP, lower panel) and in-plane polarization angle with respect to the SAW propagation direction (θ , upper panel) estimated for various h-BN defects represented as a function of SAW-induced splitting of the ZPL emission (ΔE). (b) Energy splitting ΔE as a function of the driving SAW amplitude ($\propto \sqrt{P_{RF}}$, lower panel) and of the SAW-induced hydrostatic strain (ε_0 , upper panel) at the LiNbO₃ surface. Red solid lines: Power-law fits to the experimental data reproducing the $\Delta E \propto (\sqrt{P_{RF}})^n$ dependence (lower panel) and a linear fit to the data (upper panel). Error bars for DOLP, θ and ΔE are negligible.

centers. This study reveals no clear correlation between the magnitude of the SAW-induced spectral modulation (ΔE in **Figure 8a**) and the in-plane projection of the transition dipole moment (as measured in **Figure 5a**). This finding is in contrast with the expected similar values of spectral shifts induced by the acousto-electric effect acting on defects with collinear dipole orientations. In fact, we observe defects with identical polarization angles and degree of linear polarization (DOLP) that exhibit both $\Delta E > 0$ and $\Delta E = 0$. We therefore conclude that the contribution of the SAW-induced piezoelectric field—regardless of its orientation relative to the defect symmetry axis—can be considered negligible when compared to the effect produced by the acousto-mechanical interaction.

Fit to the experimental data (red solid line) in the lower panel of **Figure 8b** reveals different strain dependencies at low and high SAW amplitudes. From the estimated slopes, we deduce a power-law behavior ($\Delta E \propto (\sqrt{P_{RF}})^n$), with an exponent n of 0.97 ± 0.01 —indicating an almost linear trend—at low excitation, and 0.27 ± 0.01 —indicating a non-linear behavior—at high acoustic power levels. The emergence of non-linear behavior at larger modulation amplitudes can be attributed to a partial detachment of the dispersed h-BN flakes from the LiNbO₃ substrate surface. Such decoupling could result from, for instance, slight flake rotation or interlayer sliding within our multilayered h-BN flake [8, 44]. This explanation is supported by the irreversible decrease or even disappearance of the PL line splitting observed for some defects affected by the propagating SAW, whereas the emission spectrum and dipole orientation of these defects remain unchanged before and after applying the acoustic wave. What's more, such non-linear and non-monotonic changes in the ZPL transition energy under static strain have also been suggested for h-BN layers containing N_BV_N defects [8, 9]. However, such effects are anticipated to occur at significantly higher strain levels than those employed in our study (see below). The behavior observed here also reinforces the claim that the acousto-electric modulation mechanism, driven by the SAW-induced piezoelectric field, plays a negligible role. This is because even the defect that becomes mechanically decoupled from the substrate when it interacts with the propagating SAW (while maintaining its dipole orientation) should still be influenced by the SAW piezoelectric field. Nevertheless, no hints of such acousto-electric modulation are evident in our experiments. The upper panel of **Figure 8b** displays the fitted ΔE values as a function of the amplitude of the hydrostatic strain induced by the applied SAW at the surface of the LiNbO₃ substrate. To determine the magnitude of hydrostatic strain acting on the optically probed defect center, we relate the mechanical deformation to the SAW amplitude using the numerical model described in Ref. [48]. Namely, for a SAW propagating on the surface of a LiNbO₃ crystal, the SAW strain field possesses three non-vanishing strain components ϵ_{XX} , ϵ_{ZZ} and ϵ_{XZ} in the reference frame defined in **Figure 4a**. At the acoustic phase marked with E_{min} (E_{max}) in **Figure 7**, corresponding to the minimum (maximum) transition energy, the shear component ϵ_{XZ} vanishes, and the hydrostatic strain s_0 is only comprised of a tensile (compressive) component ϵ_{XX} along the SAW propagation direction (i.e., x-direction), in addition to a smaller compressive (tensile) component ϵ_{ZZ} in the direction perpendicular to the substrate surface (i.e., z-direction): $s_0 = \epsilon_{XX} + \epsilon_{ZZ}$. This points to the hydrostatic strain on the surface of LiNbO₃ being on the order of $s_0 \approx 0.04\%$ for the maximum applied acoustic amplitude (see Refs. [22,41] for more details). Consequently, by assuming that most of the strain deformation is effectively transferred from the LiNbO₃ substrate to the h-BN flake positioned on top of it, and by neglecting the influence of the SAW-induced piezoelectric field, we estimate a

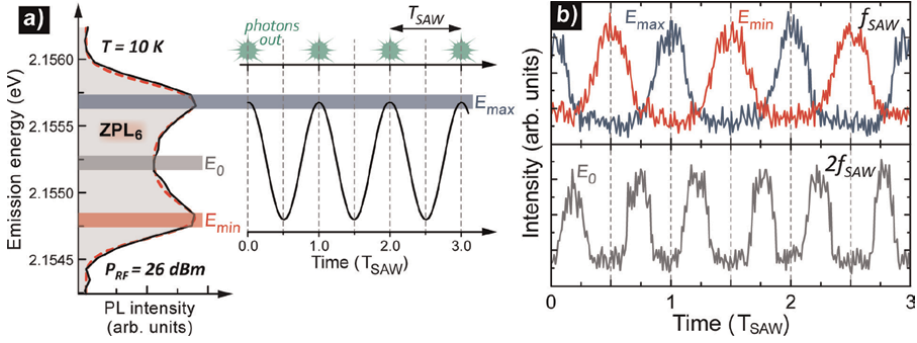


Figure 9.

(a) Left panel: Time-integrated PL spectrum showing one of the SAW-split ZPLs at $P_{RF} = 26 \text{ dBm}$ followed by the schematic drawing of the resulting spectral modulation as a function of time. Colored rectangles labeled E_{min} (red), E_0 (gray), and E_{max} (blue) represent the spectral detection window. Right panel: Temporal emission profile for the spectral window selected at E_{max} . (b) Time-resolved intensity of the ZPL emission in (a) spectrally filtered at energies marked by the corresponding colored rectangles and recorded under stroboscopic detection.

lower limit for the absolute deformation potential (i.e., the ratio $\frac{\Delta E}{\epsilon_0}$ [49]) of h-BN to be approximately 5 eV. This value is consistent with the results of DFT calculations for h-BN reported in Refs. [49, 50].

Our results show that the energy shift of the defect emission is of the order of 50 meV per % of strain. This tuning coefficient, extracted from the linear fit in **Figure 8b** (upper panel), is an order of magnitude higher than the value found experimentally in multilayer h-BN flakes under externally applied static strain [8]. This discrepancy is likely attributable to the improved quality of the physical coupling between the h-BN flake hosting the emitting defect and the oscillating substrate. Additionally, the above-mentioned decoupling of defects from the deformed substrate at high strain levels, as discussed in Refs. [8,9]—which involve strain magnitudes significantly larger than those used in our study—might also contribute to this difference. The magnitude of the elastic response of h-BN demonstrated here is an important result, given that such high elasticity of h-BN allows for effective strain engineering of its optical properties, which is particularly relevant for practical implementations of h-BN emitters into existing photonic and optoelectronic devices. The same effect can, in principle, also explain the giant spread of the emission wavelengths typically observed in the PL spectra of h-BN quantum emitters (cf. **Figure 4a**). Such a broad spectral distribution of ZPLs suggests that defects in h-BN can indeed couple to local lattice strain inducing large variations in their emission wavelength.

The SAW-driven dynamic fine-spectral tuning can further be used to control the photon emission time. As shown in **Figure 9a**, this can be achieved by collecting the photons in a narrow spectral range. Thus, by filtering the SAW modulated light at different energies, photons are emitted at different frequencies. As seen in **Figure 9b**, the light collected at the highest (E_{max} , blue trace) and lowest (E_{min} , red trace) energy is out of phase with each other and photons are emitted at the SAW frequency. In contrast, at the central energy (E_0 , gray trace), the emission takes place at twice the frequency of the SAW since the emitting state moves past this energy twice per acoustic cycle. As previously reported for III–V [46, 51] semiconductor QDs, such acoustically-regulated temporal control of photon emission provides an efficient tool for on-demand generation of single photons without the need for a pulsed laser. The advantage of this over other control schemes is that it allows on-demand production and manipulation of quantum light states over a wide frequency range (up to GHz

frequencies), which is particularly beneficial for future high-speed quantum information processing. This ability of *in situ* control of the single-photon emission time allows for the time-bin encoding of quantum information, which is a key resource in quantum cryptography. This is because this type of encoding offers various advantages over other existing schemes, including resilience against decoherence, which makes this method better suited for fiber optics applications compared to, for example, commonly used encoding exploiting the polarization degree of freedom [52]. In principle, even higher photon repetition rates can be easily achieved simply by changing the design of the acoustic transducer. Moreover, our findings further suggest that the photon emission wavelength can be selectively filtered by applying time-gating to the acoustically controlled luminescence originating from h-BN defects. This approach may be advantageous for minimizing the wavelength variations between different defect centers emitting within the spectral modulation bandwidth induced by the SAW. In general, broader bandwidths can be achieved by employing higher strain amplitudes.

4. Conclusions

In summary, we have studied the influence of surface acoustic waves on the highly linearly polarized non-classical light emitted from defect centers in h-BN flakes. Our results demonstrate that the dynamic strain induced by propagating acoustic waves emerges as a powerful “wireless” and nondestructive method for controlling the optical properties of quantum emitters embedded in h-BN flakes.

We have used this approach to achieve *in situ* and simultaneous tuning of both the photon emission wavelength and the emission time. By using the latter, we have achieved controlled photon emission timing, demonstrating the potential for acoustically driven time-bin encoding in quantum communication systems. By leveraging acousto-optoelectric modulation techniques, the functionality of h-BN-based quantum emitters can be significantly enhanced.

From a broader perspective, the strong elasto-optic coupling reported here in h-BN provides the capability to induce large spectral shifts (up to 50 meV per percentage of strain) of the emitted light, which is beneficial for eliminating the spectral inhomogeneity of atomic defects in h-BN. Furthermore, the dynamically modulated photon emission can be integrated with real-time gating techniques, thus facilitating the generation of energetically identical defect-based quantum emitters. As research progresses, we show here that h-BN combined with the dynamic sound waves stands as a promising platform for next-generation quantum photonic technologies, offering robust, scalable and tunable room temperature single-photon sources for future quantum light applications.

Altogether, our findings mark a significant step forward in the strain-engineered control of emission properties of atomic-scale defects in h-BN, holding considerable potential for applications in photonic quantum communication and information processing systems.

While SAW-driven modulation of single-photon emission in h-BN has demonstrated promising results, several challenges still remain. One key issue is to optimize the coupling between SAWs and h-BN emitters to maximize the modulation efficiency. Additionally, the integration of h-BN with piezoelectric substrates enables strain-engineered photonic devices. Future research may focus on engineering defect landscapes and optimizing emission wavelengths for targeted quantum applications.

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Conflict of interest

The authors declare no conflict of interest.

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Recent Research and Applications of Magnesium Diboride Superconductors

Agung Imaduddin and Satrio Herbirowo

Abstract

MgB₂ superconductor is known for its critical temperature of approximately 39 K, making it a strong candidate to replace Nb₃Sn and NbTi in superconducting coils for generating high magnetic fields. The Powder-In-Sealed-Tube (PIST) method is employed to synthesize bulk MgB₂, while the Powder-In-Tube (PIT) technique is used to fabricate MgB₂ wires. These synthesis methods yield materials with excellent sample quality. It is anticipated that mass production of MgB₂ wire will soon be feasible. Current research on MgB₂ primarily focuses on its application in wire form to achieve high magnetic fields to be used in MRI equipment. The primary challenge moving forward will be exploring the various applications of MgB₂.

Keywords: MgB₂, magnesium diborite, superconducting material, powder-in-sealed-tube method, powder-in-tube method, higher critical temperature

1. Introduction

Superconductors are unique materials that exhibit zero electrical resistance when cooled below a certain critical temperature. The phenomenon was first identified by Kammerlingh Onnes in 1911 [1, 2]. The subsequent discovery of superconducting materials like Nb₃Sn and NbTi around 1955, which have critical temperatures of 16 K and 8 K respectively, sparked a surge of interest in this field. One notable superconducting material is MgB₂, which boasts a higher critical temperature compared to other metal compound superconductors. This characteristic positions MgB₂ as a potential alternative to Nb₃Sn and NbTi for producing wires capable of generating high magnetic fields [3–5]. Additionally, it shows promise for use as a power cable to efficiently transport large amounts of electrical current [6]. Significant efforts are underway to develop superconducting wire from this material. In 1986, Bednorz and Muller made a groundbreaking discovery with Cu-based superconductors, leading to a continuous increase in critical temperatures, culminating in the identification of a T_c of 93 K in the Y-Ba-Cu-O system [7, 8]. This finding is particularly significant because liquid nitrogen, commonly used as a coolant for superconductors, has a melting temperature of 77 K. Thus, discovering materials with critical temperatures above 77 K was a major advancement at that time [6, 9, 10].

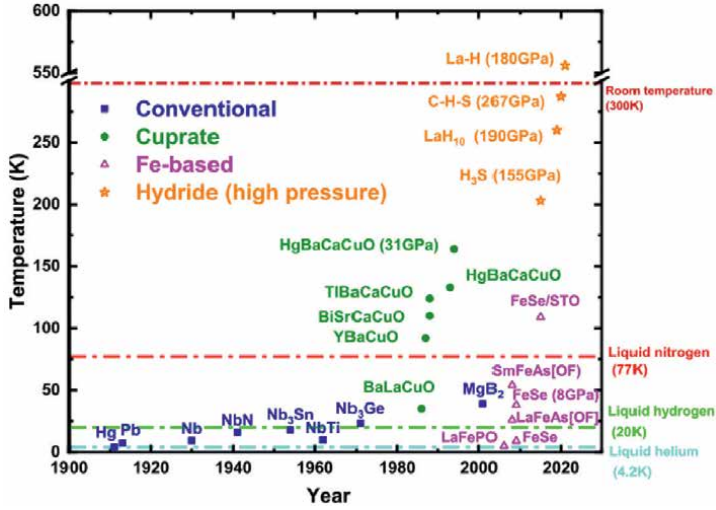


Figure 1.
Evolution of the T_c since the discovery of the superconductivity phenomenon [11].

Figure 1 illustrates the progression of T_c values identified since the initial discovery of superconductivity in 1911. MgB_2 was discovered by Nagamatsu et al. in 2001 [12]. Magnesium diboride (MgB_2) has gained attention as a promising superconducting material due to its relatively high critical temperature (~ 39 K), cost-effectiveness, and straightforward crystal structure [13]. Researchers are increasingly investigating MgB_2 for applications in energy transmission and high-field magnets [5, 14].

2. Aim and scope of this chapter

The aim of this chapter is to give an overview of the superconducting material MgB_2 , including its properties, synthesis, and applications. It brings together findings from three studies focused on the fabrication of MgB_2 superconducting wire using the powder-in-tube (PIT) method. The first study examines how powder filling density and thermomechanical treatments affect the material, the second looks at the impact of size reduction through cold rolling, and the third investigates hot working techniques with different initial filling densities. Together, these studies emphasize the importance of key process parameters on the microstructure, superconducting properties, and mechanical performance of MgB_2 wires. By combining these insights, this chapter aims to provide a thorough understanding of the optimization strategies for both MgB_2 bulk and wire fabrication.

3. Structural and physical properties of MgB_2

Figure 2 illustrates the crystal structure of MgB_2 [11]. This compound exhibits low anisotropy due to its simple hexagonal AlB_2 -type crystallographic structure [15]. It consists of two sheets of honeycomb boron atoms and a hexagonal layer of magnesium. The magnesium layer is arranged in a triangular matrix situated between the two boron layers. The lattice parameters for MgB_2 are $a = b = 3.0834$ Å and $c = 3.5213$ Å [11].

