

Review Article

Recent Development of the Synthesis and Engineering Applications of One-Dimensional Boron Nitride Nanomaterials

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One-dimensional (1D) nanomaterials with novel photoelectric, magnetic, mechanical, and electronic transport properties have long been the research focus throughout the world. Herein, the recent achievements in preparation of 1D boron nitride nanomaterials, including nanotubes, nanowires, nanoribbons, nanorods, and nanofibres are reviewed. As the most intriguing and researched polymorph, boron nitride nanotubes (BNNTs) are introduced thoroughly involving their functionalization and doping. The electronics and engineering applications of 1D boron nitride nanomaterials are illustrated in nanoscale devices, hydrogen storage, polymer composites, and newly developed biomedical fields in detail.

1. Introduction

A nanoscience and nanotechnology revolution initiated by the observation of carbon nanotubes (CNTs) [1] has rapidly swept the world over the last couples of decades. A number of peculiar phenomena were found at the nanoscale. Of the epoch-making revolution, nanomaterials lie in the central position and construct a bridge between nanoscience and nanotechnology. Synthesis and characterization of new types of nanomaterials have attracted enormous concentration of scientists in chemistry, physics and materials. Of all, 1D nanomaterials such as tubes, wires, ribbons, fibers, and rods with exceptional electronic, mechanical, optical and magnetic properties have been widely researched and used as building blocks in nanoscale devices [2]. It is generally suggested that quantum confinement in 1D nanostructures leads to novel electronic and transport properties, which provides a good model to investigate the effect of dimensionality and reduced size on the above properties of 1D nanomaterials [3]. Light-weight 1D nanomaterials with large elastic modulus can be promising candidate in engineering field and used as reinforcement agent into composite materials [4].

Hexagonal boron nitride (h-BN) with layered structure analogous to graphite is an important III-V material.

Stimulated by rapid development of carbon nanomaterials, a series of boron nitride nanostructures have been synthesized, especially 1D nanomaterials such as nanotubes, nanowires, nanoribbons, nanofibers, and nanorods (Figure 1) [5–9]. One-dimensional boron nitride nanomaterials exhibit not only excellent thermal conductivity and high elastic modulus comparable to these of carbon materials but better thermal and chemical stability. Contrary to carbon with good electrical conductivity, boron nitride is a nearly electrical insulator with a large band gap of ~5.5 eV which is independent of tube diameter and chirality [10]. Consequently, BNNT can be used as insulating protective shields encapsulating semiconducting nanowires to form nanocables [11–16]. BN nanowires and nanoribbons could play key roles in nanoscale devices [6, 7]. And BN fibers may substitute for carbon fibers under electrical insulating and rigorous environment at high temperature [8].

In this paper, the latest advances in synthesis and applications of 1D boron nitride nanomaterials in device and engineering are comprehensively introduced. Of all, BNNTs and their functionalization and doping are systematically reviewed as well as their various applications particularly in newly developed biomedical field. Other 1D BN nanomaterials are illustrated in detail as well.

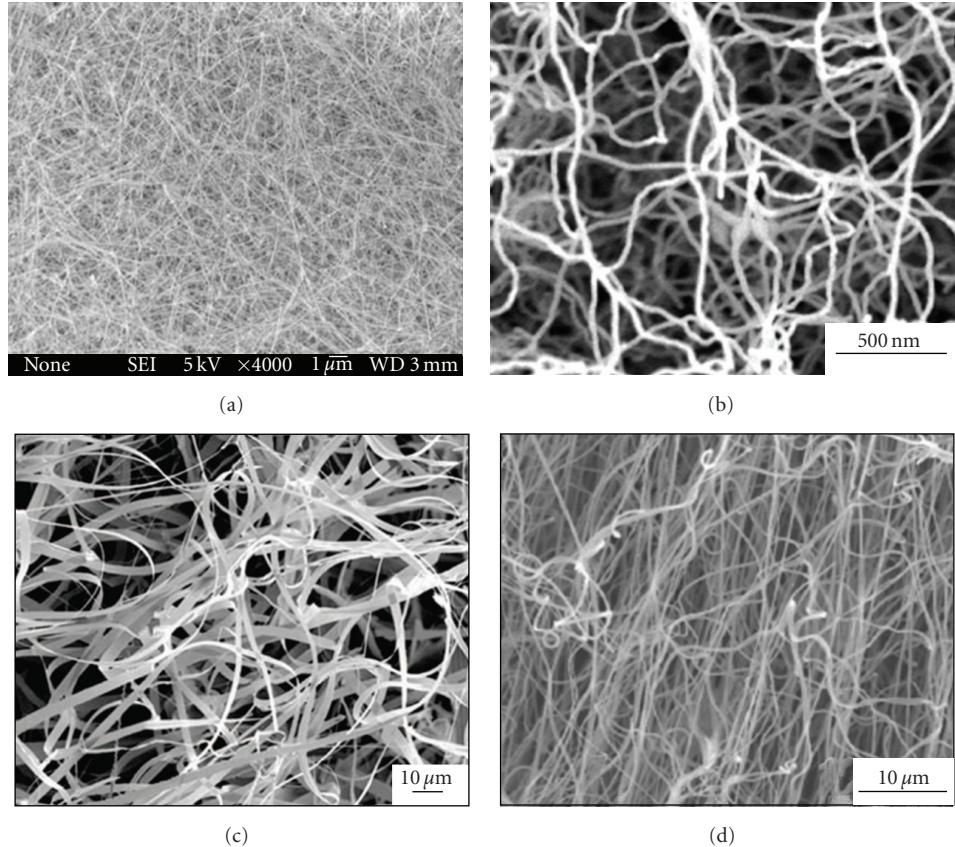


FIGURE 1: Family of 1D boron nitride nanomaterials. Scanning electron microscopy (SEM) images of (a) BN nanotubes [5], (b) BN nanowires [6], (c) BN nanoribbons [7], (d) BN nanofibers [8]. (a) Reprinted with permission of [5]. Copyright 2005 Elsevier Ltd. (b) Reprinted with permission of [6]. Copyright 2006 Institute of Physics. (c) Reprinted with permission of [7]. Copyright 2008 American Chemical Society. (d) Reprinted with permission of [8]. Copyright 2009 Institute of Physics.

2. Boron Nitride Nanotubes

2.1. Synthesis. BNNTs were first synthesized by Chopra et al. in a carbon-free plasma discharge between a BN-packed tungsten rod and a cooled copper electrode similar to CNTs synthesis in 1995 [17]. Following research improved this method by using HfB_2 [18, 19] or ZrB_2 [20] electrodes in a nitrogen atmosphere. Few-layer BNNTs including single-layer tubes were observed though with minor amounts (Figure 2). Most of the tube ends were closed by flat layers or encapsulated metal particles or amorphous BN material. When nitrogen-free, boron-rich electrodes incorporating minority of nickel and cobalt were arced in a nitrogen atmosphere, double-walled BNNTs self-assembled into bundles or ropes were mass-produced [21, 22].

