Emerging Applications of Boron Nitride Nanotubes in Energy Harvesting, Electronics, and Biomedicine

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ABSTRACT: Boron nitride nanotubes (BNNTs) are structurally and mechanically similar to carbon nanotubes (CNTs). In contrast, BNNTs exhibit unique properties for being electrically insulating and optically transparent due to the polarized boron nitride bonds. All these properties have prevented the use of BNNTs for energy harvesting and electronic devices for more than 25 years. During the past few years, researchers have started to demonstrate a series of novel applications of BNNTs based on unique properties not found on CNTs. For example, these novel applications include osmotic power harvesting using the charged inner surfaces of BNNTs, room-temperature single-electron transistors using insulating BNNTs as the tunneling channels, high-brightness fluorophores that can be 1000-times brighter than regular dyes, and transistors based on Tellurium atomic chains filled inside BNNTs. We have reviewed some of these emerging applications and provided our perspective for future work.

1. INTRODUCTION

Boron nitride (BN) materials are isoelectronic to carbon solids with similar crystalline structures. They could appear as cubic (c-BN), hexagonal (h-BN), and nanotubular (boron nitride nanotubes, BNNTs) allotropes.1 Because of the polarized BN bonds, electrons are localized at the nitrogen atoms and lead to the broad bandgap nature in all BN materials. Therefore, bulk and two-dimensional (2D) sheets of h-BN are electrically insulating. In contrast, the nonpolarized C=C bonds make graphite and graphene metallic. In another example, variation in chirality makes carbon nanotubes (CNTs) metallic or semiconducting. This contrasts with BNNTs, which exhibit wide bandgaps (6 eV) that are not sensitive to the change in chirality.2,3 Despite these differences, BNNTs share similar mechanical properties with CNTs. Therefore, the initial applications of BNNTs are similar to those of CNTs and focus on mechanical reinforcement in polymer composites.4 The synthesis of BNNTs requires very high furnace or arc plasma temperatures, involves hazardous chemicals, or uses unique synthesis methods such as ball milling and laser or plasma heating. Most of these approaches produced entangled BNNTs predominated with impurities. These BNNTs are challenging to disperse and untangle into individual nanotubes for use in applications.

During the past few years, applications of BNNTs have been demonstrated on the basis of their unique properties not found on CNTs. These include, for example, in situ probing of mechano-electronic or piezoelectricity in BNNTs,5 transistors and switches without semiconductors,6 giant osmotic energy conversion,7 the formation of atomic chains inside BNNTs for novel transistor applications,8,9 quantum dots with optical bandgaps on BNNTs,10 high-brightness fluorophores for biomedical application,11 and the formation of atomic chains inside BNNTs for novel transistor application.12 Here we will review some of these emerging applications as schematically summarized in Figure 1. The overall goal of this Mini-Review is to provide readers a collection of applications that were enabled only by using BNNTs. We aim to inspire creativity in using the charged surfaces, electrically insulating, and optically transparent nature of BNNTs for novel applications, which is now becoming the new BNNT research trend.