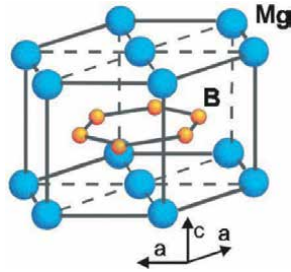


Figure 2.
 Crystal structure of MgB_2 .

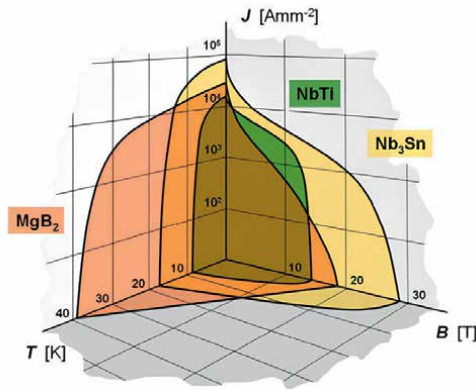


Figure 3.
 Critical surfaces of MgB_2 , NbTi , and Nb_3Sn [11].

When comparing superconductors, it is essential to evaluate their properties at critical temperature, critical magnetic field, and critical current density.

Figure 3 illustrates the critical temperature, critical magnetic field, and critical current density of MgB_2 in comparison to Nb_3Sn and NbTi . While MgB_2 boasts a higher critical temperature than Nb-based superconductors, it falls short in terms of critical magnetic field and critical current density when compared to Nb_3Sn [16].

4. MgB_2 material synthesis method

4.1 Synthesis of superconducting MgB_2 bulk using PIST method

The solid-state reaction (SSR) is the most commonly employed technique for producing MgB_2 bulks. This method involves heating a mixture of magnesium (Mg) and boron (B) powders that have been pressed at room temperature. The accompanying figure presents the phase diagram of the Mg-B system. The heating process typically occurs at temperatures ranging from 750 to 850°C for a duration of 2–3 hours. To prevent oxidation of the sample, the powder mixture is enclosed in a stainless-steel tube. A challenge encountered during the MgB_2 synthesis is the formation of cavities within the sample, which results from the evaporation of magnesium. To minimize

the formation of these cavities, the PIST process is implemented, which involves applying high pressure and adding approximately 5% more magnesium compared to the stoichiometric calculation.

Figure 4 illustrates the Mg–B binary phase diagrams. This diagram indicates that some magnesium melts at 600°C and evaporates at 1100°C. It is important to take into account the phase changes of magnesium when synthesizing MgB_2 through the solid-state reaction method [17].

This section presents a method for synthesizing bulk MgB_2 , focusing on how the composition of magnesium affects the synthesis process [18, 19]. The technique employed is known as PIST. The PIST method is advantageous for producing MgB_2 in bulk because it eliminates the need for vacuum or inert gas during sintering. A stainless-steel tube (SS316) is utilized in this method, offering greater reliability compared to Nb/Ta or Ni tubes. The synthesis involves using crystalline magnesium powders (98% purity, sized between 0.5 and 1 mm) and semicrystalline boron powders (95% purity, sized between 1 and 4 μm), which consist of both crystalline and amorphous forms. The process begins with weighing the raw materials, maintaining a mole ratio of magnesium to boron at x:2 [20]. The values of x were adjusted to 1.00, 0.95, 0.90, 1.05, and 1.10, corresponding to samples one through five, respectively. The magnesium composition for each sample is detailed in **Table 1**. The powders were mixed and ground using a hand agate mortar for 30 minutes. The blended powders were then placed into an SS304 stainless-steel tube with an outer diameter of 12.5 mm, a thickness of 1 mm, and a length of 80 mm. Both ends and the center of the tube were compressed at a pressure of 250 MPa to ensure a proper seal. The sealed tube was sintered in a furnace at 800°C for 2 hours under ambient atmospheric conditions, followed by a cooling period of approximately 24 hours within the furnace. After cooling, the samples were cut, and the MgB_2 was extracted from the tube in solid bulk form. Resistivity measurements of the samples were conducted using a four-point probe method.

Figure 5 illustrates the relationship between resistivity and temperature for all samples. The measurements ranged from room temperature down to 10 K. Each

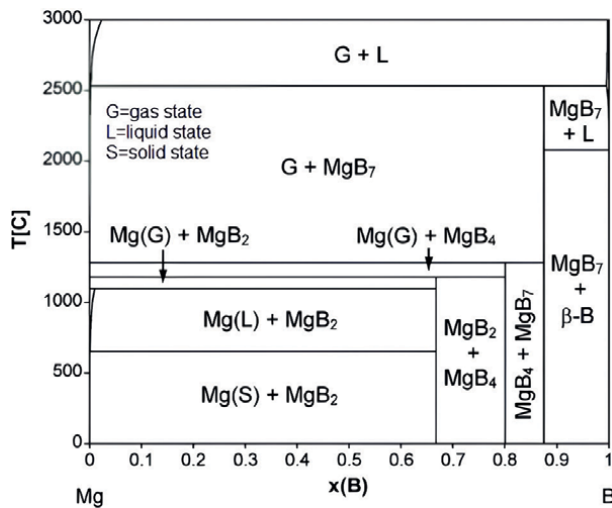


Figure 4. Mg–B binary phase diagrams at atmospheric pressure [11].

Sample	Form	x value in Mg_xB_2	Sintering temperature
1	Wire	1.0	700°C
2	Wire		800°C
3	Wire		900°C
4	Bulk		800°C
5	Wire	1.1	700°C
6	Wire		800°C
7	Wire		900°C
8	Bulk		800°C

Table 1.
List of samples.

sample exhibited a critical temperature near 40 K. As the temperature decreased, the resistivity of the samples also dropped. The resistivity versus temperature data presented in **Figure 5** can be modeled by the equation $\rho = \rho_0 + \rho_1 T^\alpha$, where α varies from 1.5 to 2.6 between T_c and 250 K.

Figure 6 presents the plots of normalized resistivity against temperature within the range surrounding critical temperature (T_c) [18]. Sample 4 ($x = 1.05$) exhibits higher $T_{C,\text{onset}}$ and $T_{C,\text{zero}}$ values compared to sample 1 ($x = 1.00$). As the Mg composition decreases (in samples 2 and 3) or becomes excessive (in sample 5), both $T_{C,\text{onset}}$ and $T_{C,\text{zero}}$ tend to decline. The optimal values for $T_{C,\text{onset}}$ and $T_{C,\text{zero}}$ occur at $x = 1.05$. The difference between $T_{C,\text{onset}}$ and $T_{C,\text{zero}}$ is minimal, approximately 0.4–0.8 K. All samples demonstrate clear superconducting properties. Notably, sample 5 displays a distinct curve in the resistivity versus temperature graph compared to the other samples. This variation is attributed to the high Mg composition, which leads to poorly formed crystallization of the MgB_2 phase and results in a high ρ_0 value, indicating poor electrical conductivity (high residual resistance).

Figure 7 illustrates the relationship between magnetization and temperature for samples 1, 2, and 4. The magnetization data were gathered under two conditions:

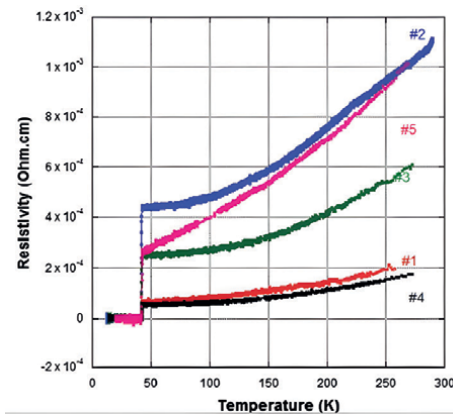


Figure 5.
Temperature dependence resistivity pada sample [18].

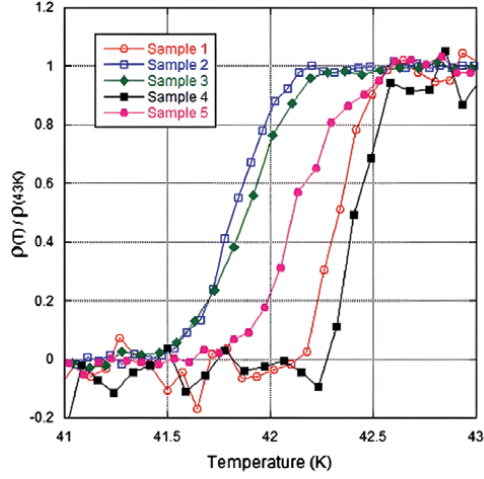


Figure 6.
Plots of normalized resistivity versus temperature [18].

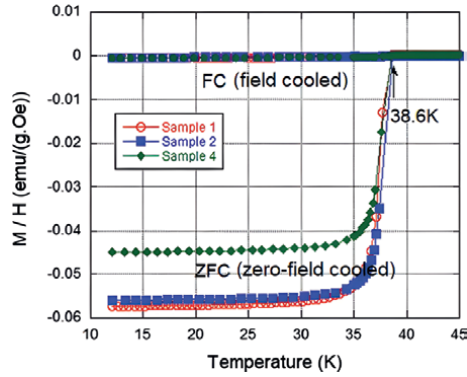


Figure 7.
Magnetization versus temperature for samples 1, 2, and 4 [18].

field cooled (FC) with an applied field of 100 Oe and zero field cooled (ZFC). The superconducting transition in all three samples appears sharp, indicating high-quality crystal formation. Samples 1 and 2 exhibit similar superconducting properties (diamagnetic), while sample 4 demonstrates weaker diamagnetic characteristics [21, 22]. There is a notable difference in the critical temperature values obtained from resistivity versus temperature measurements (approximately 42.5 K) compared to magnetization versus temperature measurements (around 38.6 K). This discrepancy is likely due to the nature of the resistivity measurement, which reflects the bulk properties of the sample, whereas the magnetization measurement focuses on the surface properties. According to this report, the absence of magnesium resulted in good crystallization of the MgB_2 phase but led to poor electrical conductivity, which in turn decreased the T_C value. Conversely, an excess of magnesium of up to 5% resulted in poor crystallization of MgB_2 but improved electrical conductivity, thereby increasing the T_C value. Additionally, a 10% excess of magnesium caused both poor crystallization of the MgB_2 phase and reduced electrical conductivity.

4.2 Synthesis of superconducting MgB_2 wire using PIT method

This section presents the synthesis research of bulk and wire MgB_2 [24, 25]. **Figure 8** illustrates the synthesis process for MgB_2 wire, utilizing the PIT method for bulk and the PIT method for wire. While the PIT and PIT processes are quite similar, the PIT method does not include the drawing and rolling stages [23]. To create the samples, crystalline Mg (98% purity, 0.3–0.5 mm size) powders and semicrystalline B (95% purity, 1–4 μm size) were used. The fabrication process began with weighing the raw materials, maintaining a mole ratio of Mg to B at $x:2$, where x can be either 1.0 (normal Mg composition) or 1.1 (excess Mg composition) [26]. Each sample was sintered at temperatures of 700°C, 800°C, and 900°C, as detailed in **Table 1**. The powders were mixed and ground in an agate mortar for 30 minutes. The resulting mixture was then placed into a stainless-steel tube (SS316), which has outer and inner diameters of 8 mm and 4 mm, respectively. For bulk samples (short samples), the SS316 tube was approximately 50 mm long, while for wire samples (long samples), it measured around 500 mm. To prevent oxidation during sintering, both ends of the samples were sealed under a pressure of 300 MPa. For the short samples, the center of the tube was also pressed. The sealed tubes for short samples were sintered in a furnace for 2 hours in an ambient atmosphere, followed by a cooling period of about 24 hours in the furnace. In contrast, the long samples were sintered in a tube furnace, and during this process, they were pulled out at a rate of 5 cm per hour. Afterward, the long samples were rolled using a rolling machine to reduce the outer diameter from 8 mm to 4 mm and then drawn with a drawing machine to decrease the diameter to 1.8 mm, creating superconducting wires. The short samples were cut, and the MgB_2 samples were extracted from the tube in solid form (bulk samples). The long samples were also cut into approximately 20 mm pieces.

Table 1 shows the list of samples made in the paper. There are eight bulk and wire samples, and variations in Mg content and sintering temperature were carried out.

Figure 9 illustrates the normalized resistivity values of MgB_2 wire with diameters ranging from 1.8 to 4.0 mm. At a diameter of 4.0 mm, no critical temperature was detected. The onset and zero values of T_c were identified in wires with diameters of 3.5 mm and 3.0 mm. However, the critical temperature was not observed again in wires with diameters of 1.8 mm and 2.5 mm.

Figure 10 shows the temperature dependence resistivity of samples 4 and 8 (bulk superconductors).

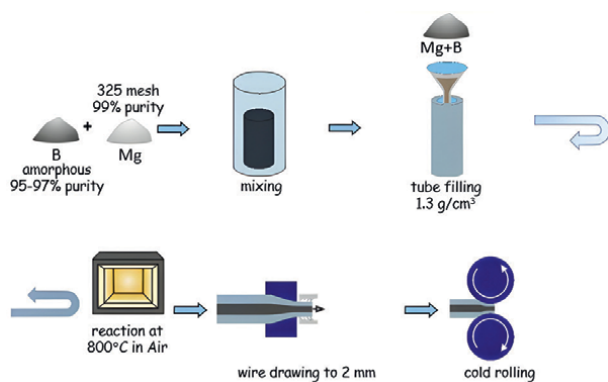


Figure 8.
 The process of synthesis of MgB_2 wires using Powder-In-Tube (PIT) method [23].

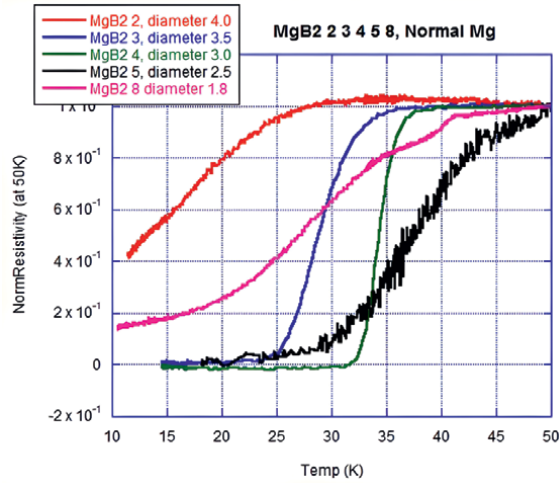


Figure 9.
Normalized resistivity value of MgB_2 wire with a diameter of 1.8–4.0 mm.

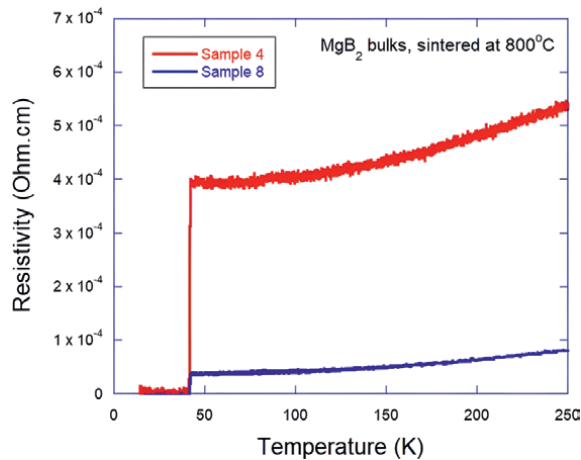


Figure 10.
Temperature dependence resistivity of bulk superconductors.

Figure 11 illustrates the temperature-dependent resistivity of superconducting wires in samples 2 (with a normal amount of Mg elements) and 6 (with an excessive amount of Mg elements). This figure indicates that synthesizing MgB_2 wire requires an excess of Mg elements. To create MgB_2 multifilament wires, SS316/ MgB_2 monofilament wires are combined and placed inside a Cu tube.

Figure 12 displays a photo of MgB_2 multifilament wire created by Imaduddin et al., featuring specifications that include three monofilaments and an outer diameter of 6 mm [23].