BNNTs, especially single-layer ones, can be prepared by laser ablation. BNNTs of only 1 to 3 layers with flat or polyhedral caps were synthesized by using excimer laser ablating BN target with nanosized Ni and Co powder at 1200°C [23, 24]. Low-energy electron-cyclotron resonance (ECR) plasma was also used to deposit single-walled BNNTs with fourfold and eightfold ring structures on tungsten substrate [25]. In the absence of metal catalysts, single-walled nanotubes could also be synthesized in gram quantities using

CO_2 laser ablation [26], which indicated different growth mechanism from carbon single-walled nanotubes. Arenal et al. modified this route to increase the yield of single-walled BNNTs to 80% [27]. A root-growth model was proposed based on the observation of boron particles at the tube ends. Noticeably, Laude et al. reported similar experiments and the products were free of single-walled nanotubes [28]. These apparently controversial results suggested the poor stability of single-walled BNNTs compared with CNTs [29]. However, this method was the unique route known to the massive preparation of single-layer BNNTs. Besides, Wang et al. reported high-yield synthesis of multiwalled BNNTs deposited on Fe films coated oxidized silicon substrates adopting VLS growth mechanism using laser ablation at 600°C [30], which indicated the feasibility of laser ablation to prepare BNNTs in large scale. However, laser ablation, combined with arc-discharge, was limited owing to the expensive and complex apparatus.

Ball milling and annealing method with facile operation and mass production was successfully introduced to the synthesis of BN nanomaterials by Chen et al. in 1999 [31]. Originally, h-BN powder was used as the starting material which was ball milled and annealing at 1300°C under nitrogen gas flow [31]. The product was composed of

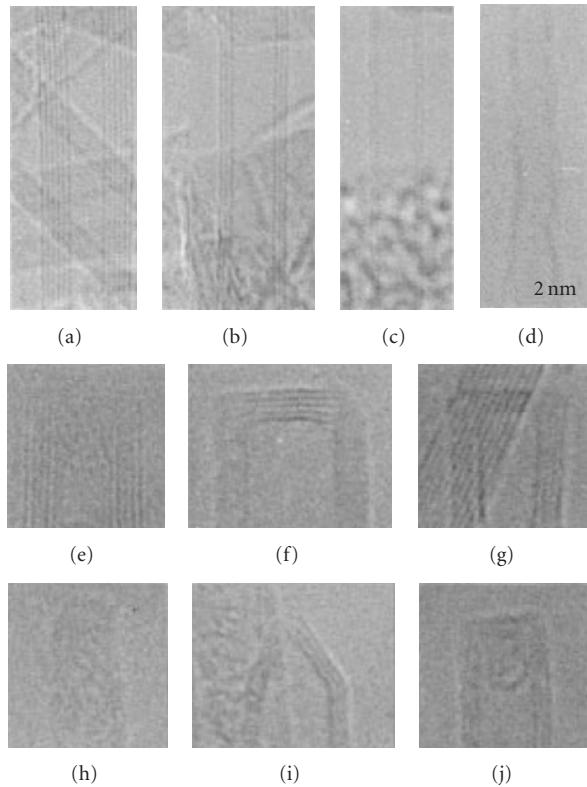


FIGURE 2: High-resolution transmission electron microscope (HRTEM) images of BNNTs prepared by arc discharge and their tube ends [18]. (a) Six layers, (b) three layers, (c) two layers, (d) one layer, (e–g) flat tips of tubes with different diameters, (h) disordered termination of a single-walled tube, (i) conical ends, (j) tube end encapsulating probably amorphous BN material. Reprinted with permission of [18]. Copyright 1996 American Physical Society.

both cylindrical and bamboo-like nanotubes. Subsequently, elemental B powder was substituted for h-BN powder as the starting material and annealing was carried out in a nitrogen atmosphere resulting in high yield (85%) production of BNNTs including cylindrical and bamboo-like nanotubes [32–34]. However, when the annealing process was conducted under ammonia gas flow keeping other conditions unvaried, pure cylindrical BNNTs were obtained which was attributed to two-dimensional growth of the BN phase without metal catalysis [35]. ^{10}BN nanotubes were prepared through substituting isotopically ^{10}B powder for B powder as the starting material catalyzed by Fe particles [36]. Based on systematic investigations of the nitriding gases on the growth of BNNTs, Chen et al. proposed that ammonia gas led to 2D growth of BN (002) planes, while nitrogen gas resulted in 3D crystal growth [37]. According to this model, selective synthesis of BNNTs, bamboos and nanowires was achieved through tuning the reaction parameters [38]. BNNTs obtained by this method were generally catalyzed by metal catalysts from the reaction vessel [39]. Besides, B_2O_3 can also be used as the starting material producing high-yield bamboo-like BNNTs [40]. Interestingly, over 1.0 mm BNNTs with huge resistivity could be synthesized by an

optimized ball milling and annealing method [41]. Overall, this method provides an effective mechanochemical route for the growth of BNNTs from solid phase, which is suitable for mass production of BNNTs.

In view of similar structures between CNTs and BNNTs and high reaction activity of carbon at high temperature, CNTs were utilized as the template for the growth of BNNTs defined as carbon nanotube substitution reaction. Han et al. initiated this field by reacting B_2O_3 with nitrogen gas in the presence of multishell CNTs at 1500°C [42]. The as-obtained BNNTs were mixed phases including h-BN and rhombohedral BN (r-BN). Precise structure investigations showed many changes from CNTs to BNNTs, for example, decreased numbers of shells, increased orderliness of nanotube shells along the tube axis, flat tip-end terminations, and so forth [43]. Novel conical nanotubes with their cone axis parallel to the tube axis were regularly found in the product [44]. Alternatively, single-walled CNTs were used successfully leading to the formation of single-layer BNNTs [45]. In a temperature-sensitive reaction single-layer BNNTs mixed with B_xC_{1-x} and $\text{B}_x\text{C}_{1-x}\text{N}_y$ single-walled bundles was produced using single-walled CNTs. Existence of carbon doped BNNTs or boron and nitrogen doped CNTs indicated the incomplete oxidation of templates. Nevertheless, these $\text{B}_x\text{C}_y\text{N}_z$ nanotubes can be further converted into BNNTs at 550°C with an efficiency of about 60% [46]. A dramatic increase in BNNTs yield was achieved by introducing low-sublimation-temperature metal oxides, for example, MoO_3 , V_2O_5 , PbO , CuO , and Cu_2O , into the reaction system as oxidization promoters [47–52]. Unique ropes of BNNTs were regularly formed containing tens of separate BNNTs via this route. The growth mechanism was considered to be oxidation-induced defect growth model. Aligned BNNTs were of great importance in the fabrication of nanoscale device which can be made interestingly via substitution reaction using aligned multiwalled CNTs [53]. Novel hollow conical-helix BNNTs were found through additional high-temperature treatment on the basis of above method and its structure was studied in detail [54, 55]. On the whole, although this route is easily subject to carbon contamination, carbon doped BNNTs with tuned band gap which is promising candidate in diverse photoelectrical device could be produced by carefully controlling the reaction parameters.