2. AVAILABILITY OF CLEAN BORON NITRIDE NANOTUBES FOR APPLICATIONS

The difficulty in synthesizing BNNTs is one of the critical reasons for the slow progress toward their applications. The synthesis of BNNTs requires very high furnace or arc plasma temperatures, involves hazardous chemicals, or uses unique synthesis methods such as ball milling and laser or plasma heating. Most of these approaches produced entangled BNNTs predominated with impurities. These BNNTs are challenging to disperse and untangle into individual nanotubes for use in...
applications. Detailed reviews about the progress of synthesis, purification, and dispersion are available elsewhere (more discussion in the Supporting Information). In 2006, Wang et al. demonstrated the patterned growth of BNNTs by plasma-enhanced pulsed-laser deposition technique at 600 °C, which started the catalytic synthesis of clean BNNTs. In 2008, Lee et al. reported the growth of clean BNNTs by thermal chemical vapor deposition (CVD) method at 1200 °C, using a regular resistive tube furnace commonly used to synthesize CNTs and ZnO nanostructures. According to the nucleation theory, the growth vapor trapping approach (GVT) can enhance the nucleation probability of BNNTs at a moderate temperature by increasing the partial vapor pressure using a closed-end inner quartz test tube. The authors further demonstrated catalytic CVD growth of BNNTs using MgO, Ni, and Fe catalysts by GVT. This approach has gained a lot of popularity for the advantages of using regular tube furnaces available in many laboratories and without using any hazardous chemicals. High-quality and high-purity BNNTs can be grown by this method, but the yield of BNNTs is just sufficient for research use. The major advantage is that these clean BNNTs could be easily dispersed for application. Plasma heating approaches can produce a larger quantity of BNNTs. However, these plasma techniques make entangled BNNTs as most nanotubes are stained and fused with boron and boron nitride particles and make the dispersion of nanotubes challenging. A recent review has made a detailed comparison of these recent synthesis approaches.

3. EMERGING APPLICATIONS BASED ON THE UNIQUE PROPERTIES OF BNNTS

3.1. Osmotic Power Harvesting. Siria et al. reported electric current generation induced by salinity gradients across the tubular channel of individual multiwalled BNNTs. As shown in Figures 1a, the authors prepared a fluidic device with one BNNT connecting two reservoirs with KCl solution of various concentrations (~0.001 to 1M) and controlled pH (~3 to 11). They insert the BNNT through a nanopore (~50–100 nm) on the Si3N4 membrane using an in situ nanomanipulator to form a transmembrane nanotube (t-BNNT). Some of the inner shells of such a t-BNNT were then removed by the telescoping approach. Such a process will leave a t-BNNT of about one micrometer long (~800–1250 nm) through the nanopore of each Si3N4 membrane. The diameter of the opened inner channel of such a t-BNNT is about 29–40 nm. Afterward, they use Ag/AgCl electrodes to measure the electric current across the BNNT tubular channel. The ionic current can be induced by an electric potential drop, a pressure drop, or a salt concentration gradient. For example, the authors can generate a few picoamperes from the pressure-driven flow through the t-BNNT. The authors found that a concentration gradient between two KCl reservoirs creates an osmotic current 2 orders of magnitude higher than those produced by pressure-driven flows. Each one t-BNNT can generate a maximum power density of 4 kW m⁻² attributed to the anomalously high surface charge carried by the BNNT’s internal surface in water at large pH. The authors suggested that this current is due to the
diffusio-osmotic flux induced by the difference in salt concentration at the inner surface of the nanotube. Such a salt concentration difference builds an osmotic pressure gradient within the diffuse layers next to the inner nanotube surfaces. The charged nanotube inner surfaces can spontaneously dissociate water and become functionalized with OH-group to induce such a salt concentration gradient. The generated osmotic pressure gradient is typical ~50 bar, much higher than the applicable pressure range for pressure-driven flows. Finally, the osmotic current for each individual t-BNNT is proportional to its radial R and inverse proportional to its length L. For the case of a closely packed BNNT membrane where nanotube density scales as 1/R², the total current scales as 1/R. There is no report on energy generation by such BNNT membranes.

3.2. Transistors and Electronic Switches without Semiconductors. 3.2.1. Transistors by Quantum-Dots Functionalized BNNTs. Shrinking the feature size of silicon field-effect transistors (FETs) is encountering fundamental limits inherent to the semiconducting channel, including current leakage, short channel effects, and high contact resistance. Semiconducting CNTs, nanowires, and two-dimensional (2D) material will encounter similar limitations.