Additionally, some researchers have reported methods for producing MgB_2 wire that involve variations in filling density, reductions in wire size, the application of hot rolling, and the use of amorphous boron [27–29]. The process utilized high-purity magnesium (Mg) and boron (B) powders. Stainless-steel (SS 316 L) tubes were chosen

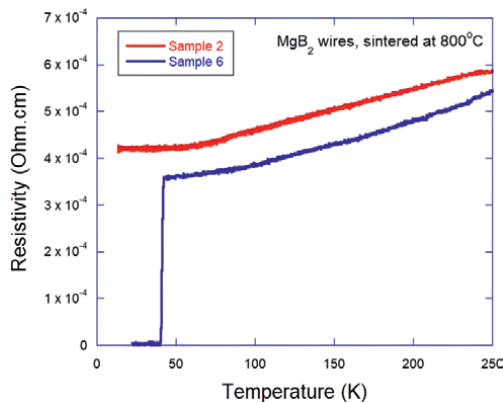


Figure 11.
Temperature dependence resistivity of superconducting wires.

as the sheathing material due to their cost-effectiveness, mechanical strength, and compatibility with MgB_2 . Key differences among the studies are outlined in **Table 2**.

The Mg and B powders were mixed in a 1:2 molar ratio and ground for uniformity. The PIT method involved compacting the powder into SS tubes, sealing both ends to prevent oxidation, and subjecting the samples to sintering at 800°C for one hour.

- Filling density study: Three filling densities (100%, 90%, and 80% theoretical maximum density) were examined. The tubes were reduced in diameter *via* hot rolling.
- Size reduction study: Post-sintering, wires underwent cold rolling with size reductions ranging from 30–60%.
- Hot working study: Initial filling densities of 60%, 80%, and 100% were tested. Tubes were sintered and hot-rolled immediately to form wires.

The study investigated the influence of powder filling density, size reduction, and hot working techniques on the properties of MgB_2 wires [27, 29, 30]. Wires with an 80% filling density demonstrated the highest superconducting properties, achieving a critical onset temperature ($T_{\text{C,onset}}$) of 39.25 K. The critical onset temperature varied



Figure 12.
Image of Cu/SS316/ MgB_2 multifilament wire.

Study	Boron type	Tube diameter (Initial)	Sheathing technique	Size reduction method
Filling Density Study	Amorphous	6 mm	Hot Rolling	Diameter reduced to 2.8 mm
Size Reduction Study	Amorphous	6 mm	Cold Rolling	Diameter reduced to 2.5 mm
Hot Working Study	Crystalline/ Amorphous	6 mm	Hot Rolling	Final dimensions varied

Table 2.
Summary of experimental parameters for MgB_2 wire fabrication.

Diameter (mm)	$T_{c,\text{onset}}$ (K)	$T_{c,\text{zero}}$ (K)
4.0	39.25	35.16
3.5	39.44	33.40
3.0	39.02	N/A

Table 3.
Summary of critical temperature for MgB_2 wire fabrication.

with diameter, with wires of 3.5 mm diameter achieving the highest $T_{c,\text{onset}}$ of 39.44 K, as summarized in **Table 3**.

Table 3 presents the T_c values for MgB_2 wire with different wire diameters. The data indicates that the critical temperature of MgB_2 wire reaches its peak at a diameter of 3.5 mm.

Figure 13 illustrates how the resistivity of MgB_2 wires varies with temperature when processed through hot rolling at filling densities ranging from 60 to 100%. The data shows that a lower filling density, specifically 60%, results in a lower resistivity. In contrast, a higher filling density of 100% corresponds to a higher critical temperature. The application of hot working techniques has further contributed to better densification and grain refinement. These wires reached a critical temperature onset of 39 K and exhibited low resistivity at cryogenic temperatures, as depicted in **Figure 13**.

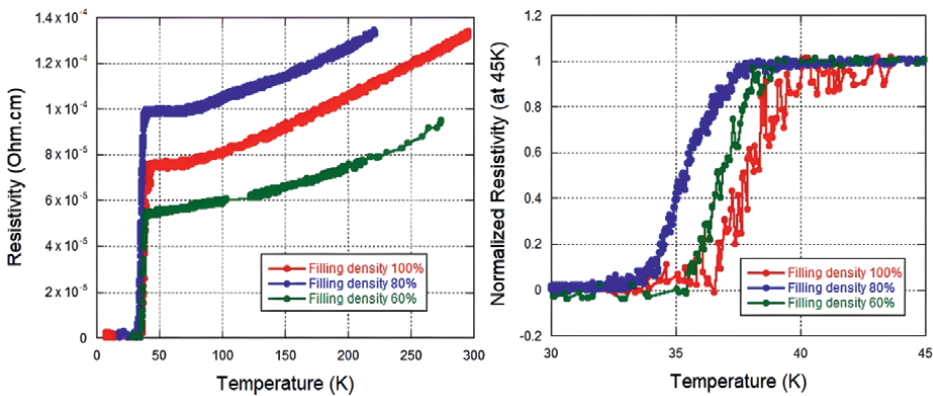


Figure 13.
Resistivity measurement of hot-worked MgB_2 wires.

Additionally, the hot rolling process improved the wires' durability and reduced porosity, leading to enhanced mechanical properties.

These studies demonstrate the critical role of filling density, size reduction, and hot working techniques in optimizing the performance of MgB_2 superconducting wires. Key findings include:

1. Lower filling densities improve crystallinity and grain connectivity, enhancing superconducting properties.
2. Cold rolling effectively reduces diameter while maintaining structural integrity, resulting in superior mechanical and superconducting performance.
3. Hot working enhances grain refinement, densification, and mechanical strength, particularly at optimal filling densities (e.g., 60%).

5. Application in medical (MRI equipment)

Magnetic Resonance Imaging (MRI) is a medical device that creates detailed images of the inside of a patient's body using a strong magnetic field and radiofrequency resonance waves. Currently, the largest commercial market for superconducting wire is in MRI systems [4, 31]. The effectiveness of these systems depends on the price-performance ratio (\$/kA m) of the wire at specific temperatures and background fields. MRI equipment is widely utilized in hospitals around the globe [14]. To achieve the necessary high magnetic field, a superconducting wire is employed. The most common superconducting wires used in MRI are Nb_3Sn and NbTi . However, both Nb_3Sn and NbTi have limitations due to their very low critical temperatures, which are 16 K and 8 K, respectively. MgB_2 superconducting wire has the potential to make a significant impact in this field. Research is ongoing to explore the use of MgB_2 as a replacement for Nb_3Sn and NbTi wires. While Nb_3Sn and NbTi have been the standard for MRI applications, MgB_2 faces challenges, particularly its reduced superconductivity in high magnetic fields. A study by Patel et al. investigated the connection methods for MgB_2 wire and its application as a superconducting wire in MRI, achieving a current value of 17.9 A at 20 K in a 0.5 T field [3, 4].

6. Conclusion

Recent studies on MgB_2 suggest that this material could potentially replace Nb-type superconducting wire. One of the key advantages of MgB_2 is its abundant raw materials and significantly higher critical temperature. Overall, the synthesis process for MgB_2 , whether in bulk or wire form, has been well established. This process is relatively straightforward and can be completed in a short timeframe. It is anticipated that mass production of MgB_2 wire will soon be feasible. The primary challenge moving forward will be to explore the various applications of MgB_2 . Future research is expected to focus heavily on the use of MgB_2 in wires for generating magnetic fields and in magnetic sensors [11, 31]. Since 2001, there has been considerable progress in developing long-length MgB_2 superconductor wires, and the performance of these wires is projected to improve significantly in the coming years [31].

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Conflict of interest

The authors declare no conflict of interest.

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Section 2

Industrial Performance and Environmental Sustainability of Boron-Based Materials

Boron Nitride for Gas Sensing Applications

Ankita Yadav and Mukesh Kumar

Abstract

Hexagonal boron nitride (h-BN) is a notable inorganic compound that has gained significant attention due to its unique combination of electrical insulation, high thermal conductivity, lightweight structure, and chemical stability. Over the past decades, various h-BN nanostructures have emerged, retaining the material's intrinsic advantages while imparting unique nanoscale properties. In this study, h-BN was synthesized using a boron oxide-assisted chemical vapor deposition (BOCVD) method, which facilitates in situ boron oxide vapor generation and promotes h-BN growth through its reaction with a nitrogen source. This method is now considered a benchmark in h-BN synthesis, offering high yield, purity, and scalability—qualities that make it highly suitable for industrial applications. Further analysis, including XRD for phase and grain size determination, as well as SEM and TEM for microstructural characterization, was performed on the synthesized h-BN, aiming to explore its potential for gas-sensing applications.

Keywords: hexagonal boron nitride (h-BN), microstructure, TEM, gas sensing, nano materials, synthesis methods

1. Introduction

Boron nitride (BN) nanomaterials are structurally analogous to those made of carbon. Like carbon, the unique properties of BN nanomaterials. BN nanomaterials exhibit exceptional thermal stability, maintaining structural integrity at temperatures above 1100 K. This makes them suitable for high-temperature applications, including thermal interface materials and high-temperature coatings. *Mechanical hardness* with a Young's modulus around 1 TPa, BN nanomaterials are extremely hard and robust [1–3]. This mechanical strength is particularly advantageous in applications requiring wear resistance, such as cutting tools and protective coatings. Chemical vapor deposition (CVD) is the most popular and successful method for synthesizing high-quality boron nitride (BN) nanomaterials [4, 5]. CVD offers several advantages that make it particularly well-suited for this purpose. The CVD process allows for precise control over the growth parameters, such as temperature,

pressure, and gas flow rates, leading to well-defined structures with tailored properties [6]. This is especially important for synthesizing hexagonal boron nitride (h-BN) with specific layer thicknesses. CVD can be used to synthesize various forms of BN, including thin films, nanotubes, nanosheets, and nanoribbons, by simply adjusting the precursor materials and reaction conditions. While CVD is a powerful method for producing high-quality BN materials, optimizing the process for even better quality and larger-scale production requires a deep understanding of the underlying growth chemistry and mechanisms. This understanding is difficult to achieve purely through experimental techniques due to various challenges like nanoscale chemistry, catalyst interface, and limitation of high energy resolutions, etc [7]. While the direct experimental observation of the nanoscale growth processes in BN nanomaterials remains challenging, the integration of advanced computational methods, in situ characterization, and data-driven approaches can help bridge the gap. By combining these tools, researchers can achieve a deeper understanding of the growth mechanisms, ultimately leading to improved control over the quality and scale of BN nanomaterial production [8]. The synthesis and characterization of h-BN are critical to understanding and optimizing its properties for functional applications. Among the techniques employed, X-ray diffraction (XRD) is essential for confirming the crystalline structure, while scanning electron microscopy (SEM) and transmission electron microscopy (TEM) provide detailed insights into the material's morphology and nanoscale features. High-resolution TEM (HR-TEM) and selected area electron diffraction (SAED) analyses further contribute to identifying the polycrystalline nature and lattice structures, shedding light on the material's intrinsic properties [9–11].

2. Materials and methods

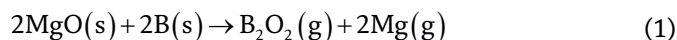
Magnesium oxide (MgO), ferrous oxide (FeO), boron powder, acetone, ammonia (NH₃), synthesized powder, and HNO₃.

2.1 BOCVD synthesis of BNNTs

The preparation and setup for synthesizing boron nitride nanotubes (BNNTs) on silicon (Si) wafers using a chemical vapor deposition (CVD) method. The process involves preparing a catalyst mixture, setting up the reaction environment, and placing the Si wafers in a specific configuration within the furnace [12–15]. (**Figure 1**) depicts a schematic illustration of a synthesis process for boron quantum dots (B-QDs) via a high-power sonication and chemical exfoliation technique.

2.2 Experimental

In the BOCVD method B, MgO and FeO powders are used as precursor materials, and NH₃ is used as nitrogen source. Boron reacts with MgO and FeO when heated by induction heating to $\leq 1300^{\circ}\text{C}$ according to reactions



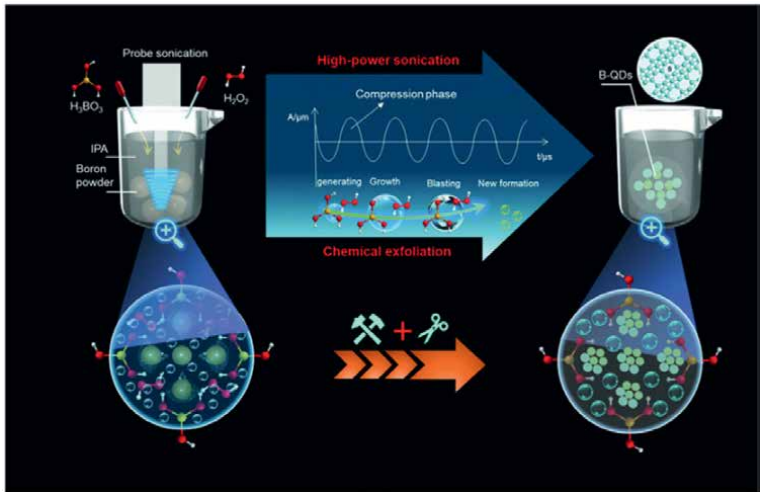
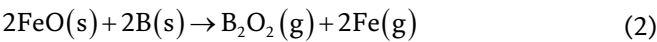
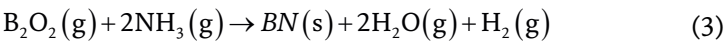


Figure 1.
Illustration of procedure of BN-QDs.



The resulting boron oxide gas reacts with the decomposed ammonia gas and creates BNNTs as the reaction



This method allows BNNTs to be synthesized in vertical induction furnaces with high yield. However, this method requires specially designed vertical induction furnace, high temperature, and large temperature gradient, all of which are undesirable demands for the large-scale synthesis of BNNTs. **Figure 2** showcases multiple configurations for synthesizing BN materials: Tube furnaces (a, b, c) offer high-temperature environments and NH₃ as a nitrogen source, with setups adapted for different precursors and deposition methods. The induction furnace (d) provides precise temperature control, suitable for advanced BN synthesis techniques like boron nitride nanotubes or thin films.

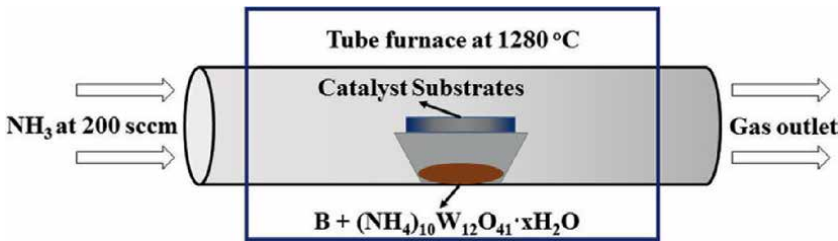


Figure 2.
Schematic experimental setup for the CVD synthesis of BNNTs. Source Ref. [16].

3. Results and discussion

3.1 Morphology of synthesized BNNTs

SEM images reveal both BN nanotubes (BNNTs) and BN nanostructures. BNNTs are characterized by their long, thin shapes with relatively uniform diameters, while the nanostructures exhibit irregular shapes, clustering together to form cauliflower-like patterns of varying sizes (as seen in **Figure 3** which is reprinted from ref. [6]). The SEM image of the samples prepared at 1200°C reveals BN nanospheres of varying sizes along with BNNTs dispersed across the sample's surface.