In the carbon nanotube substitution reaction, CNTs acted as both templates and reducing agent. Another commonly used template, porous anodic aluminum oxide (AAO), was utilized to fabrication of highly ordered BNNTs arrays. Borazine or related materials such as 2,4,6-trichloroborazine, polymeric borazine, borazine oligomer and BH_3NH_3 or B_2H_6 was generally selected as the starting materials [56–60]. The synthesis procedure was divided into three steps, that is, pyrolysis of the precursors at relatively low temperature (below 1000°C) to produce amorphous or turbostratic or poor crystalline BNNTs, removal of AAO template by immersing in 0.1 M NaOH solution or HF, and finally, not always needed, high-temperature treatment aiming at improved crystallinity. The key factor of this route is to choose a compatible precursor with the AAO templates. More important is that the route can also provide a facile way

to synthesize coaxial C/BN/C heterostructure by sequential pyrolysis of acetylene and trichloroborazine over alumina templates [57]. C/BN/C nanotubules are essentially a nano-sized capacitor with the specific capacitance ~ 100 pF/m exhibiting huge potential in microelectronics applications. Recently, a mixture of smooth boron nanowires and branched boron nanofeather nanojunctions was employed as precursor to react with nitrogen gas forming novel boron nitride nanotube branched nanojunctions which can act as functional units in nanoelectronic and nanomechanical devices [61]. The results demonstrated the template role of boron nanowires as well as reactant. Moreover, other hard templates may be attempted for the synthesis of BNNTs.

Actually, both the above-mentioned template methods are performed in chemical vapor deposition (CVD) equipment. CVD is the widely used method for BNNTs synthesis, which usually needs catalytic growth by metal particles following typical VLS mechanism. However, available boron source suitable for CVD synthesis is severely restricted. Frequently used gaseous boron-contained compounds in the synthesis of boron nitride films, most notably BCl_3 [62] and B_2H_6 , can also be beneficial to the growth of BNNTs. Recently, two groups reported the massive production of BNNTs by using B_2H_6 -based gas precursors catalyzed by Ni or Fe nanoparticles on similar substrate at low temperature (below 1000°C) [63–65]. Volatile BN compounds, typically borazine or $\text{H}_3\text{N}\cdot\text{BH}_3$, enriched the family of BNNTs precursors. Pioneering work of CVD synthesis of BNNTs was conducted by Lourie et al. using an *in situ* generated borazine as precursor on NiB or Ni_2B catalysts at 1000 – 1100°C [66]. Recently, double-walled BNNTs with ~ 2 nm diameters were obtained from borazine with a floating nickelocene catalyst at 1200°C [67]. Tang et al. developed a new synthetic system based on a novel precursor composed of B_2O_2 and Mg that was *in situ* generated by reacting B and MgO at 1300°C to synthesize bulk amounts of pure BNNTs in an ammonia atmosphere [68]. Later on, they improved this method by adding FeO to the starting mixtures aiming to promote the formation of highly reactive B_2O_2 intermediate (Figure 3) [5]. The self-formed Fe nanoparticles catalyzed the growth of BNNTs. Up to now, this route remains one of the most effective carbon-free routes to BNNTs in large scale, which advanced further functionalization and performance research of BNNTs. Subsequently, Tang attempted other metal oxides, such as, SnO , Fe_2O_3 , GaO , and so forth [69–72]. The results showed that the combined promoters of MgO and FeO apparently presented the predominant efficiency. Besides, some homemade precursors were employed to effectively synthesize BNNTs [73]. Ma et al. employed a B-N-O precursor prepared from boric acid and melamine to synthesize BNNTs under a nitrogen gas atmosphere at 1700°C [74, 75]. Structure analysis showed that the obtained BNNTs exhibited zigzag arrangement and rhombohedral stacking order. The nanotubes growth was suggested to be promoted by boron oxynitride nanoclusters at the tips involving Si, Al and Ca. Subsequently, controlled synthesis of BN nanotubes, nanobamboos, and nanocables was implemented using the B-N-O precursor under different conditions [76]. Ma also attempted to use NaBH_4 and

NH_4Cl to generate $\text{H}_3\text{N}\cdot\text{BH}_3$ which acted as precursors to yield BNNTs by pyrolysis at 1050°C under nitrogen gas atmosphere [77]. The as-produced nanotubes were found to grow in a layer-by-layer fashion. In view of its successful application in the mass production of CNTs, CVD technique may play a similar key role in the mass production of pure BNNTs in the future by selecting proper precursors and optimizing the reaction parameters.

One facile and easy-controlled method based on confined reactions in a sealed autoclave developed by our groups has been successfully utilized to preparation of various nanostructures including carbon, carbide, and nitride nanomaterials from 1D to 3D at low temperature [78–83]. The confined environment allows full diffusion and homogeneous distribution of reactive species leading to more efficient production and high anisotropic growth. We produced pure h-BN nanotubes with a maximum yield of $\sim 50\%$ via co-pyrolyzing NH_4BF_4 , KBH_4 , and NaN_3 at low temperature (450 – 600°C) under the synergetic effect of Zn and Fe powders (Figure 4) [84]. Diverse tips like acute and obtuse conical and square tips were observed in the product and many BN nanocages were found to coexist with BNNTs. The conical nanotubes with unique geometry may possess specific properties and promising applications. The experimental results demonstrated the crucial role of NH_4BF_4 in the formation of BNNTs. Recently, straight BNNTs with diameters ranging 30 – 300 nm were conveniently prepared by the similar method using $\text{Mg}(\text{BO}_2)_2 \cdot \text{H}_2\text{O}$, NH_4Cl , NaN_3 , and Mg powder as reactants at 600°C [85]. The BNNTs were closed at one ends or both ends and presented cone-like tips as well as the traditional tip-ends. Several BNNTs filled with Mg nanorods were occasionally observed. Therefore, it was suggested that Mg powder played a catalytic role in the formation of BNNTs. More recently, well crystalline BNNTs were synthesized as a side product together with hollow spheres [86]. The control experiments showed that crystalline BNNTs could be formed at quite low temperature of 300°C . Therefore, low temperature copyrolysis method in stainless steel autoclaves has been proved to be an effective way for the high yield production of crystalline BNNTs.

Other methods towards BNNTs were exploited. Arc plasma jet can generate high-temperature (~ 4000 K) resulting in evaporating exposed BN sintered disk to grow multiwalled BNNTs with square tips [87]. The innermost tube with diameter of 0.6 nm was found in the product.