Lee et al. demonstrated a novel method for creating FETs by using electrically insulating BNNTs without involving semiconductors. Quantum tunneling between gold quantum dots decorated on the surfaces of BNNTs (QDs-BNNTs) is the transport mechanism of these room-temperature single-electron transistors (SETs), as graphically illustrated in Figure 1b. The authors used four-probe scanning tunneling microscopy (4-probe STM) to characterize an isolated QDs-BNNT in contact with two STM probes at the desired transport length (L). The turn-on voltages (V_on) of this QDs-BNNT decreases from about 34 V to 2.0 V as transport length L decreases from 2.37 to 1.29 μm. This means these SETs can perform better with lower turn-on voltages without the sign of short channel effects. The turn-on voltages below 0.2 V also can be modulated by a back-gate potential. The on-off ratio is estimated to be on the order of 10⁴. In addition to gold QDs, the authors have reported that Fe nanoparticles coated BNNTs could work as the tunneling channels. They further demonstrated that randomized Fe QDs on BNNTs forming the flexible channels where the transport properties are invariant even under large-angle bendings up to 75° as shown in Figure 1c.¹¹

3.2.2. Electronic Switches by Graphene-BNNT Heterojunctions. Graphene exhibits the high electron mobility needed for high-frequency analog devices. The h-BN substrates are known to enhance the electron mobility of graphene devices. Unfortunately, the gapless graphene is not applicable for digital switches. Parashar et al. show that heterojunctions between BNNTs and graphene could switch current flows. They characterized the as-grown graphene-BNNT heterojunctions at room temperature using 4-probe STM system. Figure 1d shows the graphic of an upright BNNT grown from the graphene surface. Such BNNT was experimentally synthesized and probed by a tungsten STM probe 1, while another STM probe 2 was in contact with the graphene surface. The distance, d, between probe 1 and the vertical graphene-BNNT heterojunction, was controlled by changing the contact point on the BNNT. For the case of d = 1.23 μm, no current was detected across the graphene-BNNT heterojunction for a range of bias voltages. The noise level (i.e., “Off” state) is about ~10⁻¹¹A. For d = 0.62 μm, a significantly higher current across the heterojunction (i.e., “On” state) was detected with a threshold turn-on voltage, V_on ~ 0.5 V. The current level detected at d = 0.10 μm is even higher, up to mA level (as limited by the preset 1 mA current limit), with V_on ~ 0.15 V. These conducting behaviors with obvious “on” and “off” states are different from the reported insulating nature of BNNTs. Such a switching behavior is also different from the metallic nature detected when both STM probes are in contact with the graphene surface. These results suggest that graphene-BNNT heterojunctions could be used as electronic switches with an estimated switching ratio of 10⁶.

3.3. High-Brightness Fluorophores (HBFs) for Biomedicine. Multicolor flow cytometry (FCM) is a laser spectroscopy technique that can detect, count, and sort thousands of cells per second. FCM is used to analyze and sort live cells and other biological or artificial particles. For example, FCM can measure multiple characteristics of individual cells suspended in a fluidic sample that flows through the laser focus points. Laser light scattering at different angles can distinguish differences in size, granularity, and internal complexity. In contrast, light emitted from fluorescently labeled antibodies can identify a wide array of cell surfaces and cytoplasmic antigens. The multiparametric and high throughput capabilities have made FCM a standard technique for research and clinical laboratories to characterize stem cells, cancer cells, nucleic acids, and so on.

There has been tremendous development in hardware, software, and fluorophores in FCM. Unfortunately, spillover between adjacent fluorescence spectra has made multicolor measurements challenging. Figure 2a conceptually illustrates two fluorescent signals from two antigens with high (blue-green) and low (yellow-green) populations. Since the weak signal is significantly overlapped (spillover) with the strong signal, it is not easy to quantify these two antigens’ populations accurately by using traditional dyes low in brightness (Figure 2b). Researchers can label antigens with high-brightness dyes to resolve the spillover issue. Figure 2c shows that brighter dyes can make stronger signals. Selectively, one can use a brighter dye to mark the rare antigen and vice versa. This approach can improve the resolution of the two adjacent spectra and make rare antigen detection possible (Figure 2d). The use of traditional dye also suffers from autofluorescence, especially at the green fluorescence channel. Autofluorescence is fluorescence noise from biological substances such as mitochondria and lysosomes. Autofluorescence increases the noise level and makes dim signal detection in the green spectra challenging (Figure 2e). As illustrated in Figure 2f, high-brightness dyes can enhance the signal-to-noise levels and resolve the autofluorescence issue.