The provided SEM images showcase the nanoscale morphology and structural features of hexagonal boron nitride (h-BN) synthesized under specific experimental conditions. The top left image reveals entangled tubular nanostructures with diameters ranging from 30 to 50 nm, likely representing BN nanotubes or nanosheets with tubular formations, which offer a high surface area crucial for applications such as gas sensing and adsorption [15, 17–19]. The top right image displays a combination of densely packed spherical and tubular structures, suggesting the formation of boron oxide or BN particles during synthesis, indicative of controlled reaction parameters. The bottom left image highlights a mixture of tubular nanostructures and larger spherical agglomerates, with diameters of 28–36 nm, demonstrating a balanced morphology that can enhance the material's multifunctional properties, such as thermal insulation and gas adsorption. The bottom right image features larger, highly entangled structures with rod-like formations, which indicate polycrystallinity and defect sites—key features that could improve the material's catalytic activity and

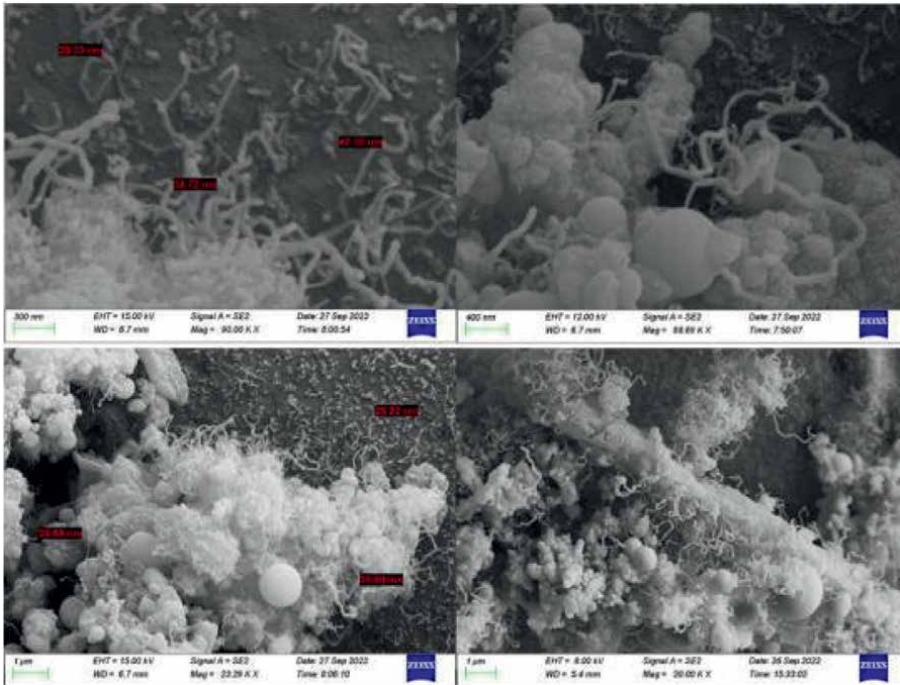


Figure 3. SEM image of the samples prepared at 1200°C reveals BN nanospheres of varying sizes along with BNNTs dispersed across the sample's surface. Image is reprinted from Ref. [6].

gas adsorption capabilities. Overall, these SEM images underscore the uniformity, morphological diversity, and high surface area of the synthesized h-BN, making it highly suitable for advanced applications in gas sensing, thermal management, and catalysis, while also reflecting the effectiveness of the synthesis process, likely aided by catalytic control and optimized reaction conditions [20–24].

The Stopping and Range of Ions in Matter (SRIM) simulations show how boron ions (B^+) implanted into h-BN at an energy of 150 keV create atomic vacancies along their path. The graph demonstrates that these ions penetrate to a depth of about 0.4 μm in the material. The number of vacancies formed per ion and per unit length (measured in $\text{\AA}$) is shown in the curve. To calculate the total damage or vacancy concentration, $(C(x))$, the average number of vacancies created per ion (C_p) is multiplied by the ion fluence (D), which is the total number of ions implanted per unit area (**Figure 4**).

In order to better understanding of the provided image, a detailed explanation is given.

- a. *XPS survey spectrum*: The wide-scan X-ray photoelectron spectroscopy (XPS) survey shows the presence of boron (B 1 s) and carbon (C 1 s) in both B-QDs and bulk boron. The intensity of the B 1 s and C 1 s peaks is noticeably higher in B-QDs, indicating surface chemistry differences and the potential presence of functional groups enhancing quantum dot properties.
- b. *High-resolution B 1s spectrum*: This panel focuses on the detailed analysis of the B 1 s peak. For B-QDs, the B 1 s peak shifts to 192.2 eV compared to bulk boron (189.9 eV), suggesting oxidation states or surface modifications, likely due to the formation of boron-oxygen bonds.
- c. *High-resolution C 1s spectrum*: The C 1 s spectrum highlights peaks at different binding energies for B-QDs and bulk boron. For B-QDs, the C 1 s peak shifts to 286.1 eV, indicating the presence of oxygenated carbon species, which may result from surface functionalization or interaction with the environment.

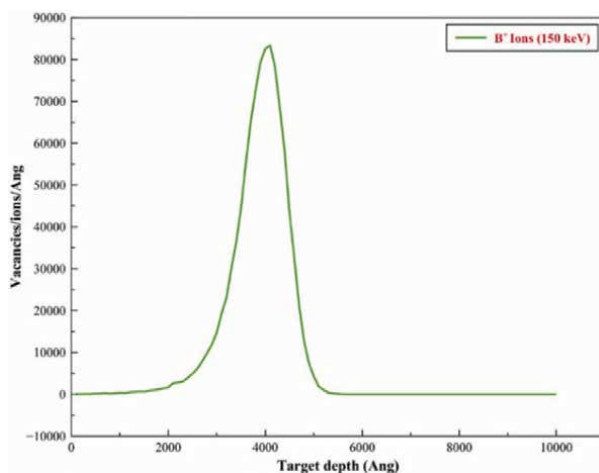


Figure 4.
 Graph produced by the SRIM model depicts the relationship between penetration depth and the number of vacancies per ion per unit length (in $\text{\AA}$) for boron ions at an energy of 150 keV [6].

- d. *XRD patterns*: The X-ray diffraction (XRD) patterns display distinct differences between B-QDs and bulk boron. B-QDs exhibit broadened peaks, indicating smaller particle sizes and a possible amorphous or semi-crystalline nature, while bulk boron shows sharper, well-defined peaks, reflecting its crystalline structure.
- e. *Raman spectra*: The Raman spectra for B-QDs show characteristic peaks, including a strong band at 2433 cm^{-1} , which corresponds to unique vibrational modes of B-QDs. Bulk boron exhibits distinct peaks at lower intensities, signifying differences in molecular vibrations and structural properties.
- f. *FTIR spectra*: The Fourier transform infrared (FTIR) spectra show characteristic peaks corresponding to specific functional groups. For B-QDs, a broad peak at 3415 cm^{-1} indicates O-H stretching, and peaks at 1635 cm^{-1} and 1385 cm^{-1} represent other oxygenated species, confirming surface functionalization. In bulk boron, these peaks are less prominent, indicating fewer functional groups.
- g. *NMR spectra*: The nuclear magnetic resonance (NMR) spectra of ^{11}B demonstrate the chemical environment around boron atoms. For B-QDs, the peaks are spread across a wider range, reflecting a more diverse chemical environment due to functional groups and quantum effects. Bulk boron shows narrower peaks, representing its homogeneous chemical environment.
- h. *Schematic representation*: The schematic illustrates the structural differences between B-QDs and bulk boron. It highlights the core-shell structure of B-QDs with functionalized surfaces and compares the χ_3 and β_{12} lattice types, explaining the quantum effects and unique properties of B-QDs.

SEM images of the growth results for MgO , K_2CO_3 , and B with a molar ratio of 1:1:4 reveal the deposition of white products, including slender BNNTs, on SiO_2/Si substrates at growth temperatures of 1000 and 1100°C (**Figure 5a, b**). At a growth temperature of 900°C , a minimal quantity of product was acquired on the SiO_2/Si substrate (**Figure 5c**). The synthesis was unsuccessful at 800°C (**Figure 5d**). X-ray diffraction (XRD), and Raman spectroscopy analyses were performed on the products obtained on SiO_2/Si substrates at growth temperatures of 900, 1000, and 1100°C . The peaks in the XRD patterns correspond to characteristic signals of h-BN (JCPDS No. 73–2095), confirming that the as-grown samples exhibit h-BN structures (**Figure 5e**). The Raman spectra display a distinct absorption band at 1368 cm^{-1} , corresponding to the $\text{E}_{2\text{g}}$ in-plane vibration mode of h-BN (**Figure 5f**). The XRD patterns indicate that the products in the BN boat include BN (JCPDS No. 73–2095), MgO (JCPDS No. 87–0652), and a small amount of B_{13}C_2 (JCPDS No. 71–0108), which forms due to the reaction between CO_2 released from K_2CO_3 decomposition and B. These results suggest that the precursors efficiently transform into BN even at relatively low temperatures.

The morphologies of the derivative boron nanospheres (DBS) were examined using TEM and SEM. (**Figure 6a**) clearly shows that the obtained DBS have a spherical shape with a diameter of approximately 150 nm. The TEM image in **Figure 6b** reveals that the nanospheres are composed of ultra small boron quantum dots (B-QDs) and some cavities. These cavities likely result from the removal of organic components due to electron beam irradiation during the TEM measurements. The HR-TEM image in **Figure 6c** shows the interconnected B-QD particles with a lattice

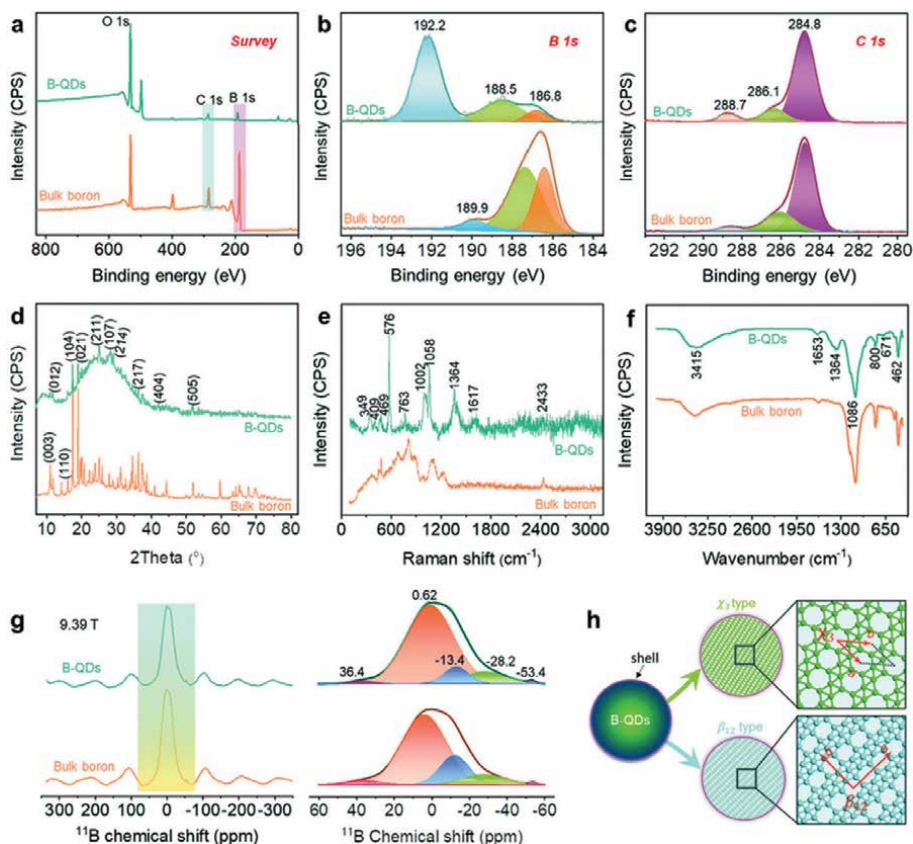


Figure 5. Comprehensive structural and chemical analysis of boron quantum dots (B-QDs) compared to bulk boron using XPS (a-c), XRD (d), Raman spectroscopy (e), FTIR (f), NMR (g), and structural modeling (h), highlighting their unique characteristics and surface functionalities. Images reprinted from Ref. [6].

fringe of 0.46 nm, corresponding to the (021) lattice planes of boron. The selected area electron diffraction (SAED) pattern in **Figure 6d** displays a spotty ring pattern, indicating a mild polycrystalline nature, clearly attributed to the diffraction of the (021) boron lattice planes. The HAADF-STEM images in **Figure 6e** confirm the inner structural features observed in **Figure 6b**. The EDX mappings reveal a uniform distribution of B, C, N, and O elements, with nitrogen originating from the hydrazine hydrate. This suggests that hydrazine hydrate interacts with numerous B-QDs to form spherical particles. After calcining the DBS at 500°C, the diameter of the particles shrinks to around 30 nm due to the removal of hydrazine hydrate, resulting in amorphous boron [25–34].

This image provides a comprehensive analysis of synthesized calcium oxide (CaO) nanoparticles, using various characterization techniques:

- (a) and (b): TEM images:** These transmission electron microscopy (TEM) images showcase the morphology and distribution of CaO nanoparticles. The highlighted regions (yellow circles) indicate agglomerated clusters of nanoparticles. The particles appear irregularly shaped with sizes in the nanometer range, confirming their nanoscale nature.

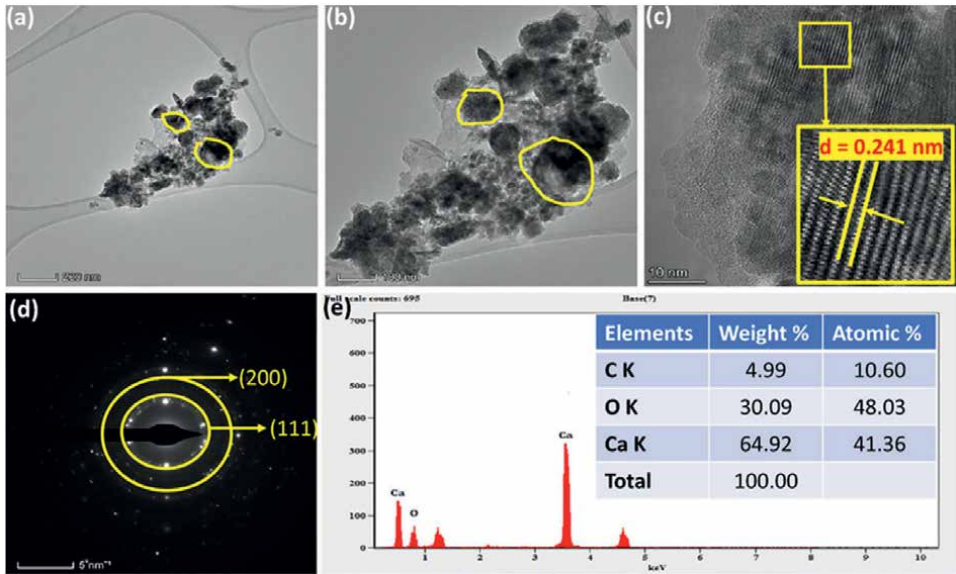


Figure 6. Characterization of calcium oxide (CaO) nanoparticles: (a, b) TEM images showing aggregated nanoparticles with highlighted regions of interest. (c) High-resolution TEM (HRTEM) image showing lattice fringes with an interplanar spacing of 0.241 nm. (d) Selected area electron diffraction (SAED) pattern displaying diffraction rings corresponding to the (200) and (111) crystal planes. (e) Energy-dispersive X-ray spectroscopy (EDAX) analysis revealing the elemental composition, indicating the presence of calcium (Ca), oxygen (O), and minor carbon (C).

b. HRTEM image: The high-resolution TEM (HRTEM) image provides detailed insight into the crystal structure of the CaO nanoparticles. A measured lattice fringe spacing of $d = 0.241 \text{ nm}$ is observed, corresponding to the (111) crystal plane of CaO [35–39]. This highlights the crystalline nature of the synthesized material.

c. SAED pattern: The selected area electron diffraction (SAED) pattern displays distinct concentric rings with bright spots, indicating a polycrystalline structure. The rings are indexed to specific planes of CaO, such as (111) and (200), further confirming the crystalline characteristics of the nanoparticles.

d. EDX spectrum: The energy-dispersive X-ray (EDX) spectrum provides the elemental composition of the CaO nanoparticles. It reveals the presence of calcium (Ca), oxygen (O), and a small amount of carbon (C) [40, 41], likely due to surface contamination or residual organic compounds from the synthesis process. The weight percentages of elements are as follows:

Calcium (Ca): 64.92% (weight), 41.36% (atomic).