Based on the above discussion, synthesis of pure multiwalled BNNTs have been achieved, although single-walled and aligned BNNTs which is of significance in device applications still remain a challenge. For industrial production, current methods developed in labs cannot meet the requirement yet. Moreover, precisely controlled synthesis of BNNTs with pure hexagonal or rhombohedral stacking and zig-zag or armchair arrangement needs further research. Remarkably, commercial BNNTs are currently available from READE Advanced Materials, Australian National University and/or other companies. Although the price is still very high now, this suggests huge application future of BNNTs undoubtedly. In order to fabricate devices using BNNTs, operation of BNNTs in solution via functionalization and

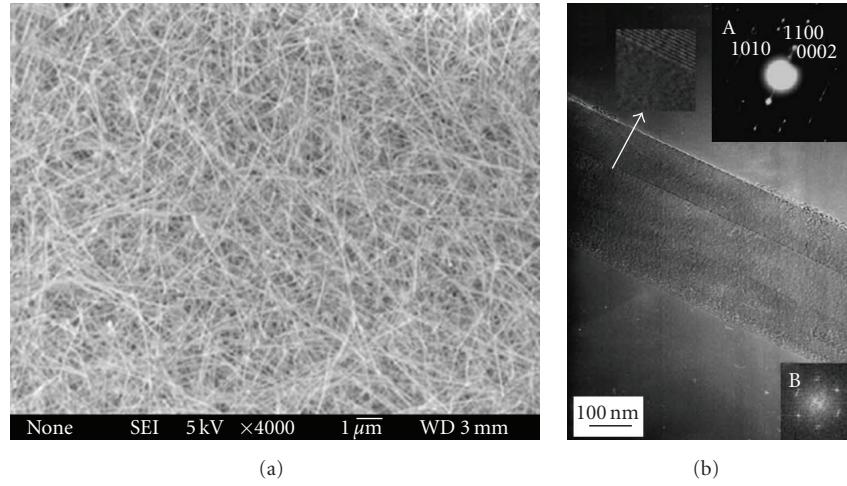


FIGURE 3: BNNTs obtained by oxide-assisted CVD method [5]. (a) SEM image of BNNTs. (b) HRTEM image of a BNNT. Selected area electron diffraction (SAED) in the inset A and the fast Fourier-transform (FFT) pattern in the inset B collectively confirmed the zigzag arrangement of this nanotube layers. Reprinted with permission of [5]. Copyright 2005 Elsevier Ltd.

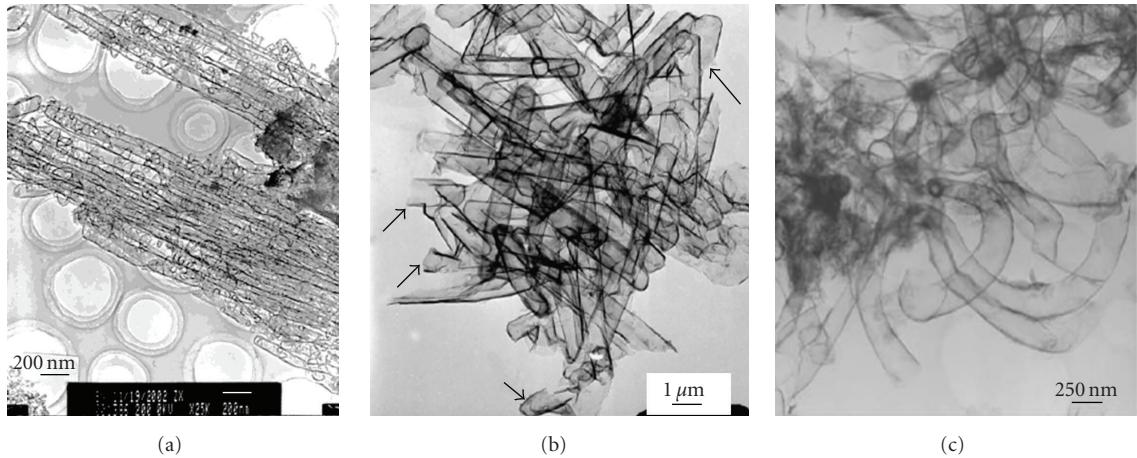


FIGURE 4: Typical transmission electron microscope (TEM) images of BNNTs prepared at low temperature. (a) Pure and high-yield straight BNNTs bundles [84]. (b) Straight BNNTs with large diameters of hundreds of nanometers and lengths ranging from 400 nm to 5 μm [85]. (c) BNNTs jungles crystallized at 450°C [86]. (a) Reprinted with permission of [84]. Copyright 2003 American Chemical Society. (b) Reprinted with permission of [85]. Copyright 2007 Elsevier Ltd. (c) Reprinted with permission of [86]. Copyright 2009 Elsevier Ltd.

doped BNNTs with tuned band gap are fulfilled, which is introduced below.

2.2. Functionalized Boron Nitride Nanotubes. Processability of BNNTs in solution is of great significance for the fabrication of nanodevice. In the last few years, following the successful massive production of pure BNNTs, many reports about surface modification of BNNTs towards solubility in aqueous or organic media sprung out [88]. Functionalization could modulate the band gap of BNNTs in some cases.

Covalent functionalization was achieved through forming acylamino bond between chemical reaction of COCl group of stearoyl chloride or chloroacetyl chloride and amino groups on the BNNTs [89, 90]. It was noteworthy that long alkyl chains introduced by stearoyl chloride induced changes in band structure of BNNTs with reduced band gap.

Another method for covalent functionalization based on boron site at the surface of BNNTs was established via introducing amine groups by using ammonia plasma irradiation or amine-terminated oligomeric poly(ethyleneglycol) [91, 92]. Introduced alien groups at the BNNTs surface enabled facile development of composite structure on this scaffold [93]. Solvothermal treatment of BNNTs in dimethyl sulfoxide (DMSO) solution caused weakened B–N bonds and chemical peeling of BN sheets due to cycloaddition reaction of DMSO and BNNTs [94]. Very recently, hydroxyl groups ($-\text{OH}$) were successfully introduced onto the surface of BNNTs via solvothermal treatment in hydrogen peroxide (Figure 5) [95]. X-ray photoelectron spectroscopy (XPS) studies showed that $-\text{OH}$ groups were bonded to B sites of BNNTs. Hydroxylated BNNTs enabled a series of further chemical reactions based on hydroxyl bonding.

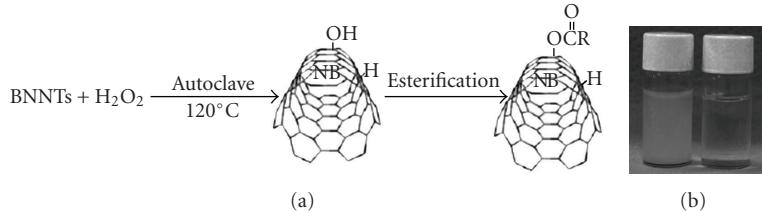


FIGURE 5: Hydroxylated BNNTs [95]. (a) A schematic diagram of hydroxylation of BNNTs. (b) Hydroxylated BNNTs (left) and pristine BNNTs (right) in water. Obviously, hydroxylated BNNTs were soluble in water forming uniform aqueous solution. Reprinted with permission of [95]. Copyright 2009 Wiley InterScience.