There are four types of fluorophores: (1) small organic dyes such as FITC (Fluorescein isothiocyanate), RhB (Sulforhodamine B), Cy5 (Cyanine 5); (2) protein-based dyes such as PE (Phycocerythrin), APC (allophycocyanin), and PerCP (peridinin chlorophyll); (3) quantum dots; (4) polymer dyes such as Brilliant Violet and Super Bright dyes. Only three of these fluorophores (PE, APC, BV421) offers bright fluorescence with molar extinction coefficients of 0.7 × 10⁶ to 2 × 10⁶ M⁻¹cm⁻¹. Therefore, multicolor panel designs with more than six parameters are limited. Therefore, the development of high-brightness dyes is essential to overcome the spillover and autofluorescence issues and enable practical
Yap et al. proposed to use BNNTs as the nanocarriers to create high-brightness fluorophores (HBFs) by controlling the molar extinction coefficients. Fluorescence intensity (\(I_f\)) of fluorophores is scaling with the excitation light intensity (\(I_o\)), quantum yield (\(\Phi_f\)), the concentration of the fluorophores (\(C\)), geometrical excitation length (\(L\)), and molar extinction coefficient (\(\varepsilon\)) in the following relation, \(I_f = 2.303 I_o \varepsilon C L \Phi_f\). Under the same experimental setting (\(I_o, C\), and \(L\) are constants), fluorescence intensity depends on \(\varepsilon\) and \(\Phi_f\). Since \(\Phi_f\) is between 0 and 1, and \(\varepsilon\) could be varied by several orders of magnitude, it is very effective to create HBFs by modulating \(\varepsilon\). One can define \(\varepsilon\) as the amount of light (photons) absorbed by the fluorophores at specific wavelengths per concentration (\(C\)) per unit length (\(L\)) and represented by the unit of M\(^{-1}\)cm\(^{-1}\). Therefore, by organizing thousands of dye molecules on each BNNT, one could enhance \(\Phi_f\) and therefore \(I_f\) by 3 orders of magnitude per fluorophore (1000\times brighter).

As shown in Figure 3a, the authors could conjugate one FITC molecule with one linker, amine-functionalized (1,2-distearoyl-sn-glycero-3-phosphoethanolamine-N'-(polyethylene glycol)\(_n\)), i.e., DSPE-PEG5K-NH\(_2\), to form one unit of dye-linker (Figure 1e). Figure 3b shows the noncovalently functionalization of dye-linkers onto each BNNTs by dialysis exchange protocol.\(^{15}\) BNNTs functionalized with DSPE-PEG are exceptional in dispersity in water for biomedical application (Figure 1f).\(^{17}\) A similar approach is applicable when using RhB and Cy 5. The authors found that the quantum yield (QY) of these dyes after being functionalized on BNNTs remains the same as the QY of free dye-linkers. Furthermore, the extinction coefficients of these HBFs (NovoLux dyes by StabiLux Biosciences) are scalable between \(10^8\) to \(10^{11}\) M\(^{-1}\)cm\(^{-1}\), up to 3 orders of magnitude brighter than the organic dye.\(^{15}\) This means the electrically insulating BNNTs can prevent quenching of these FITC dyes and allow scaling of the molar extinction coefficient to achieve HBFs. This is consistent with an earlier report where polymeric dyes can emit bright fluorescence.
fluorescent when adsorbed on BNNTs while not on CNTs. These HBFs can be conjugated with antibodies to detect antigens on peripheral blood mononuclear cells by FCM. In contrast, researchers reported that dye molecules labeled on CNTs would quench by 20−80% due to electron transfer.