Oxygen (O): 30.09% (weight), 48.03% (atomic).

Carbon (C): 4.99% (weight), 10.60% (atomic).

4. Conclusion

The synthesis of BNNTs was successful at CVD temperatures of 1100 and 1200°C. The BNNTs exhibited long, thin structures with diameters in the range of approximately 30–80 nm, while the nanostructures appeared as amorphous clumps, forming cauliflower-like shapes of varying sizes. The average crystallite domain size values derived from Raman analysis confirmed that after ion implantation, the phonon lifetime was longer due to the larger domain size, indicating that the BN nanostructures became more crystallized. The increase in the Raman-derived crystallite domain size of the BN nanostructures after boron ion implantation can be attributed to several factors.

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Sequential Electrodeposition of Platinum-Nickel on Boron Doped Diamond Electrode for Ammonia Oxidation Reaction

Abayomi Omoogun, Suzatra Chatterjee, Sharif Uddin Ahmed and Carlos R. Cabrera

Abstract

This study explores the sequential electrodeposition of platinum (Pt) and nickel (Ni) bimetallic structures on boron-doped diamond (BDD) electrodes to enhance the ammonia oxidation reaction (AOR). Leveraging BDD's wide potential window and low background current, we optimize Pt-Ni catalyst morphology and composition to improve AOR kinetics. The research explores the synergistic effects between Pt and Ni to maximize electrocatalytic performance for AOR. It focuses on the role of Ni in modifying Pt's electronic structure and creating beneficial surface defects. Characterization methods such as Raman spectroscopy, scanning electron microscopy, Energy dispersive X-ray (EDX) analysis and electrochemical measurement are employed to correlate structural features with catalytic activity. Future work of this study will investigate the integration of the Pt-Ni catalyst with ureolytic bacteria, such as *Proteus mirabilis*, for comprehensive urea-to-ammonia conversion. This approach addresses challenges in urine purification for space applications, particularly in the International Space Station (ISS), while advancing sustainable energy production through efficient ammonia oxidation to hydrogen. This research also contributes to developing high-performance electrocatalysts for AOR, addressing critical challenges in renewable energy and environmental remediation. The findings have potential implications for wastewater treatment technologies and sustainable energy systems.

Keywords: boron doped diamond electrode, ammonia oxidation, Electrocatalyst, electrodeposition, bio catalysis

1. Introduction

Boron-doped diamond electrodes (BDDEs), first introduced in 1992, have quickly gained prominence as a versatile and effective tool across various fields of electrochemistry [1–10]. In the realm of electroanalysis, BDDEs exhibit a range of

outstanding properties that make them highly valuable. These include an extensive usable potential range, which can reach up to 3.5 V in aqueous solutions depending on factors like doping concentration and surface modification. In addition, BDDEs are characterized by their extremely low background currents, excellent signal-to-noise ratio, and remarkable resistance to surface passivation, making them ideal for precise and reliable electrochemical measurements [11–13]. Boron-doped diamond electrodes (BDDEs) are widely recognized for their biocompatibility, making them valuable in electro-neurochemical applications. Additionally, BDDEs are effective in breaking down organic compounds, such as those found in wastewater, owing to their ability to generate hydroxyl radicals through anodic polarization under neutral or basic conditions. They also exhibit high resistance to degradation and corrosion, particularly on the anodic side [14–23]. Another rapidly advancing application is the use of BDDEs in electrosynthesis, where they function both as an anode and a cathode [24–32].

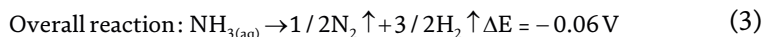
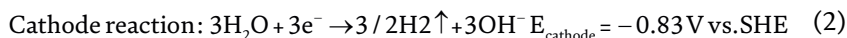
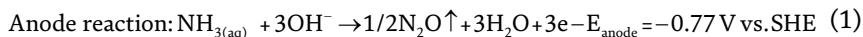
BDD is not a conventional electrode material. Diamond has a very wide band gap semiconductor, containing $\sim 2 \times 10^{23}$ C atoms per cm^3 in the diamond lattice [33]. Pure diamond has an extremely high electrical resistivity, approximately ($1020 \text{ } \Omega \text{ cm}^{-1}$). To reduce this resistivity and enable electrical conductivity, it is necessary to incorporate a suitable dopant into its structure [34]. Incorporating a high concentration of boron to achieve a significant wave function overlap of acceptor atoms allows hole propagation without the need for thermal activation to the valence band [35]. This results in the material exhibiting semi-metallic or ‘metal-like’ properties, albeit with a lower number of charge carriers or density of states (DOS) compared to a traditional metal [36].

The surface termination of boron-doped diamond (BDD) electrodes also plays a pivotal role in shaping their electrochemical behavior, directly impacting their performance in various applications, which is a key factor in shaping their performance and suitability for diverse applications [37]. Common surface terminations for diamond electrodes include hydrogen and oxygen, each imparting distinct electrochemical characteristics to boron-doped diamond (BDD) electrodes. The characteristics of hydrogen-terminated BDD electrodes differ significantly from those of oxygen-terminated ones. These variations influence key aspects such as reactivity, stability, and overall electrochemical performance, making it possible to optimize BDD electrodes for specific applications.

The global concern over water contamination by ammonia nitrogen ($\text{NH}_3\text{-N}$) has grown significantly due to its harmful effects. These include promoting excessive algae growth (eutrophication), producing unpleasant odors, and introducing toxic elements into aquatic environments [38, 39]. Research indicates that $\text{NH}_3\text{-N}$ depletes the oxygen dissolved in wastewater. As it undergoes oxidation, it transforms into either nitrite (NO_2^-) or nitrate (NO_3^-). This process has severe consequences, leading to the degradation of aquatic ecosystems and posing potential risks to human health [40–43].

Recent years have seen a growing interest in the electrochemical ammonia oxidation reaction (AOR). This is because AOR can be seen as a potential pathway toward sustainable and renewable energy sources for direct ammonia fuel cells (DAFCs). Ammonia, with its carbon-free composition and established infrastructure for production, storage, and transportation, emerges as a more promising fuel candidate compared to organic molecules like methanol and ethanol [44]. Ammonia’s considerable energy density makes it a valuable resource that can be efficiently harnessed. This is achieved through the ammonia oxidation reaction (AOR), which converts ammonia

into nitrogen and hydrogen. The process takes place at the anode and involves a three-electron oxidation reaction [45–47].



The process of ammonia electrooxidation at the anode faces significant hurdles due to its sluggish nature and the need for high overpotential. Therefore, the development of efficient electrocatalysts is essential to overcome this challenge [48]. While platinum and platinum-based catalysts demonstrate high activity and low overpotential, their widespread use is hindered by their high cost and susceptibility to poisoning from adsorbed nitrogen species (N_{ads}), which restricts the electrolysis current [49–51]. In response to these limitations, researchers have explored a diverse range of electrode materials. These include other noble metals such as ruthenium, palladium, iridium, and gold, as well as platinum-metal binary alloys. Additionally, metal oxides like RuO_2 , IrO_2 , and PbO_2 have been investigated [52, 53].

Boron-doped diamond (BDD) electrodes have demonstrated remarkable effectiveness in water treatment applications. They have proven successful in water disinfection and have shown the ability to completely break down organic pollutants into carbon dioxide and water, achieving current efficiencies close to 100% [54, 55]. While the treatment of organic compounds is significant, it's equally important to investigate the electrochemical oxidation of inorganic substances in wastewater treatment. Among these inorganic compounds, ammonia deserves particular attention due to its prevalence as a major component in domestic wastewater. Therefore, studying the electrochemical oxidation of ammonia using BDD electrodes is crucial for developing comprehensive wastewater treatment solutions.

Research on using boron-doped diamond (BDD) anodes for the electro-oxidation of ammonia, particularly in the context of converting urine to ammonia, has been relatively sparse. This study presents an innovative approach by employing a boron-doped diamond electrode modified with a bimetallic platinum-nickel catalyst (Pt-Ni/BDD) for ammonia oxidation. The BDD substrate was chosen due to its outstanding corrosion resistance and stability in the challenging conditions associated with ammonia oxidation. The study outlines a quick and simple method for preparing the bimetallic Pt-Ni catalyst through sequential electrodeposition, which was then used for the electrochemical oxidation of ammonia.

2. Experimental part

2.1 Materials and method

BDD purchased from Element Six are polycrystalline chemical vapor deposition (CVD) diamond with a resistivity of 0.02–0.18 $\Omega\text{-cm}$, a thickness of 1 mm and boron concentration $> 1020 \text{ cm}^{-3}$. Sulfuric acid Optima from Fisher was used to prepare 0.5 M H_2SO_4 solution. Potassium tetrachloroplatinate(II) K_2PtCl_4 , 98% from Sigma-Aldrich was used to prepare 1 mM platinum solution. Nickel chloride NiCl_2 , 98%,

Sigma Aldrich was used to prepare 5 mM nickel solution. Potassium perchlorate 99.99% trace metal basis was used to prepare 0.1 M KClO_4 solution used as the supporting electrolyte for electrodeposition. A solution of 0.1 M of ammonia in 0.2 M NaOH was used to study the ammonia oxidation. Nanopore water with 18 M Ω of resistivity was used for the preparation of the solution. Platinum wire and Ag/AgCl electrodes were used as counter and reference, respectively. All solutions were purged using ultra high pure argon gas.

2.2 Electrochemical measurement

The CHI 6044 E electrochemical workstation was utilized for all electrochemical experiments, which were conducted at room temperature. In this setup, a three-electrode system was employed where the bare BDD and Pt-Ni-BDD electrodes served as the working electrode, the Ag/AgCl electrode was used as the reference electrode, and a platinum wire electrode functioned as the counter electrode. Cyclic voltammetry (CV) was performed for cleaning the BDD electrode in 0.5 M H_2SO_4 and for its characterization in 0.1 M KClO_4 , with scan rates of 50 mV/s and 20 mV/s, respectively. For ammonia oxidation, CV was conducted at a scan rate of 1 mV/s.

2.3 Boron-doped diamond surface characterization

Boron-doped diamond electrodes from Element Six were activated by aqua regia and ten cycles of cyclic voltammetry in a 0.5 M H_2SO_4 solution. Boron-doped diamond electrodes were characterized by cyclic voltammetry in a potential range of -0.5 V and 0.6 V vs. Ag/AgCl at a scan rate of 10 mV/s in a 0.5 M H_2SO_4 solution and in 0.1 M KClO_4 in a potential range of -0.3 V and 0.8 V. Scanning electron microscopy (SEM), and Raman spectroscopy, were used to characterize the surface of boron-doped diamond electrodes. BDD electrodes were characterized by SEM using a high-resolution field emission JEOL JSM-7500 F. The accelerating voltage was 15 kV and secondary electron image (SEI) as imaging mode. The Raman spectra was obtained with mIRage IR/Raman module, spectrometer, SN KY-04529.

2.4 Preparation of the Pt-Ni/BDD

Before modifying the electrode, a cathodic reduction treatment was conducted as a pre-treatment on the boron-doped diamond electrode. This involved immersing the electrode in 0.5 M H_2SO_4 at -3 V for 5 minutes. For the actual modification, sequential electrodeposition of NiCl_2 and K_2PtCl_4 solutions was performed on the pre-treated BDD electrode. Initially, 5 mL of 5 mM NiCl_2 and 5 mL of 0.1 M KClO_4 was added to a three-compartment electrochemical cell that contained the working, reference, and counter electrodes. Electrodeposition was then carried out at a potential of -0.95 V for 120 seconds. The non-deposited particles were cleaned by rinsing with ultrapure water at 18.20 m Ω , resulting in the formation of a Ni/BDD electrode. Subsequently, 5 mL of 1 mM K_2PtCl_4 and 5 mL of 0.1 M KClO_2 was electrodeposited onto the cleaned and dried Ni/BDD electrode at a potential of -0.3 V for 120 seconds. The modified electrodes was cleaned with ultrapure water, leading to the final preparation of a bimetallic Pt-Ni catalyst electrode (Pt/Ni/BDD).

3. Results and discussion

3.1 Electrochemical cleaning and characterization of boron doped diamond electrode

The CV curve from **Figure 1a** shows the current density (j) as a function of applied potential, with values ranging from -0.5 V to $+0.6$ V vs. Ag/AgCl for bare BDD electrode in 0.5 M H_2SO_4 . The current density remains low and stable over much of the range, which is typical for BDD in acidic solutions. From approximately -0.4 V to $+0.6$ V, the CV curve shows a stable and flat current response, with minimal faradaic currents, suggesting that this potential range lies within the double-layer region of the BDD electrode which is primarily due to capacitive charging/discharging rather than any electrochemical redox reactions, which is consistent with the inert properties of BDD. At potentials below -0.4 V, the current density begins to slightly decrease in the negative direction, indicating the onset of the hydrogen evolution reaction (HER). While the current density is relatively small, this is likely due to the high overpotential for HER on BDD. Also, CV of bare BDD electrode in 0.1 M KClO_4 which was our electrolyte for electrodeposition shows significant capacitive behavior, suggesting a high double-layer capacitance. In addition, minimal background current was observed, emphasizing the electrode's electrochemical inertness and suitability for sensitive measurements.

3.2 Raman characterization

The Raman spectra for bare poly crystalline boron doped diamond electrode sheets is shown in **Figure 2**. Raman spectrum for BDD at 1330 cm^{-1} , characteristics of carbon sp^3 and the characteristic peak of the diamond structure itself, corresponding to the zone-center optical phonon of diamond [56]. The Peak at 594 cm^{-1} is often associated with the presence of sp^2 carbon impurities or defects in the diamond lattice [57]. The peak at 1230 cm^{-1} can be attributed to the vibrational modes of the boron-doped regions within the diamond [58, 59].

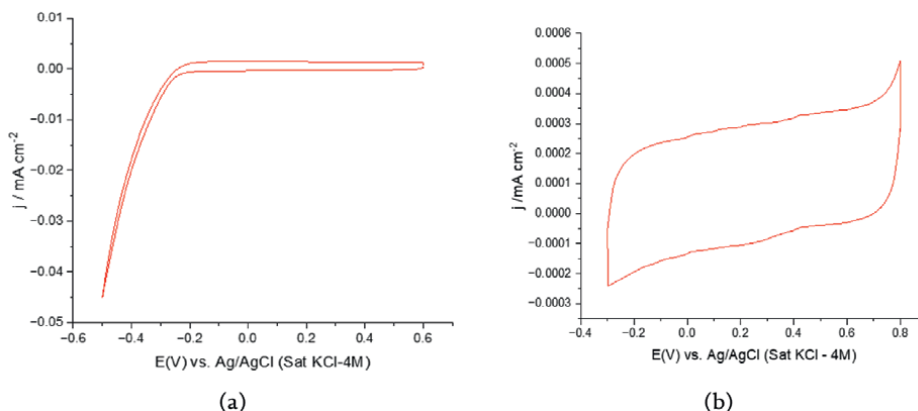


Figure 1.
(a) Cyclic voltammetry of boron doped diamond electrode in 0.5 M H_2SO_4 ; (b) CVs of BDD electrode in 0.1 M KClO_4 .