BNNTs can be noncovalently functionalized by introducing carboxylate groups from perylene-3,4,9,10-tetracarboxylic acid tetrapotassium salt (PTAS) in aqueous solution [96]. Presumably, π - π interactions between PTAS and nanotubes played a key role in this process. B-C-N/BN coaxial nanotubes with *p*-type semiconducting behaviors were then prepared from PTAS-BNNT by vacuum annealing. Polymer-wrapped BNNTs by poly[*m*-phenylenevinylene-*co*-(2,5-diethoxy-*p*-phenylenevinylene)] (PmPV) perfectly dissolved in common organic media were prepared obeying similar mechanism [97]. However, impurities out of BNNTs presented no interaction with PmPV. Consequently, an effective purification method based on a conjugated polymer wrapping was established [98]. Noncovalent coating of the nanotube wall with biomolecules such as salmon DNA and poly-L-lysine were also reported [99, 100]. Lately, stable BNNTs aqueous solution was obtained with the help of ammonium oleate ionic surfactants drove by weak Coulombic attraction [101]. Photoluminescence (PL) analysis showed that the intrinsic optical properties of BNNTs were preserved.

A simplified strategy, where the boron site in BNNTs was referred to as Lewis acid, then enabling reaction with Lewis bases such as trialkylamine and trialkylphosphine, was proposed as an alternative way for soluble BNNTs [102]. Unfortunately, the commonly used routes were found to be exclusively applicable to multiwalled BNNTs. Just several months earlier, soluble single-walled BNNTs in organic solvents were obtained for the first time using quinuclidine molecules as Lewis bases [103]. It was therefore developed to be a versatile strategy for the functionalization of both multiwalled and single-walled BNNTs.

Very recently, systematically theoretical study of functionalization of BNNTs by substituted carbenes (CR_2) where $\text{R} = \text{H}, \text{F}, \text{Cl}, \text{CH}_3, \text{CN}$, and NO_2 was conducted using density functional theory [104, 105]. The electronic properties of functionalized BNNTs by $\text{C}(\text{NO}_2)$ can be violently tuned from large band gap insulators to excellent semiconductors. These calculation results might be useful guide to the ongoing experimental research in this field.

2.3. Doped Boron Nitride Nanotubes. Analogous to CNTs counterpart, BNNTs can be doped with other elements in the hope of novel properties including mainly modified

band gaps. Owing to their similarity in structure, carbon-doped BNNTs were firstly and intensively studied from both theoretical predictions and experiments. Two types of carbon-doped BNNTs, homogeneous B-C-N nanotubes and BN/C heterostructures, are generally considered [106–109]. The carbon nanotubes substitution reaction, AAO-templated route and covalent functionalization as discussed above could well produce carbon-doped BNNTs [57, 96, 110]. However, precise characterization of their fine structures is limited by current instruments [29].

Tang et al. reported CVD synthesis of fluorine-doped BNNTs with a doping concentration of ~5% using BF_3 as reactant [111]. Electron energy loss spectroscopy (EELS) mapping described uniform distribution of F atoms within the nanotubes. The current-voltage (I - V) characteristics demonstrated semiconducting behaviors of BNNTs after being doped with highly electronegative fluorine. Several theoretical studies of fluorine-doped BNNTs were also conducted [112–114].

Si-doped bamboo-like BNNTs were synthesized via catalyst-assisted pyrolysis of a silicon-containing polymeric precursor under N₂ atmosphere [115]. Intense visible PL spectrum showed emission ranging between 500 and 800 nm presumably contributed to Si doping. Very recently, similar structure with Si dopant concentration of 5% was produced using B pieces, BN powder and a piece of Si wafer as reactants (Figure 6) [116]. XPS confirmed the formation of Si–B and Si–N bonding. The valence band analysis showed that the band gap of BNNTs were reduced by ~1.7 ev after Si doping.

Apart from light elements, heavy elements doped BNNTs were investigated theoretically and experimentally. BN nanobamboos doped with Eu were prepared through annealing of ball milled mixture of B and Eu powder [117]. Cathodoluminescence (CL) spectrum showed a new strong band emission centered at 490 nm, indicating its possible application as a nanoscale visible-light source. Recently, Cu-doped BNNTs self-assembling into interesting microbelts were synthesized using nanoscale Cu powders as catalyst [118]. The Cu doping induced a strong and red-light emission at 695 nm in BNNTs.

Other elements, such as O, Pt, Be, Mn, and Cr [119–122] were theoretically studied to be doped into BNNTs expecting modified transport and magnetic properties, which needed further experimental investigation.

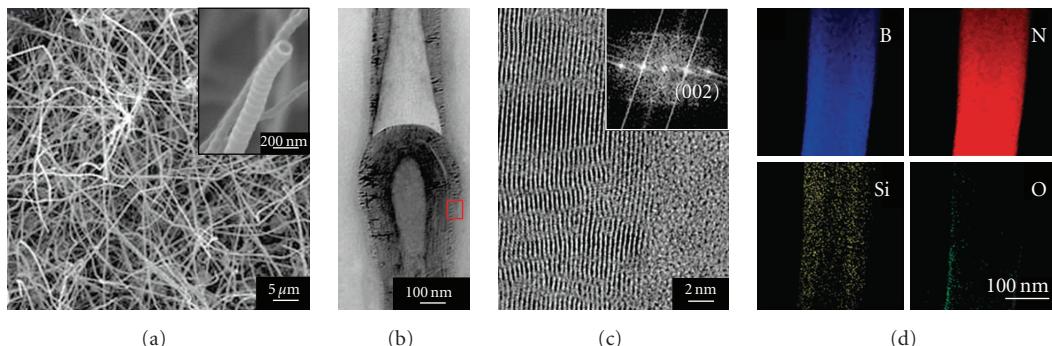


FIGURE 6: Si-doped BNNTs [116]. (a) SEM image of Si-doped BNNTs with worm-like (inset in (a)) or bamboo-like (b) structures. Lattice-resolved TEM image for the edges of the wall layers with its corresponding FFT ED pattern (inset in (c)) showed the (002) basal planes aligned along the tube axis with high crystallinity. EELS mapping of B, N, Si, and O atoms (d) demonstrated the homogeneous distribution of Si and the thin oxide outlayers. Reprinted with permission of [116]. Copyright 2009 American Chemical Society.

3. Boron Nitride Nanowires

Apart from nanotubes, boron nitride nanowires (BNNWs) are another important member of 1D boron nitride nanomaterials. They can act as insulating components in nanoscale devices. Compared with BNNTs, there are only several reports describing the synthesis of BNNWs. Maybe due to some aspects of BNNWs and BNNTs in common, such as, microscopic morphology and growth direction, BNNWs can be synthesized using slightly modified routes for BNNTs. Huo et al. prepared well crystalline BNNWs through the reaction of homemade B-rich FeB nanoparticles with the mixed nitrogen and ammonia at 1100°C [123]. While using the same preparation procedure except for the dramatically reduced boron content in the FeB precursor, bamboo-like BNNTs were obtained [124]. Recently, selective synthesis of boron nitride nanotubes, nanobamboos and platelet nanowires was implemented using a floating catalyst CVD method through different catalysts and reaction temperature [125]. More recently, similar selective synthesis of polycrystalline BNNWs was achieved by annealing a stainless-steel substrate primarily immersed in homemade B nitrate solution under the atmosphere of nitrogen and hydrogen [38].