3.4. Unique Atomic Structures on BNNTs and inside BNNTs. The wide bandgap nature of BNNTs is responsible for their electrically insulating and optically transparent properties. The fully coordinated sp2 B = N nanocylindrical network of BNNTs has made them atomically smooth, defect-free, and chemically inert substrates to deposit atomic structures on their exterior surfaces or inside the tubular channels, as discussed in the following subsections.

3.4.1. Monatomic Thick, Two-Dimensional Gold Quantum Dots. Gold nanoparticles (Au NPs) are of interest for their intriguing properties for plasmonic and metamaterial devices, field-effect transistors without semiconductors, and catalysts for chemical reactions and biological sensing. Recently, Bhandari et al. reported the discovery of 2D gold quantum dots (Au QDs, Figure 1g) with monolayered atoms and dot sizes as large as ∼4 nm (more than 100 atoms). The results are consistent with their theoretical modeling, in which 2D Au, with n as large as about 100 atoms can be stabilized on the hexagonal BN (h-BN) substrates. Atomic resolution images obtained by aberration-corrected scanning transmission electron microscopy (STEM) show that thinner and smaller 2D Au QDs are coated on surfaces facing away from the gold vapor generated by PLD.

The formation of the 2D Au QDs is attributed to the directional gold plume and the cylindrical BNNT substrates. For example, gold vapor generated by laser ablation propagates from left to right toward a cylindrical BNNT. The left BNNT surface facing the gold vapor will be coated with gold adatoms to form 3D Au NPs. In contrast, the other surfaces will be blocked from the direct coating because of the directionality of the vapor generated by the PLD. Some of the more energetic adatoms would diffuse away from the 3D Au NPs and land on the side or back surfaces of the BNNTs. The authors detected multiple sharp optical absorption states from these 2D Au QDs. These absorption states corresponded to energy bands ranging from 1.46 eV to 2.52 eV, well-matched to the calculated bandgaps. The atomically flat surfaces of BNNTs allow them to determine the Au monolayers with atomic resolution. In addition, the optically transparent and electrically insulating BNNTs enable the detection of sharp optical band gaps in the visible spectrum range.

3.4.2. Tellurium Atomic Chains inside BNNTs for FETs. Nanotubes are unique tubular structures without any dangling bonds on the exterior and interior surfaces. Therefore, the adsorption of atoms or molecules on these surfaces is limited to van der Waals interaction. The filling of C60 carbon, sulfur, and metals inside CNTs has stimulated many theoretical and experimental interests in using nanotubes as the hosts to confine other atomic and molecular structures inside the tubular channel. However, researchers could not directly measure the electronic properties of these filled materials as current flows would have interfered with the metallic or semiconducting nature of CNTs.

Recently, Qin et al. demonstrated the filling of tellurium (Te) atomic chains inside CNTs and BNNTs. These Te atoms are covalently bond within each atomic chain. All the individual atomic chains are stacked on each other by van der Waals forces. The authors have characterized the structural and Raman properties of these Te atomic chains inside CNTs and BNNTs. They could not measure the electronic properties of Te inside CNTs, as current will be interfered with by the conducting or semiconducting CNTs. Interestingly, BNNT's successfully confined Te chains with diameters from 2 to 10 nm and allowed FETs to construct with a channeling length of 100 nm, as schematically shown in Figure 1b.

The transport nature of Te-BNNT FETs is n-type behavior after Al2O3 capping, indicating that the classical Fermi-level pinning issue does not exist in such Te-BNNT FETs. This result is in contrast to the p-type behavior of FETs using bare Te nanowires. Besides, the 2 nm FETs exhibit a large “on” state current of up to 50 mA/mm (Figure 4e). The authors found that Te-BNNT is among the semiconducting materials with high ampicity.