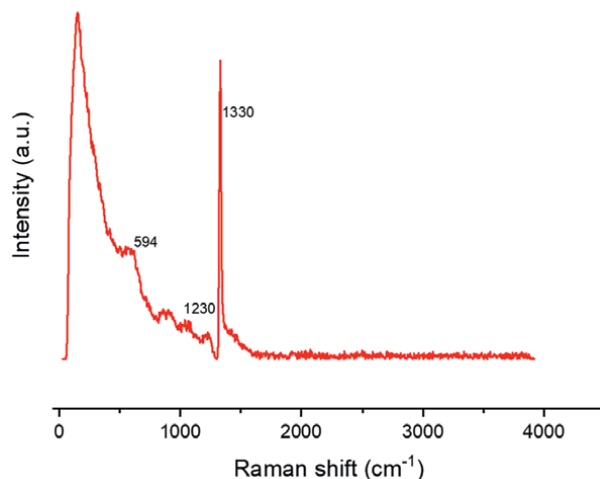


Figure 2.
Raman spectra for bare boron doped diamond electrode.

3.3 Scanning electron microscopy

SEM image for poly crystalline boron doped diamond electrode is shown in **Figure 3**. The BDD electrode shows a polycrystalline diamond sheet with a mix of diamond grains of several sizes characterized by imperfections and borders only on smaller diamond particles.

3.4 Characterization of Pt-Ni BDD electrodes and ammonia oxidation

3.4.1 Scanning electron microscopy

Figure 4 shows SEM images for platinum-Nickel nanoparticles electrodeposited through chronoamperometry on boron doped diamond electrode. Metal particles were deposited on the active surface area of the boron doped diamond electrode uniformly,

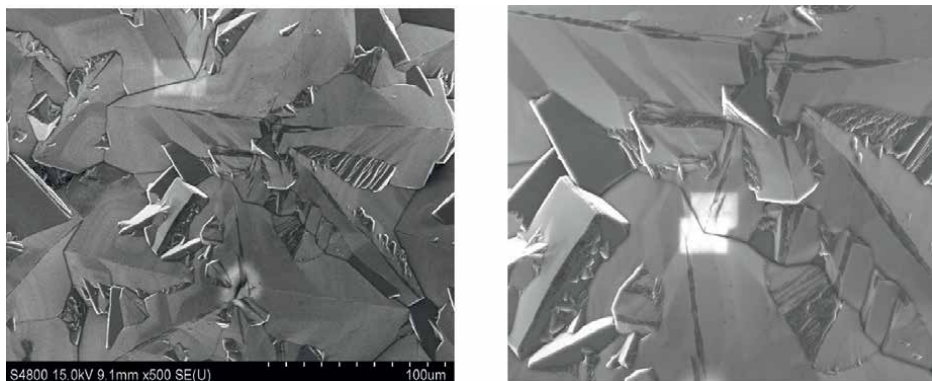


Figure 3.
Scanning electron microscopy images of polycrystalline boron doped diamond electrode. Magnification $\times 500$ and scale bar = 100 μm .

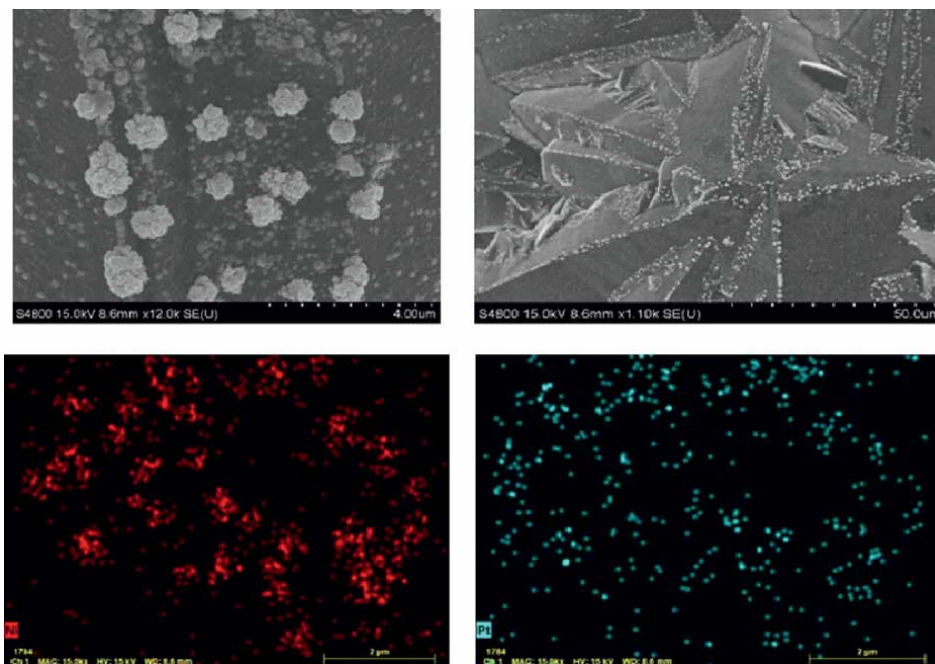


Figure 4.
Scanning electron microscopy and X-ray fluorescence images of Platinum-Nickel catalyst on boron doped diamond electrode at different magnification.

though some agglomeration of particles was observed. It is worth noting that the concentration of platinum to nickel is 1:5 which shows why there is more agglomeration of nickel particles when compared to platinum as evident from the EDX analysis and this shows that the distribution of Pt and Ni are not evenly homogenous.

3.4.2 Raman spectroscopy

The Raman spectrum of the Pt-Ni/BDD film shows three distinctive bands at 1231, 1332, and 1545 cm^{-1} in **Figure 5**. The bands at 1231 cm^{-1} are ascribed to the locally disordered structures induced by the heavily boron-doped level [60], which confirms that boron atoms are incorporated into the diamond crystal lattice. The sharp Raman resonance band at around 1332 cm^{-1} is a typical characteristic diamond band [61] and the broad band at about 1545 cm^{-1} (G band) is ascribed to the amorphous sp^2 -carbon contained in the BDD layer which demonstrates the presence of poly crystalline graphite on the Pt-Ni on BDD surface [62].

3.4.3 Ammonia oxidation

Figure 6 shows the voltammograms for ammonia oxidation on platinum-Nickel nanoparticles BDD electrode. The first peak is observed at a specific potential at -0.24 V, and this peak indicates the onset potential of ammonia (NH_3) oxidation. Also, the oxidation of ammonia showed good current density of around $8 \text{ mA cm}^{-2}/\text{mg}$. The high peak of this current density indicates the Platinum-Nickel catalyst on BDD electrode is an active and a good catalyst for ammonia oxidation.

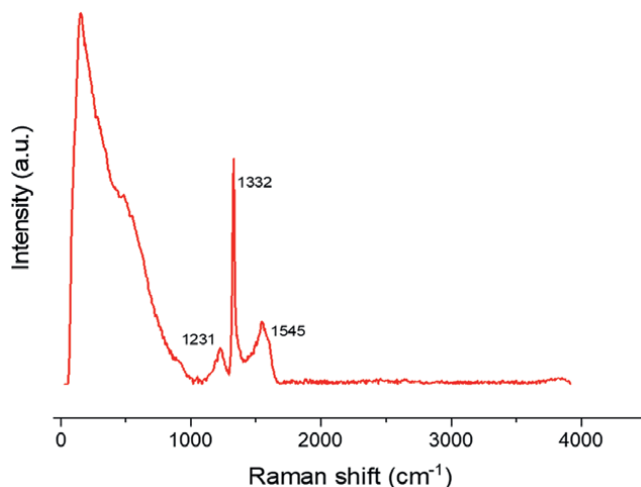


Figure 5.
Raman spectra for Pt-Ni modified boron doped diamond electrode.

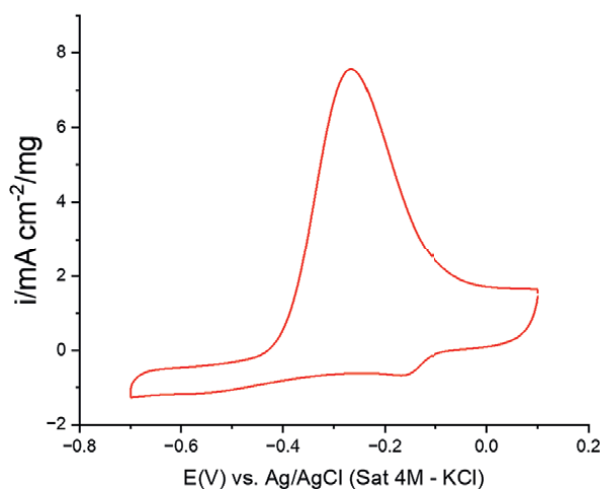


Figure 6.
Cyclic voltammograms for ammonia oxidation on Pt-Ni electrodeposited through chronoamperometry on BDD in 0.1 M NH_3 in a 0.2 M NaOH solution at 1 mV/s.

4. Conclusion

Studies have shown that $\text{Ni}/\text{Ni}(\text{OH})_2$ is an effective electrode material for ammonia electrochemical oxidation. The choice of nickel in this research is due to its affordability, making it a promising candidate for wastewater treatment applications. The addition of platinum serves to boost the electrode's electrocatalytic performance. In this study, a Pt-Ni/BDD catalyst using a sequential electrodeposition method was developed. The interaction between platinum and nickel components, along with their optimized structure, greatly improved the catalyst's electrochemical properties, enhancing its ability to oxidize ammonia. The BDD electrode proved to be an excellent support, allowing for even distribution of the metal catalysts while maintaining

durability and electrochemical efficiency. Also, future study of this work will involve immobilizing the bimetallic Pt-Ni catalyst with bacteria which can be used for urine to ammonia conversion.

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Conflict of interest

The authors declare no conflict of interest.

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Sustainable Water Treatment with Boron-Doped Diamond Electrodes: Advancing a Green Economy

Nejmeddine Rabaaoui and Houcine Naili

Abstract

This chapter delves into the transformative potential of boron-doped diamond (BDD) electrodes in revolutionizing advanced oxidation processes (AOP) for sustainable industrial water treatment. Leveraging the remarkable electrochemical properties of BDD, industries can effectively apply AOP technologies to eliminate stubborn pollutants and persistent contaminants, significantly reducing environmental impact. As these laboratory-driven innovations transition to full-scale industrial applications, BDD electrodes hold the potential to spark a true revolution in wastewater treatment, moving beyond experimental promise to redefine industry standards. This eco-innovative technology not only enhances treatment efficiency but also aligns seamlessly with global efforts toward a green economy, promoting cleaner production practices and a reduced ecological footprint. Through this sustainable approach, BDD electrodes offer an unparalleled solution for industries seeking both environmental responsibility and regulatory compliance in water management.

Keywords: boron-doped diamond (BDD) electrodes, advanced oxidation processes (AOPs), sustainable water treatment, industrial wastewater management, green economy

1. Introduction

In the face of growing pressure on natural resources driven by population growth and intensified economic activities, adopting sustainable water management practices has become a global priority. This challenge lies at the heart of Sustainable Development Goal (SDG) 6, which aims to ensure universal access to clean water and sustainably managed sanitation services [1]. According to United Nations estimates, global demand for freshwater could exceed available resources by 40% by 2030, directly affecting 1.6 billion people and depriving them of safe access to drinking water [2]. Currently, nearly 4 billion people live in water-scarce regions, with one in four cities experiencing severe water crises [3], underscoring the urgency of innovative and sustainable solutions to tackle this challenge.

However, this crisis extends beyond simple scarcity; it is compounded by complex factors such as water resource pollution. Population growth intensifies water demand

to meet food production, energy, and urbanization needs. At the same time, the mismanagement of wastewater is a significant issue. Reports from the World Bank and the World Health Organization (WHO) reveal that approximately 80% of industrial and domestic wastewater is discharged untreated, contaminating groundwater and surface water with toxic chemicals [4, 5]. These pollutants, including arsenic, fluoride, and lead, present a dual challenge: they harm human health and weaken natural ecosystems, complicating the achievement of SDG 15, which focuses on protecting and sustainably managing terrestrial ecosystems [6].

The health consequences of this crisis are alarming. According to the WHO, water contamination is responsible for 1 million annual deaths, often caused by preventable diseases like diarrhea, which claims the lives of 395,000 children under five each year [7]. In regions with limited access to clean water, hygiene practices such as handwashing become secondary, facilitating the spread of diseases like cholera and typhoid [8]. Furthermore, water pollution contributes to the persistence of chronic diseases such as schistosomiasis, which affected over 251 million people in 2021 [9].

In this context, innovations in water treatment emerge as crucial solutions, extending far beyond technical advancements. They represent a genuine investment in a sustainable future, where the protection of natural resources, the promotion of public health, and the preservation of ecosystems harmoniously intertwine. Among these innovations, boron-doped diamond (BDD) electrodes stand out as a revolutionary technology. With their unique electrochemical properties, BDD electrodes offer an efficient and sustainable solution for the complete degradation of organic and inorganic pollutants, making them a preferred choice for industrial water treatment [8, 10–12].

The adoption of BDD electrodes represents a significant milestone in the industrial sector. Transitioning from laboratory research to large-scale applications, this technology illustrates its potential to redefine industry standards while meeting the demands of sustainable development and a green economy [13, 14]. By significantly reducing environmental footprints, BDD electrodes contribute to more responsible water resource management and open new pathways for ecological transitions [15–17].

This chapter delves into the revolutionary potential of boron-doped diamond electrodes, exploring their exceptional technical characteristics and their impact on sustainable industrial practices. It highlights their central role in shaping an integrated approach to industrial water treatment, supporting a forward-looking vision centered on sustainability, innovation, and environmental stewardship.

2. Technical characteristics of BDD electrodes

2.1 Diamond: Between myth and technology

For millennia, diamonds have captivated humanity, inspiring awe and desire across eras. The earliest recorded use of diamonds dates back approximately 3000 years in India, where they were extracted from rivers and revered as sacred stones imbued with mystical properties. Until the seventeenth century, India dominated the global diamond supply, before Brazil and, later, South Africa in the late nineteenth century, emerged as major production centers [18].

Among the most famous diamonds in history, the Koh-i-Noor stands out as a symbol of cultural and political significance. Mined in India, this legendary gem, now

part of the British Crown Jewels, reflects the diamond's enduring historical impact. Similarly, the Hope Diamond, with its deep and enigmatic blue hue, originates from seventeenth-century India. Surrounded by fascinating legends, this iconic gem is now housed at the Smithsonian National Museum of Natural History in Washington, underscoring its status as a global treasure [19].

In antiquity and the Middle Ages, diamonds were reserved for the elite, symbolizing power and prestige. Regarded as protective talismans, they were shrouded in mysticism. It was not until the Renaissance that diamonds became widely used in jewelry, emerging as symbols of wealth and eternal love.

Today, diamonds are categorized into three main types, tailored to specific and diverse applications. Natural diamonds, formed over millions of years, are highly valued for their rarity and purity. Their varying hues—ranging from clear to vibrant pinks, blues, yellows, and even blacks—result from trace elements such as nitrogen or boron, or from structural imperfections. These unique attributes endow them with both esthetic and chemical significance, paving the way for specialized uses (**Figure 1**) [15].

Laboratory-grown diamonds, created through advanced techniques like chemical vapor deposition (CVD), replicate the characteristics of natural diamonds while offering precise control over properties such as purity and color [16, 17].

Lastly, industrial diamonds, predominantly synthetic, are indispensable for technological applications requiring exceptional durability and chemical resistance. These advancements highlight how science has transformed diamonds from precious gemstones into cutting-edge industrial materials.

This evolution—from mystical stones to modern technological assets—reflects human ingenuity and the profound influence of scientific progress. With promising applications such as boron-doped diamond (BDD) electrodes, diamonds have become pivotal to industrial innovation, addressing contemporary technological and environmental challenges.

2.2 Structure and physicochemical properties

Diamond, a fascinating and unique material, is distinguished by its remarkable physicochemical properties, positioning it as a top-tier functional material for advanced technological applications. These characteristics arise directly from its crystalline structure and chemical composition, making it ideal for innovative electrochemical uses.