Moreover, new synthetic systems were developed exclusively for BNNWs. Pure BNNWs with a uniform diameter of 20 nm were prepared through deposition of boron triiodide (BI_3) on a Si (100) substrate under an ammonia atmosphere (Figure 7) [6]. The as-obtained product possessed a disordered turbostratic structure maybe due to the low deposition temperature (1100°C). Crystalline BNNWs were also prepared by heating milled mixture of ZnO and B powder on stainless-steel foils under N_2/H_2 flow [126]. PL spectrum of the product showed a new emission band at 728 nm. Presumably, it was caused by the defect-trapped states in the BNNWs.

4. Other 1D Boron Nitride Nanomaterials

Compared with BN nanotubes and nanowires, there are only a few reports on other 1D BN nanomaterials including nanoribbons, nanorods and nanofibers.

In view of the importance of nanoribbons as an excellent component in electric circuits and devices, BN nanoribbons received extensive research from theory to experiment. Unfortunately, most of the researchs still remained theoretic [127, 128]. The only relevant experimental report about BN nanoribbons was that using homemade B-N-O-Fe as precursor and ZnS nanoribbon as template hollow BN nanoribbons were obtained after evaporation of the template [7]. An extraordinary ultraviolet CL emission at 5.33 ev was found, which was promising in optoelectronic applications especially in ultraviolet region (Figure 8). Actually, this is not the typical model of BN nanoribbons.

BN nanorods or, more strictly, shortened nanowires with a aspect ratio of ~100 were synthesized by nitridation of B_4C powders in nitrogen gas on nitrate-coated Si (100) substrate at 1300°C [9]. The crystalline nanorods presented conical stacking along the nanorod axis. Subsequent field-emission characterization showed that electrons were emitted from both the tips and side surface of the nanorods [129]. Polyhedral BN nanorods with ordered or defective tips were also reported [130].

As far as BN fibers are concerned, most of the methods used borazine-based polymeric precursors to produce boron nitride micrometer-scale ceramic fibers [131–134], which was of striking significance in engineering applications. Ma et al. synthesized crystalline BN nanofibers with the (002) basal planes unusually perpendicular to the fiber axis via pyrolysis of BN, B_2O_3 and B mixed powders under N_2 atmosphere [135]. These nanofibers exhibited a hydrogen storage capability of 2.9 wt.% under ~10 MPa at room temperature. Recently, through nitridation of B_2O_3 -coated polyacrylonitrile (PAN) template fibers with NH_3 , continuous BN nanofibers were successfully produced [8]. The group also synthesized aligned BN nanofibers in bulk by a more simple nitridation process of dissolved B_2O_3 and polyvinylbutyral (PVB) in ethanol [136]. The as-obtained BN nanofibers were crystalline with (002) planes parallel to the fiber axis analogous to BNNTs.

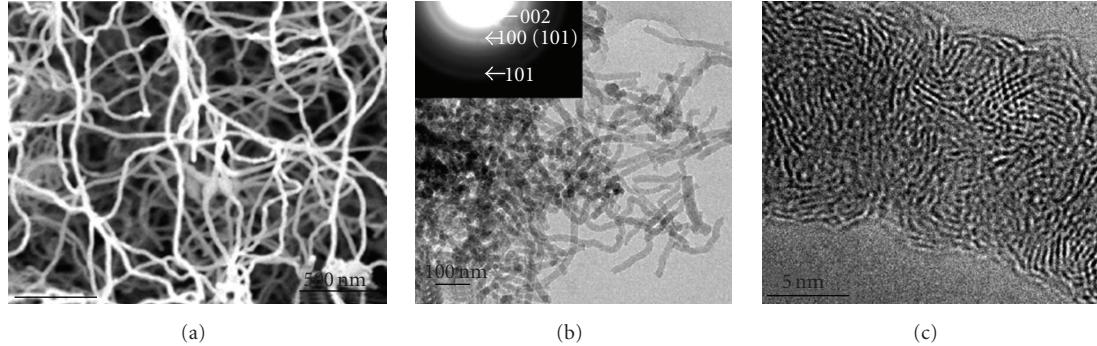


FIGURE 7: BNNWs synthesized from BI_3 [6]. (a) A high magnification SEM image of BNNWs with uniform diameters. (b) TEM and ED pattern (inset) of BNNWs showing high purity. (c) HRTEM of a nanowire exhibiting disordered turbostratic structure. Reprinted with permission of [6]. Copyright 2006 Institute of Physics.

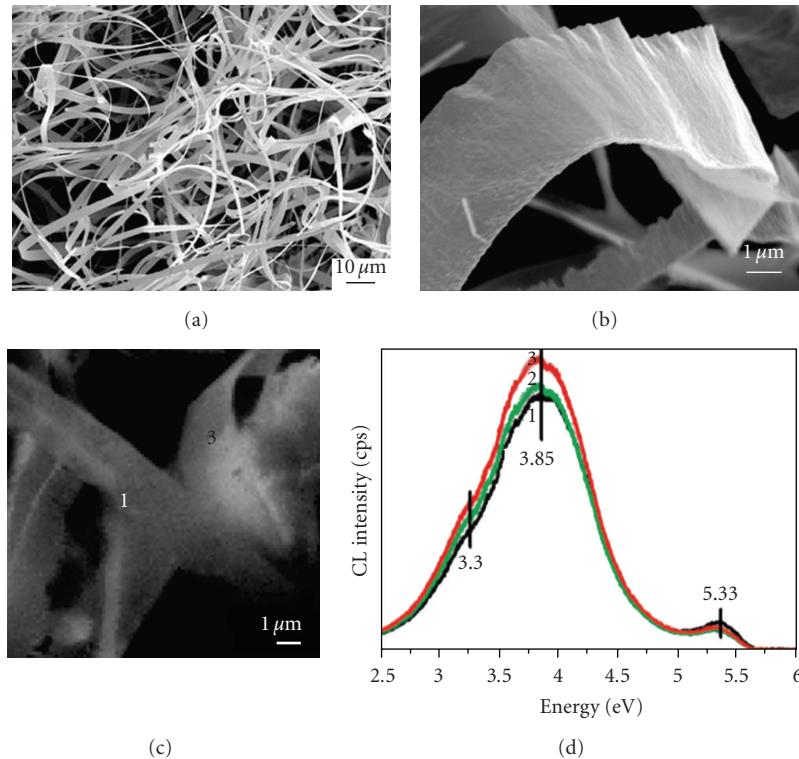


FIGURE 8: BN nanoribbons prepared by using ZnS nanoribbon as template [7]. Low (a) and high (b) magnification SEM images of BN nanoribbons showing their hollow characteristics. (c) CL image at 336 nm showing the uniform contrast across the nanoribbons. (d) CL spectra recorded from the three sites in (c) at 20 K. An ultraviolet emission at 5.33 eV was found. Reprinted with permission of [7]. Copyright 2008 American Chemical Society.