4. FUTURE PERSPECTIVE AND SUMMARY

Since the experimental realization of BNNTs in 1995, the electrically insulating nature of BNNTs has prevented their usage for energy production and electronic devices for more than 25 years. As discussed, researchers started to break through such hurdles from 2013, when Siria et al. reported electric current generation by salinity gradients across the tubular channel of individual BNNTs and Lee et al. demonstrated SETs based on QDs-BNNTs. For now, giant osmotic power generation is the only method to use BNNTs for energy production. Thus far, such a demonstration is based on devices with individual t-BNNTs. The next step would be to integrate arrays of BNNTs into fluidic membranes for practical usage. Centindag et al. has started in some initiatives by aligning BNNTs in aqueous solution under spatially uniform electric fields. Apparently, large-scale dispersion and integration of BNNTs into membranes are still challenging and would be an essential topic for the practical use of BNNTs in power production. In addition, condensation of salt crystals is reported within the channel of the t-BNNTs, signifying an issue to continuously generate power by such an approach. Future research in resolving overcoating of the BN charged surfaces by salt crystals will be necessary for the practical use of such energy-harvesting devices. For the application in SETs, researchers face a similar challenge of the scalable and controllable fabrication of SET devices using QDs-BNNTs. In particular, consistent fabrication of SETs with very short tunneling channel lengths would be of interest for high on−off ratios.

The technological challenges in making devices out of graphene-BNNT heterojunctions are substantial. Such a 3D heterojunction will require a surrounding-gate or a gate-all-around (GAA) configuration to complete device fabrication. Furthermore, heterojunctions with monolayer-graphene would be more desired, which will combine high electron mobility and digital switching behaviors. On the other hand, the more recent discovery of 2D Au QDs with monolayered atoms is exciting. These tiny QDs may become future semiconductors with less than 100 atoms. Since the dot sizes are smaller than ∼4 nm, device fabrication would be highly challenging. The filling of Te atomic chains inside BNNTs is a beautiful demonstration of the practical use of electrically insulating BNNTs for FETs. We are expecting more attempts to fill other semiconducting materials inside BNNTs for electronic applications. Instead of filling BNNTs, Xiang et al. reported overgrown with BN shells on single-walled CNTs. The authors show that BNNTs are excellent materials for...
encapsulation of otherwise chemically/physically sensitive CNTs.

More recently, Allard et al. demonstrated the filling of BNNTs with dye molecules (dye@BNNTs).22 They found that BNNTs could protect the dyes against photobleaching and chemical degradation, allow aggregation of dyes that produce fluorescence red-shifts, and reduce the inherent toxicity of dyes on living tissues. The disadvantage of dye@BNNTs is that the quantum yield (QY) of the dye, for example, 6T (ππ-sesquithiophene), has reduced to 8%−16%, much lower than that of free 6T dye (QY ~ 0.6−1.0).

Last but not least, let us revisit HBFs made by using BNNTs as the carriers for FCM application. As discussed, these HBFs can be 1000× brighter than regular dyes, thanks to the electrically insulating BNNTs that prevent quenching. The self-assembly of linker-dyes on the exterior surfaces of BNNTs is proven to be versatile to prevent collisional quenching of dyes, which allows scientists to scale the brightness of each fluorophore by loading different amount of dye molecules on each BNNTs. Preliminary FCM data that demonstrated the high fluorescent brightness presented in conferences are promising.23−25 Detailed FCM data in detecting rare antigens would be interesting to establish actual use in biomedicine fully.

In summary, emerging applications of BNNTs have started to attract a lot of attention during the past few years. Researchers have demonstrated these novel applications based on the unique properties of BNNTs that are not found in CNTs, such as charged surfaces, electrically insulating, and optically transparent. We believed that creativity constructed around this direction would lead to more attractive applications in years to come, both experimentally and theoretically. This Mini-Review aims to inspire efforts in developing novel applications of BNNTs based on their unique properties. We hope that this will be developed into a new research trend of BNNT research. The crucial step for developing more BNNT applications would be a further advancement in the controlled synthesis of clean and dispersible BNNTs in a more scalable manner. Creativity in these regards is critically important; without which, further exploration of unique BNNT applications will be prevented.

■ ASSOCIATED CONTENT

! Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsomega.1c02586.

The Supporting Information contains a section on “Large-Quantity Synthesis and Purification of Boron Nitride Nanotubes (Figure S1)” (PDF)

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