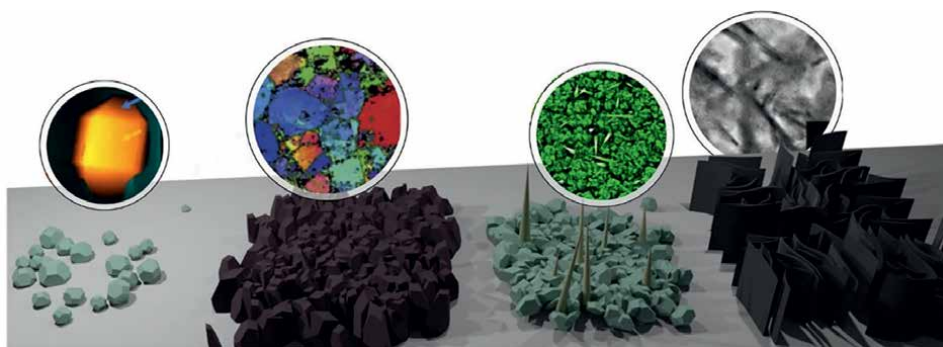


Figure 1.
Structural evolution of diamond and carbon materials.

2.2.1 Crystalline structure and chemical composition

The structure of diamond is a true masterpiece of molecular engineering, distinguished by a three-dimensional network of sp^3 -hybridized carbon atoms, each covalently bonded to four neighbors with remarkable precision at a bond length of 1.54 Å. This flawless tetrahedral geometry extends infinitely, leveraging the unique ability of carbon to form highly robust covalent networks. Diamond's crystal adopts a face-centered cubic (FCC) configuration (space group: $Fd\bar{3}m$, No. 227) with a lattice parameter of 3.54 Å [20]. Within this precise architecture, carbon atoms occupy all lattice points as well as alternating positions in the tetrahedral voids, maintaining an ideal spacing equivalent to half the face diagonal $\langle 110 \rangle$ in the unit cell.

This structural organization endows diamond with exceptional mechanical and physical properties, making it one of the most remarkable natural materials. Its unrivaled hardness, reaching 10 on the Mohs scale [21], allows it to resist abrasion and wear even in extreme conditions. Additionally, its high atomic density, approximately 3.52 g/cm³, enhances its stability and durability, establishing diamond as an indispensable material for applications demanding both performance and reliability. With this unparalleled combination of structural perfection and unique properties, diamond stands as a material of unmatched significance in both scientific and practical domains [22].

2.2.2 Thermal and electronic properties

Diamond also stands out for its exceptional thermal and electronic properties, ranking among the most efficient materials in these domains. With an extremely high thermal conductivity of approximately 2200 W/m K at room temperature [23], diamond is capable of efficiently dissipating heat, making it indispensable in applications where thermal management is critical. Furthermore, in its pure state, diamond is an electrical insulator due to its wide bandgap of approximately 5.5 eV [24]. However, when doped with boron atoms, its electronic structure is modified, transforming it into a semiconductor [25]. This transformation opens the door to a multitude of advanced electrochemical applications, where the optimal combination of its thermal and electronic properties can be leveraged.

2.2.3 Chemical resistance and stability

Diamond is renowned for its remarkable chemical inertness and its ability to effectively withstand highly aggressive environments. Its exceptional chemical stability enables it to remain inert in various media, including highly acidic, alkaline, or oxidative solutions, making it a material of choice for demanding industrial applications. Additionally, diamond's naturally hydrophobic surface limits the adsorption of organic contaminants, further optimizing its efficiency in advanced electrochemical processes [26]. These properties, combined with its intrinsic robustness, establish diamond as an essential material for environments where performance and durability are paramount.

2.2.4 Fabrication and doping of boron-doped diamond (BDD) electrodes

2.2.4.1 Chemical vapor deposition (CVD)

The chemical vapor deposition (CVD) method is the most widely used technique for producing synthetic diamond films for boron-doped diamond (BDD) electrodes.

First developed in the 1980s by John Angus and his team at Case Western Reserve University [27], this technology revolutionized material science by transforming natural diamond, inherently an insulator, into a semiconductor perfectly suited for electrochemical applications. Over the decades, CVD processes have undergone continuous improvement, enabling greater control over the physical and chemical properties of diamond films [28, 29].

The fabrication process involves a carefully controlled sequence starting with substrate preparation, where the surface is meticulously cleaned and textured to optimize diamond film adhesion. Next, gas activation occurs through a high-temperature plasma, often generated by microwaves or hot filaments, which decomposes methane (CH_4) molecules, releasing carbon radicals ready to deposit on the substrate. Finally, the growth of diamond films is regulated by adjusting critical parameters such as temperature, pressure, and gas composition, ensuring an optimal crystalline structure and properties tailored to industrial applications (**Figure 2**). This precise control allows the production of high-quality boron-doped diamond films capable of meeting the demands of modern electrochemical processes, including wastewater treatment and the degradation of organic pollutants.

2.2.4.2 Boron doping and metal-insulator transition

Boron doping is a crucial step in transforming diamond, a natural insulator, into a semiconductor ideally suited for advanced electrochemical applications. This process alters the diamond's electronic properties by reducing the bandgap to approximately 1 eV while introducing charge carriers in the form of holes, significantly enhancing its electrical conductivity (**Figure 3**) [30]. Despite these modifications, boron-doped diamonds retain their exceptional chemical inertness,

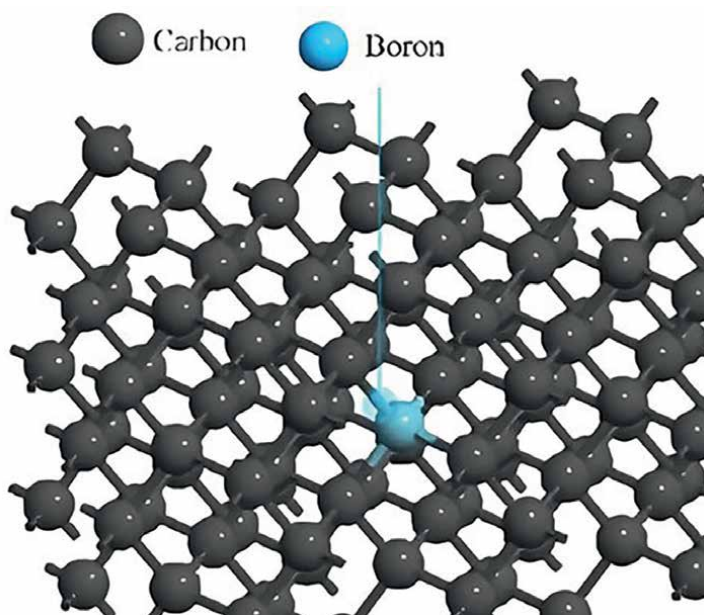


Figure 2.
Boron-doped diamond (BDD) structure.

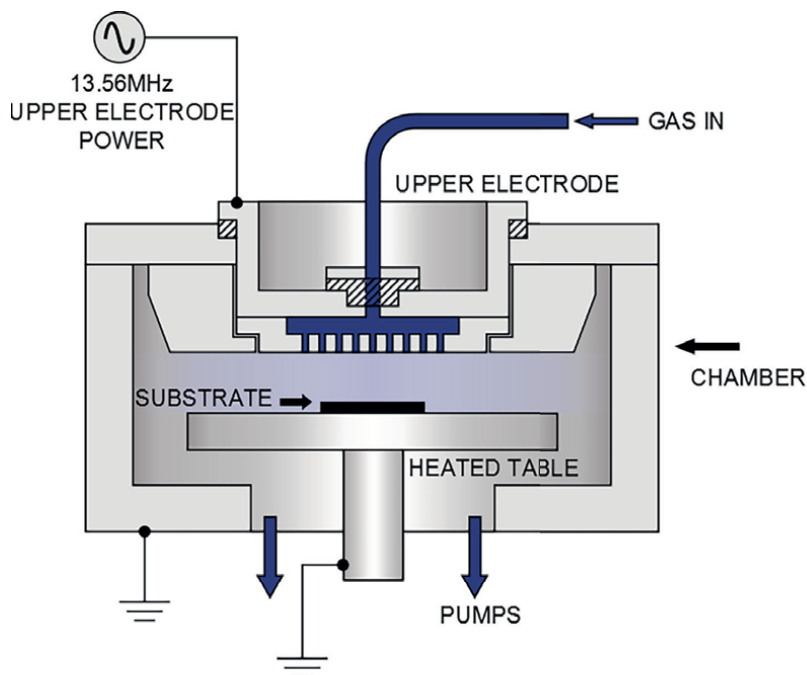


Figure 3.
Chemical vapor deposition (CVD).

allowing them to maintain stability in corrosive and aggressive environments. Additionally, the doped surface provides an expanded potential window of approximately 3 V, ideal for advanced oxidation processes such as the degradation of organic pollutants.

2.3 Durability and longevity

Boron-doped diamond (BDD) electrodes are renowned for their exceptional durability and their ability to maintain optimal performance over extended periods of use. This remarkable longevity results from the unique combination of the diamond's physical, chemical, and electrochemical properties. Thanks to their chemical inertness, BDD electrodes resist attacks from corrosive media, whether acidic, alkaline, or oxidative, enabling them to remain effective even in the most demanding environments.

Furthermore, their hydrophobic surface minimizes the adsorption of organic contaminants, reducing the risks of fouling or passivation—two phenomena that often compromise the performance of conventional materials. The mechanical stability of BDD electrodes, combined with their resistance to wear and abrasion, ensures long-term reliability under intensive operating conditions [31].

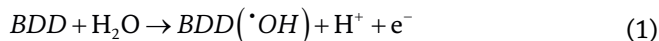
This exceptional durability translates into significant reductions in maintenance and replacement costs for industrial applications while enhancing environmental sustainability through decreased waste and operational interruptions. In summary, BDD electrodes represent a durable and economical solution to modern industrial challenges, combining robustness and long-term efficiency.

3. Principle of operation and applications

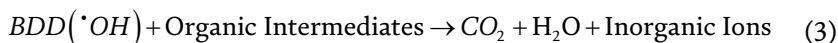
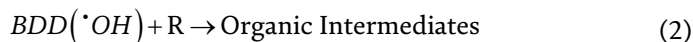
3.1 Kinetic performance model of BDD anodes

Boron-doped diamond (BDD) electrodes play a critical role in the mineralization of organic compounds due to their exceptional ability to generate hydroxyl radicals ($\cdot\text{OH}$), reactive oxygen species (ROS) that are essential for the degradation of complex pollutants and their conversion into harmless end products such as carbon dioxide (CO_2) and water (H_2O) [32–34]. The kinetic model describes the interactions between hydroxyl radicals and pollutants, as well as the parameters influencing their efficiency.

The process begins with the generation of hydroxyl radicals on the BDD electrode surface during water electrolysis, represented by the following reaction [35]:



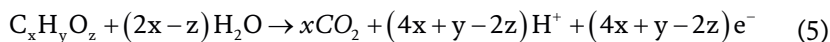
Thanks to their high redox potential (~ 2.8 V vs. SHE), these radicals oxidize nearly all recalcitrant compounds into CO_2 , H_2O , and inorganic ions through the following reactions [36]:



This model relies on three fundamental assumptions: first, the adsorption of organic compounds on the BDD electrode surface is negligible, simplifying the analysis of electrode-solution interactions. Second, all organic compounds in the solution are assumed to have identical diffusion coefficients, eliminating disparities related to individual pollutant properties. Finally, the model assumes that the mineralization rate is entirely controlled by the mass transport of organic compounds to the anode surface, regardless of their chemical nature. These assumptions provide a uniform and predictive description of BDD anode behavior in removing various organic pollutants, highlighting their exceptional efficiency in electrochemical degradation processes.

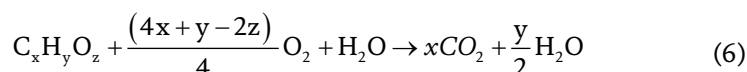
Under these conditions, the limiting current density (i_{lim}) for the electrochemical mineralization of an organic compound is given by [37]:

$$i_{\text{lim}} = nFk_m C_{\text{org}} \quad (4)$$



$$\text{For } n = (4x + y - 2z), i_{\text{lim}} = (4x + y - 2z)nFk_m C_{\text{org}}$$

For organic compounds of general formula $\text{C}_x\text{H}_y\text{O}_z$, their oxidation is described as:



The chemical oxygen demand (COD) is related to the organic concentration by the formula:

$$DCO = C_{org} \left(\frac{(4x + y - 2z)}{4} \right) \quad (7)$$

$$i_{lim}(t) = 4Fk_m DCO(t) \quad (8)$$

and

$$i_{lim,0} = 4Fk_m DCO_0 \quad (9)$$

i_{lim} : limiting current density (A/m^2), n : number of electrons exchanged in the mineralization reaction, F : Faraday constant (96,487 C/mol), k_m : mass transport coefficient (m/s), C_{org} : organic compound concentration in solution (mM), $COD(t)$: chemical oxygen demand at time t , COD_0 : initial chemical oxygen demand.

These equations illustrate that the current density is directly proportional to the concentration of organic compounds in the solution, emphasizing the importance of concentration as a parameter in the mineralization process. The mass transport coefficient (k_m) plays a crucial role, influenced by hydrodynamic conditions such as flow rate and turbulence around the electrode, which promote effective transport of organics to the anode surface. Additionally, the parameter n , determined by the stoichiometry of the mineralization reactions, reflects the number of electrons exchanged during the process, enhancing the model's predictive capability.

These relationships demonstrate that the limiting current density depends directly on the COD, reflecting the concentration of organic compounds in the solution. Higher initial organic concentrations (COD_0) lead to higher $i_{lim,0}$, indicating more intense electrochemical activity required for mineralization. These equations also distinguish two electrolysis regimes based on the parameter $\alpha = \frac{i}{i_{lim,0}}$.

i. Current-controlled regime ($\alpha < 1$)

In this regime, the applied current density remains below the limiting current density ($i < i_{lim}$), ensuring that all reactive species generated on the anode surface are fully utilized for the mineralization of organic compounds. Current efficiency reaches 100%, with minimal secondary reactions such as oxygen evolution. This regime optimizes energy consumption, providing a constant mineralization rate directly proportional to the applied current density. However, its performance is limited by the available current, potentially slowing the process for high pollutant concentrations.

ii. Mass transport-controlled regime ($\alpha > 1$)

Here, the applied current exceeds the limiting current density ($i > i_{lim}$), meaning that the diffusion rate of organic compounds to the electrode surface cannot match the current intensity. This leads to secondary reactions, such as oxygen evolution, reducing the instantaneous current efficiency (ICE). Although this regime allows faster mineralization rates, it increases energy consumption and reduces overall efficiency due to losses from undesirable reactions.

- iii. The transition between these two regimes occurs at $\alpha = 1$, where the applied current matches the limiting current density ($i = i_{\text{lim}}$). At this critical point, the process balances efficiency and treatment speed but begins to show limitations due to mass transport. Hydrodynamic conditions play a pivotal role here: turbulent flow, by increasing the mass transport coefficient (k_m), can alleviate these limitations, whereas laminar flow exacerbates them [38].

By selecting the appropriate operating regime and optimizing hydrodynamic parameters, the system can be tailored to specific needs, whether for maximum energy efficiency or accelerated treatment rates. This operational flexibility underscores the value of BDD anodes for modern electrochemical applications that demand both high performance and optimal durability.

3.2 Applications in water treatment

Thanks to its unique properties, boron-doped diamond (BDD) anodes produce a significant amount of hydroxyl radicals ($\cdot\text{OH}$) during electrolysis in the water discharge region. These radicals are weakly adsorbed on the electrode surface, granting them high reactivity for the oxidation of organic compounds. This characteristic makes BDD anodes highly effective for wastewater treatment. Numerous studies have demonstrated that at high potential, electrolysis using BDD electrodes achieves complete mineralization of various organic pollutants through reactions with electrochemically generated $\cdot\text{OH}$ radicals [39–43].