5. Device and Engineering Applications

As to device application, nanotubes and nanowires have been the research focus in this field due to their fascinating electrical, magnetic and transport properties induced by well-known quantum confinement effects at the nanoscale. For the fabrication of devices, understanding of the electronic structures of 1D BN nanomaterials can not be sufficient enough. Due to the insulating nature, much effort

has been devoted to BN nanotubes and nanowires with tuned band structure from insulators to semiconductors, which can be defined as “bandgap engineering”. As demonstrated above, doping with C, Si, F, and so forth, and covalent functionalization can lead to BNNTs with *n*-type or *p*-type semiconducting features [137]. Recently, the effects of isotope (^{10}B) substitution and different tube diameters on the band gap of BNNTs were systematically studied [138, 139].

In addition to excellent thermal stability below 800°C [140], thermal conductivity and mechanical properties of BNNTs was evaluated theoretically and measured experimentally. The results showed that BNNTs possess high thermal conductivity parallel to the tube axis, which is close to that of CNTs [141]. Moreover, the axial Young's modulus was measured up to 1.22 ± 0.24 TPa, which is maybe the highest value of other known insulating materials [142]. These alluring performances of BNNTs endowed them with enhanced fillers in polymer or ceramics matrix [143–146]. Previously, the boron nitride-filled polymer composites were fabricated using boron nitride powers rather than nanotubes as fillers aiming for high thermal conductivity [147–150]. Just in the last year, Zhi et al. reported the fabrication of a series of BNNT-polymer composites including poly(methyl methacrylate) (PMMA), polystyrene (PS), poly(vinyl butyral) (PVB), and poly(ethylene vinyl alcohol) (PEVA) with high BNNT fractions up to 18–37 wt.% through a simple solution method [144]. These composites exhibited improved performance including high thermal conductivity, low coefficient of thermal expansion (CTE) and insulating properties (Figure 9). Engineering ceramics, for example, Al_2O_3 , Si_3N_4 , with reinforced superplasticity by adding BNNTs was studied by the same group [145]. It was found that even a low addition concentration of BNNTs (0.5 wt.%) can remarkably improve the high-temperature superplastic deformation of engineering ceramics. All these fruitful explorations in the lab can undoubtedly extend the industrial applications of BNNTs in engineering field.

Continuous BN fibers of distinctive scientific and technological significance can be used as insulating fibrous reinforcements within ceramic composite in replacement of carbon fibers for use at high temperature in rigorous environment. The decent mechanical performance of BN fibers can be assured by well crystallinity and preferential orientation of the BN crystallites in the direction parallel to the fiber axis [151]. Polybutylene terephthalate (PBT), a widely used plastic as an insulator in the electrical and electronics industries, was reinforced in thermal conductivity by introducing carbon fibers. However, this led to synchronous increased electrical conductivity of PBT. The addition of BN fibers into the present composite was found to effectively reduce its electrical conductivity. Moreover, better tensile properties and processability of the hybrid composite with combined carbon and BN fibers was obtained [152].

One-dimensional materials offer interesting gas adsorption properties, specially, hydrogen storage. CNTs were confirmed to possess the hydrogen uptake of up to 20 wt.%, although the reported value fluctuated markedly [153]. Boron nitride nanomaterials with uniform electrical properties were anticipated to exhibit good stability and high storage capacity. Ma et al. described for the first time the hydrogen uptake of multiwalled BNNTs as 1.8–2.6 wt.% under ~ 10 MPa at room temperature [154]. Surface-modified BNNTs by SO_2 with high surface area was measured to have hydrogen storage of 1.2 wt.% at 77 K [69]. Collapsed BNNTs with increased specific surface area of $789.1 \text{ m}^2/\text{g}$ displayed enhanced hydrogen storage of up to 4.2 wt.% under ~ 10 MPa at ambient temperature, the highest value

for BNNTs reported up to now [155]. This significant increase was in fairly good agreement with the theoretical predication that high-surface-area BNNTs with ordered pore structure may be beneficial to the increase in hydrogen storage [156]. Another concern stemmed from the used Pt nanoparticles existing in the final product. Both theoretical calculations and electrochemical hydrogen storage study proposed that hybrid structure of BNNTs and hydrogen-adsorbing metallic nanoclusters may lead to improved hydrogen storage performance [157–159]. Another solution can be the usage of carbon-doped BNNTs as theoretically calculated [160]. On the other hand, BN nanofibers can also adsorb hydrogen with storage capacity of 2.9 wt.% under ~ 10 MPa at room temperature as mentioned [135].

Recently, particularly in the last couples of years, BNNTs was emerging as innovative tool for nanomedicine [161], which was not discussed previously. Their counterpart, carbon nanotubes, has showed huge potential in this field and was investigated widely and intensively. However, little exploitation was made to BNNTs due to their poor dispersivity and unconfirmed biocompatibility. In 2002, Zhi et al. attempted primarily to check the compatibility of BNNTs and biomacromolecules by immobilizing ferritin protein onto BNNTs [162]. After long-time stirring, they observed the success immobilization, indicating a natural affinity of a protein to BNNTs. If BNNTs were noncovalently functionalized by a complicated ester molecule, the immobilization can be done more efficiently with shortened stirring time. Very recently, several studies of their biocompatibility and interaction with living cells were addressed. Chen et al. verified experimentally the noncytotoxicity of BNNTs with high purity and quality through culturing HEK 293 cells with BNNTs [163]. Fluorescence microscopy analysis revealed that BNNTs coated by glycodendrimer with amphipathic dendritic structure can be directly bound to cell surfaces without causing any cellular toxicity (Figure 10). Their usage as cell delivery agents similar to carbon nanotubes was also confirmed. Another study further verified the benign cytocompatibility of BNNTs on human neuroblastoma cells for the first time [164]. BNNTs functionalized by Poly-L-lysine (PLL) can be used as nanotools to enable cell electroporation with low electric fields in an *in vitro* test [165]. Ciofani et al. studied the feasibility of using PLL coated BNNTs as nanovectors in boron neutron capture therapy (BNCT), a treatment method for several violent cancers, with folic acid as selective tumor targeting ligand [100]. These experimental observations created new applications of BNNTs in cell therapy, drug delivery, and other biomedical fields.

Other promising applications in field-emission devices, ultraviolet lasers, and insulating nanocables (e.g., AlN-BN, GaN-BN, SiC-BN) were also envisioned and investigated, which could be found elsewhere [29, 166].

6. Perspectives and Conclusions

Over the last two decades, synthesis, properties and applications of 1D BN nanomaterials have been exploited and developed as above reviewed.