Oturan et al. extensively investigated the behavior of Si/BDD anodes in acidic solutions for the oxidation of a broad range of pollutants. Their research revealed that complete mineralization was achieved under all experimental conditions studied. They concluded that oxidation is primarily controlled by the diffusion of pollutants toward the electrode surface, where hydroxyl radicals are generated. Current efficiency was shown to be enhanced by a high mass transport coefficient, elevated organic concentrations, and low current densities [44, 45]. Under optimized conditions, without diffusion limitations, the current efficiency of BDD anodes can approach nearly 100%.

Further studies by Nejmeddine et al. focused on the electrochemical oxidation of various phenolic compounds (methyl phenols, chlorophenols, nitrophenols, and nitrobenzene) and carboxylic acids on BDD anodes [46–48]. They reported that organic compounds were fully mineralized regardless of the characteristics of the wastewater (initial concentration, pH, and supporting electrolyte) or the operational conditions (temperature and current density). This adaptability highlights the versatility of BDD electrodes in handling diverse wastewater matrices.

Comparative research has highlighted the outstanding performance of boron-doped diamond (BDD) electrodes in the electrochemical degradation of organic pollutants when compared to other materials such as SnO_2 , PbO_2 , and RuO_2 . Yingying et al. [49] demonstrated that BDD anodes achieve remarkable removal efficiency, as illustrated in **Table 1**. For instance, BDD electrodes achieved up to 100% COD removal for various pollutants, including washing machine effluents within 240 minutes, Congo Red dye in just 5 minutes, and Eriochrome Black T within 360 minutes. These results surpass those of conventional anodes like Pt, IrO_2 , and SnO_2 , which often require longer processing times or yield lower removal rates.

Anodes	Pollutants	Electrolytes	Current density (mA cm ⁻²)	COD removal rate (%)	TOC removal rate (%)
SnO ₂ /BDD	Clofibric acid	0.05 M Na ₂ SO ₄	30	90 (240 minutes)	95
Yb—PbO ₂	Acetamiprid	0.05 M Na ₂ SO ₄	10	74.57 (120 minutes)	99
Si/BDD	Acid violet 7 dye	0.05 M Na ₂ SO ₄	15	87	97
Nb/BDD	Acid violet 7 dye	0.05 M Na ₂ SO ₄	15	85	95
BDD-NCD	Dye	0.1 M Na ₂ SO ₄	30	85	90
Pt	Carbaryl	0.05 M Na ₂ SO ₄	300	66	77
Ti/Pt	Salicylic acid	0.05 M Na ₂ SO ₄	150	65	87
Ti/RuO ₂	Ofloxacin	2 g L ⁻¹ NaCl	75	63	80
Ti/RuO ₂	Highly acidic wastewater	0.1 M Na ₂ SO ₄	75	60	80
0.8RuO ₂ 0.2/TiO ₂ / Ti	1,4-Dioxane	0.1 M Na ₂ SO ₄	60	60	90
Ti/IrO ₂ / SnO ₂	Chlorpyrifos	0.05 M Na ₂ SO ₄	20	50	56
Ti/IrO ₂	Norfloxacine	0.05 M NaCl	6.53	79	100
RuO ₂ - TiO ₂ / NaO-GC	Ceftriaxone sodium	0.1 M Na ₂ SO ₄	20	65	77
GNS-PbO ₂	2-Chlorophenol	0.05 M Na ₂ SO ₄	50	55	62
PbO ₂ /SnO ₂	3-CP	0.5 M Na ₂ SO ₄	20	53	78
Ti/Pt/ PbO ₂	Humic acid leachate	0.05 M Na ₂ SO ₄	30	50	77
MI-meso SnO ₂	2,4-Dichlorophenoxyacetic acid	0.1 M Na ₂ SO ₄	6.34	60	93
SnO ₂ -TiO ₂ / NaOG	Ceftriaxone sodium	0.1 M Na ₂ SO ₄	60	70	98
Ti/IrO ₂ / SnO ₂	Chlorpyrifos	0.05 M Na ₂ SO ₄	20	73	60
BDD	Atenolol	0.014 M Na ₂ SO ₄	33	92	99
BDD	Eriochrome Black T	0.05 M Na ₂ SO ₄	50	93	99
BDD	Congo red	0.05 M NaCl	50	90	99

Table 1.

Performance comparison of anodic materials for organic pollutant degradation: Highlighting the superior efficiency of BDD electrodes.

In addition to their superior efficiency, BDD electrodes exhibit exceptional versatility, effectively treating a broad spectrum of contaminants such as pesticides, dyes, pharmaceuticals, and industrial effluents. This adaptability makes them a highly promising choice for advanced water treatment applications. Their unparalleled performance is attributed to their ability to efficiently generate hydroxyl radicals, which facilitate rapid oxidation and extensive mineralization under various operating conditions. These characteristics underscore the transformative potential of BDD electrodes in addressing complex water pollution challenges and advancing sustainable wastewater treatment solutions.

3.3 Applications in advanced electrochemical systems

Advanced electrochemical systems (AES) represent a major breakthrough in addressing modern environmental challenges, particularly in the treatment of complex wastewater and disinfection processes. Boron-doped diamond (BDD) electrodes play a pivotal role in these technologies due to their unique electrochemical properties, such as a wide potential window, exceptional chemical inertness, and the ability to generate highly reactive hydroxyl radicals ($\cdot\text{OH}$). These attributes allow BDD electrodes to outperform traditional materials like SnO_2 , PbO_2 , and IrO_2 electrodes, especially in integrated processes like electro-Fenton and photoelectro-Fenton.

The electro-Fenton process, combined with BDD electrodes, generates hydrogen peroxide (H_2O_2) in situ, which reacts with Fe^{2+} ions to produce hydroxyl radicals. These radicals efficiently oxidize recalcitrant pollutants such as pesticides [39, 49], dyes, and pharmaceutical compounds [50]. A recent study by Zhang et al. in 2024 demonstrated that an electro-Fenton system using BDD electrodes achieved a 95% reduction in organic load within just 45 minutes, compared to 70% with conventional SnO_2 electrodes [51]. The photoelectro-Fenton process, incorporating a light source to regenerate Fe^{2+} ions, further enhances efficiency, achieving near-complete mineralization in a shorter timeframe.

In addition, BDD electrodes are particularly effective for microbial disinfection in water. Their ability to generate reactive oxygen species (ROS) facilitates the direct oxidation of pathogenic microorganisms, a critical feature for treating hospital wastewater and industrial effluents. A comparative analysis conducted by Smith et al. in 2022 showed that BDD electrodes eliminated 99% of *Escherichia coli* bacteria in less than 20 minutes, a result significantly superior to PbO_2 electrodes under similar conditions [52].

The applications of BDD electrodes extend beyond organic pollutant treatment. They are also promising for the removal of heavy metals such as mercury, cadmium, and lead. These metals are converted into insoluble compounds that can be easily removed, providing a significant advantage in treating water contaminated by mining and chemical industries. Furthermore, BDD electrodes maintain stable efficiency in chloride-rich environments, a condition that often hampers the performance of metallic electrodes.

Lastly, recent advancements have enabled the deployment of BDD electrodes in a variety of industrial applications, such as effluents from textile and pharmaceutical industries. Their chemical robustness and energy efficiency ensure compliance with the strictest environmental standards. Systems incorporating BDD electrodes significantly reduce operational costs while minimizing environmental impact, establishing BDD as a cornerstone of modern electrochemical technologies.

3.4 Key advantages of applications

Boron-doped diamond (BDD) electrodes offer unparalleled advantages, establishing themselves as a leading technology in electrochemical applications. These electrodes excel in their ability to achieve complete mineralization of organic pollutants through the generation of highly reactive hydroxyl radicals ($\cdot\text{OH}$). Unlike other materials, such as SnO_2 or PbO_2 , BDD electrodes facilitate non-selective oxidation of recalcitrant compounds, converting them into CO_2 , H_2O , and inorganic ions without generating toxic by-products. Their wide potential window ($\sim 3\text{ V}$) gives them a decisive advantage, enabling optimal efficiency in complex environments, including those containing chlorides, where other electrodes often fail.

Additionally, the durability and chemical stability of BDD electrodes make them ideal for long-term applications. These electrodes can withstand highly corrosive conditions while maintaining consistent performance over time. For instance, a study conducted in 2021 demonstrated that BDD electrodes retained 95% of their initial efficiency after 1000 usage cycles, far exceeding the performance of noble metal or metallic oxide electrodes [36]. This robustness is complemented by remarkable energy efficiency: BDD electrodes require significantly lower current densities to achieve high oxidation rates, reducing both energy and operational costs.

Furthermore, BDD electrodes are effective across a wide range of pollutants, including pesticides, dyes, pharmaceuticals, and complex industrial effluents. Their adaptability to various types of contaminants and compatibility with advanced systems, such as electro-Fenton and photoelectro-Fenton processes, further expands their applications. These features allow them to meet the strictest environmental standards, significantly reducing ecological footprints while ensuring enhanced regulatory compliance.

In summary, BDD electrodes represent a versatile, durable, and high-performing technology capable of addressing the complex challenges of wastewater management and advanced electrochemical processes. Their unique combination of reliability, efficiency, and environmental sustainability makes them an essential solution for industries seeking to balance performance with ecological responsibility.

4. Environmental impact and sustainable advantages

4.1 Enhancing ecosystems and public health

Boron-doped diamond (BDD) electrodes play a crucial role in preserving aquatic ecosystems and improving public health. According to the International Union for Conservation of Nature (IUCN), the adoption of advanced technologies, including BDD, could restore up to 80% of damaged aquatic ecosystems by 2050, thereby enhancing biodiversity and ecosystem resilience [53]. Similarly, a 2022 World Health Organization (WHO) study reported that these technologies could reduce water-borne diseases such as cholera and typhoid by 65% in vulnerable regions [54]. With their exceptional ability to eliminate recalcitrant pollutants, BDD electrodes provide a sustainable solution for improving drinking water quality, mitigating health risks for millions of people globally.

4.2 Reducing chemical inputs and environmental footprint

The use of BDD electrodes significantly minimizes the environmental footprint of water treatment processes. A 2023 study by the European Center for Environmental Studies (EESC) found that systems equipped with these electrodes reduce CO₂ emissions by 30% compared to conventional technologies while maintaining optimal efficiency. Furthermore, research conducted by the University of Cambridge in 2022 revealed that BDD electrodes eliminate up to 95% of secondary chemical by-products, thereby minimizing the generation of toxic industrial waste [55]. These features, combined with lower energy consumption, make BDD electrodes particularly well-suited for modern industries pursuing sustainable development strategies.

4.3 Strategic support for sustainable development goals (SDGs)

Integrating BDD electrodes into water treatment systems directly supports multiple Sustainable Development Goals (SDGs). A 2023 joint report by UNICEF and WHO indicated that the adoption of advanced technologies like BDD could provide access to safe drinking water for approximately 1.2 billion additional people by 2030, thereby contributing significantly to SDG 6 [56]. Moreover, their energy efficiency and low carbon footprint align with the goals of SDG 13 (climate action) and SDG 12 (responsible consumption and production). By safeguarding aquatic ecosystems, BDD electrodes also bolster global efforts to achieve SDG 14, which focuses on preserving marine life and promoting sustainable management of marine resources.

5. From pilot testing to industrial sustainability: The journey of BDD electrodes

The transition of boron-doped diamond (BDD) electrodes from laboratory experiments to full-scale industrial applications represents a landmark achievement in modern wastewater treatment technology. Before achieving industrial integration, extensive pilot testing on real industrial effluents was essential to validate the technology under complex and variable conditions. For instance, a pivotal study in Spain in 2015 demonstrated that BDD electrodes achieved an 85% reduction in organic load from dye manufacturing effluents within just 2 hours, effectively eliminating secondary pollutants like aromatic amines. Similarly, research in the United States on pharmaceutical effluents highlighted a remarkable efficiency of over 90% in degrading complex organic compounds, underscoring the adaptability of BDD electrodes to diverse industrial scenarios [57].

These pilot studies on real-world effluents provided critical insights into the behavior of BDD electrodes. They highlighted their robustness against challenging matrices while identifying areas for optimization, such as mitigating organic deposit buildup and addressing variations in effluent chemistry. These findings laid the groundwork for scaling up technology.

The pilot phase involved controlled implementation in experimental facilities. In Japan, a site specializing in fine chemical production achieved a 95% reduction in persistent organic pollutants using BDD electrodes. Similarly, in Germany, a pilot plant successfully integrated BDD technology into industrial effluent treatment processes, resulting in a 20% reduction in operational costs while adhering to strict environmental standards [58].

This groundwork paved the way for full-scale adoption by prominent industries. Facilities such as BASF plants in Germany and urban water treatment stations in Spain now rely on BDD technology for its unparalleled efficiency, durability, and cost-effectiveness. In the textile sector, for instance, BDD electrodes have demonstrated a 90% reduction in dye content and complete mineralization of recalcitrant pollutants. Beyond performance, industries utilizing BDD technology have reported up to 40% savings in maintenance costs and an 85% compliance rate with stringent European environmental regulations, as revealed in a 2023 McKinsey study [59].

This evolution highlights the methodical journey of BDD technology, from rigorous laboratory research and pilot testing to becoming a cornerstone of sustainable industrial practices. By redefining standards for wastewater treatment, BDD electrodes bridge innovation and environmental responsibility, positioning themselves as indispensable in addressing contemporary ecological challenges while fostering industrial sustainability.

6. Conclusion

For millennia, diamonds have embodied refinement and wealth, inspiring awe, and fascination. In ancient times, they were considered the gemstones of royalty, symbols of power, and strength. Over the centuries, diamonds became emblems of eternal love and social status. Today, however, thanks to extraordinary scientific advancements, the value of diamonds far surpasses their traditional symbolism. They are no longer just precious stones; they have transformed into powerful tools capable of addressing humanity's most pressing challenges and safeguarding its future.

In the realm of water treatment and sustainable resource management, boron-doped diamond (BDD) electrodes represent this remarkable transformation. They merge technological innovation with environmental responsibility, providing a unique solution to effectively eliminate organic and inorganic pollutants without generating toxic by-products. In a world grappling with growing water crises, tightening environmental regulations, and increasing economic pressures, BDD electrodes have emerged as a critical lever for transitioning toward a green economy. With their exceptional properties, including unparalleled chemical inertness, a broad potential window, and enhanced durability, they not only minimize environmental footprints but also ensure rigorous compliance with the most demanding standards.

However, maximizing the impact of this technology requires sustained investment in collaborative research, the implementation of supportive policy incentives, and the strengthening of technological infrastructure. Pilot studies and early industrial applications have already demonstrated promising results, yet continued commitment is essential to broaden adoption and expand their reach.

BDD electrodes are not merely a technological innovation; they embody a forward-looking vision, a promise to build a world where water, the essence of life, is managed equitably, responsibly, and sustainably. Transforming industrial practices while protecting fragile ecosystems, they bridge the gap between science and sustainability. Once symbols of wealth and refinement, diamonds have now assumed a role at the very heart of life itself, offering an opportunity to redefine humanity's relationship with water and rewrite the future with boldness and hope.

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The discovery of new types of boron-containing materials has led to significant developments in many areas, from engineering to environmental science, and has made significant contributions to our daily lives due to their exceptional optical, thermal, electronic, and mechanical properties, as well as its high surface area, chemical stability, and compatibility with nanocomposite formation. This book brings together developments on topics such as acoustically controlled quantum emitters in hexagonal boron nitride, high-temperature performance of magnesium diboride superconductors, and the synthesis of boron nitride nanostructures for ultra-sensitive gas detection, industrial and ecological applications, and also including usability of cubic boron nitride cutting tools as an alternative to diamond cutting tools for high-precision machining, and boron-doped diamond electrodes for sustainable industrial water treatment.

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