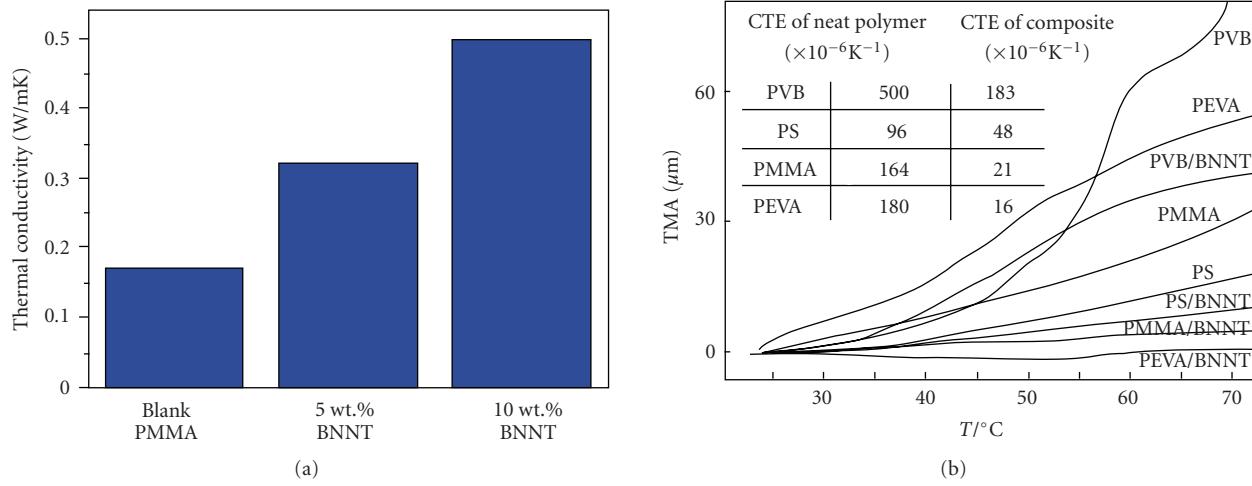


FIGURE 9: (a) A histogram for comparative study of thermal conductivity of BNNT-PMMA composites showing almost 3-fold enhancement with BNNT loading fraction of 10 wt.% [143]. (b) Thermal mechanical analysis (TMA) and CTE improvement of four composites, PVB-BNNT, PS-BNNT, PMMA-BNNT and PEVA-BNNT [144]. It can be found that after introducing BNNTs into the pristine polymers, the CTE values were dramatically decreased. (a) Reprinted with permission of [143]. (b) Reprinted with permission of [144]. Copyright 2009 Wiley InterScience.

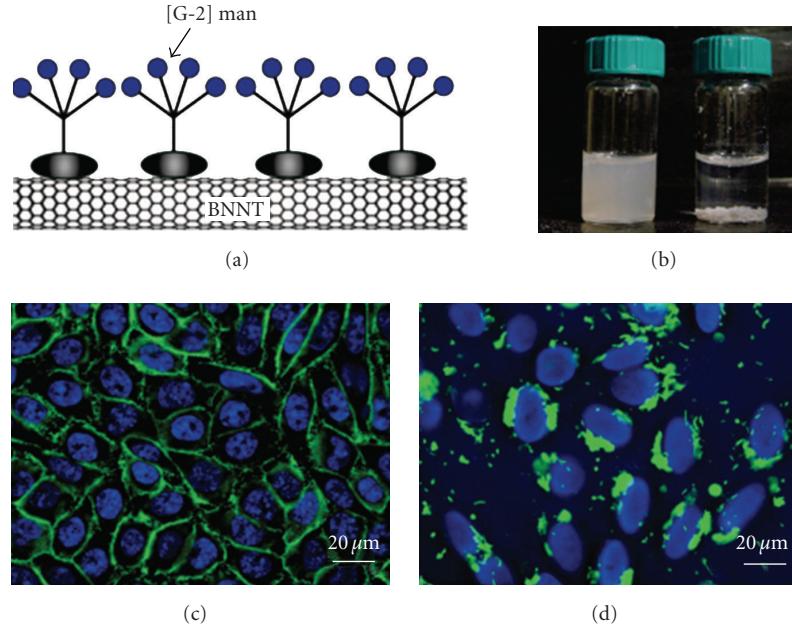


FIGURE 10: BNNTs for biotechnology [163]. (a) Illustration of glycodendrimer coated BNNTs in aqueous media. (b) [G-2] Man-BNNTs (left) were stable in aqueous solution for weeks and uncoated BNNTs (right) sedimentated. (c) [G-2] Man-BNNTs can be directly bound onto the CHO cell surface. (d) Depending on the carrier BNNT, BNNTs coated with FITC-labeled DNA were internalized by the CHO cells. Reprinted with permission of [163]. Copyright 2009 American Chemical Society.

As for BN nanotubes, large-scale production has been achieved. However, much effort is still necessary to be made to synthesis pure nanotubes with homogeneous structure. Alternatively, development of a more efficient purification method is of great significance. Among the mass production of various kinds of BNNTs, single-walled BNNTs is still one of the research focus which are followed by comprehensive property characterization. On the other hand, doped BNNTs

with modulated band structure, which may exhibit novel magnetic and electrical properties, also have large space for further development.

BN nanowires can be studied intensively as building blocks in nanoscale devices. Synthesis of BN nanoribbons still requires unceasing exploitation from both theory and experiment for the future application in nanoelectronics. As a promising candidate for super-lightweight engineering

fibers with high strength and excellent thermal and chemical stability, boron nitride microscale and nanoscale fibers can progress in solving basic theoretical issues and developing cost-effective methods for industrial production.

Overall, one-dimensional BN nanomaterials can be used as promising building blocks concerned about the electronic devices, materials engineering, biomedical engineering and other emergent and important issues.

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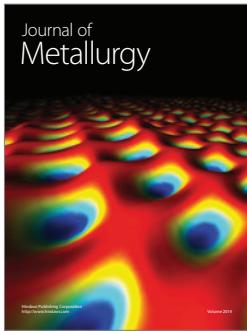


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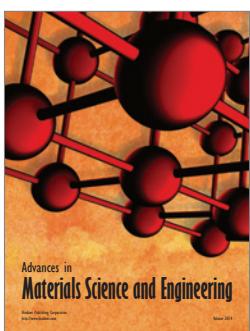
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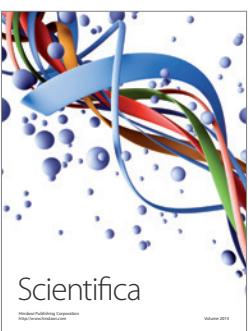
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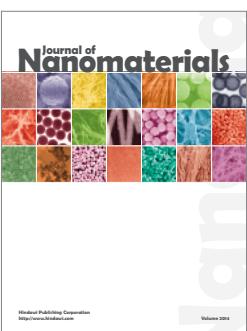
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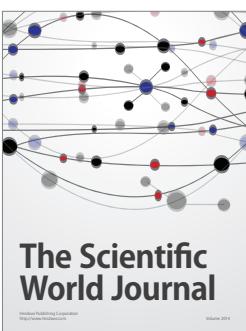
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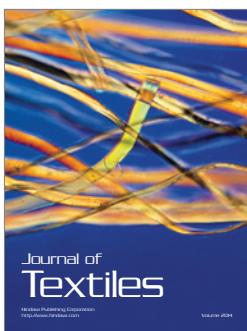
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