



Alkali metal surfactant-facilitated formation of thick boron nitride layers on carbon nanotubes by dip-coating

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ARTICLE INFO

Article history:

Received 4 May 2014

Accepted 6 July 2014

Available online 14 July 2014

Keywords:

Carbon nanotubes

Ceramics

Microstructure

Particles

ABSTRACT

In this paper, we introduced sodium dodecyl benzene sulfonate (SDBS) to the impregnation solution to promote boron nitride (BN) formation on multi-walled carbon nanotubes (MWNTs) by dip-coating. BN coating formation on MWNTs can be greatly improved by SDBS. The resultant BN-coated MWNTs showed increased BN shell thickness, enhanced MWNT dispersion in the impregnation solution, and lower formation temperatures and times (at 1000 °C for 2 h under flowing nitrogen). The shell thickness of the MWNTs may be adjusted from about 10 nm to 25 nm. Electrical property testing also showed that increasing the shell thickness induces higher resistivity (from 0.12 Ω cm to 430 Ω cm) in the MWNTs.

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1. Introduction

Over the last decade, numerous reports on surface-modified carbon nanotubes (CNTs) have been published [1]. Coating of other materials, such as polymers, metals, and inorganic materials, on CNTs to form a core-shell structure has promoted the special properties of CNTs [2,3]. Boron nitride (BN) is a material with excellent chemical resistance and self-lubricating properties. Several researchers have developed approaches by which to coat CNTs with BN. Zhang et al. [4], for example, synthesized BCN nanotubes by laser ablation and reported heterogeneous BN growth on the surface of single-walled CNTs. Yoo et al. [5] also synthesized BN films of 3–10 nm thickness and deposited them on vertically aligned CNTs by reactive sputtering. The methods used by these scholars, however, are generally expensive and their reaction conditions could damage the resultant tube walls.

Some authors have attempted to develop simple means for fabricating BN-coated CNTs. Chen et al. [6] coated BN onto the surface of CNTs, which involved boric acid (H₃BO₃) infiltration followed by NH₃ treatment at 1100–1200 °C. Subsequent research has indicated that the same core-shell structure could be obtained by changing the B or N source [7–9] into B₂O₃, sodium borohydride, ammonia chloride, urea, or melamine, among others.

Despite the amount of research done on the topic, however, the thickness of the final BN shell is always lower than 10 nm and cannot be adjusted [10].

In the present research, the traditional synthesis method in which H₃BO₃ is used as a B source and urea is used as a N source is modified to provide a novel strategy by which to obtain large and thickness-adjustable BN layers on CNT surfaces. In the proposed method, two surfactants [sodium dodecyl benzene sulfonate (SDBS) and polyvinyl pyrrolidone (PVP)] are added to the impregnation solution. The coated CNTs exhibit controlled electrical property, which provide good possibilities and directions for further preparation of devices and composites.

2. Experimental

Acidified MWNTs (MWNT-COOH; weight percentage of carboxyl groups = 1.23 wt%, diameter = 20–40 nm, length = 10–50 μm, aspect ratio > 100; Beijing DK Nano Technology Co., Ltd.) were dispersed in distilled water and ethanol (1:1) solutions of boric acid and urea at predetermined B/N ratios with or without SDBS and PVP-100 (degree of polymerization, 100). Detailed formulations are presented in Table 1. The mixtures were ultrasonically stirred at room temperature for 30 min to disperse agglomerates, dried in air, and then ground to powders with an agate mortar. The powders were loaded into an alumina boat and placed in a horizontal quartz tube furnace at 1000 °C (heating rate, 10 K/min) for 2 h under a high-purity nitrogen flow. The mixtures were cooled to room temperature in the tube furnace, filtered through a 0.22 μm PTFE membrane, and then washed with distilled water to

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remove excess adherent ions. The purified products were dried in a vacuum oven at 60 °C overnight to obtain BN-coated MWNT (BN@MWNT) hybrids. The structure and morphology of the hybrids were determined by X-ray photoelectron spectroscopy (XPS, ESCALAB 250) and transmission electron microscopy (TEM, Tecnai G²20), respectively. The electrical resistivity of the hybrid MWNTs was tested using a four-probe tester (RTS-8; Guangzhou Four Probes Tech Co., Ltd.).

3. Results and discussion

The morphologies of the BN@MWNT hybrids are shown in Fig. 1. The surface of pristine MWNTs was smooth. However, when the MWNTs were coated with BN, the nanotube surface became rough. In the traditional method of fabricating BN-coated CNTs [7], increasing the thickness of the BN layer to over 10 nm by changing the ratios of H₃BO₃ to CO(NH₂)₂ is difficult (Fig. 1a–c). By dipping the MWNTs in the H₃BO₃+CO(NH₂)₂ solution, as in the current study, B and N ions were absorbed on the MWNT surfaces. BN@MWNT hybrids were obtained according to a synthesis method in which two surfactants of different ratios are introduced to improve the dispersion of MWNTs in aqueous solutions [11]. TEM images of these hybrids are shown in Fig. 1d–f. Based on our previous experiment that PVP didn't play obvious effect on the BN thickness in

BN@MWNT after sintering, in the present study, a thick BN layer was achieved via a simple process, and the BN layer thickness may be increased by increasing the SDBS content in the impregnation solution. The thickness of the BN coating layer on the surface of the MWNTs prevented clear viewing of the MWNT core, especially as seen in Fig. 1f. At the same time, the thick BN layer on MWNTs is not so smooth, and is not so uniform in thickness. The reason maybe caused by the nonuniform absorption of SDBS on MWNTs' surface, in which the BO₃[−] and NH₄⁺ dispersion on SDBS' alkyl chains are more uneven than that of the MWNT surface. To the best of our knowledge, large and thickness-adjustable BN layers on CNT surfaces have yet to be reported. The hybrids featured insulative external shells and high-specific conductance inner cores; these characteristics promote the thermal conductivity of the hybrid CNTs.

XPS spectra of BN@MWNT-5 and the raw material MWNT-COOH are shown in Fig. 2a; in this figure, photoelectron peaks [12] from B 1s, N 1s, C 1s, and O 1s were clearly recognizable in the BN@MWNT-5 spectrum and only C 1s and O 1s peaks were detectable in the MWNT-COOH spectrum. The oxygen peak observed may be attributed to O₂, CO₂, and H₂O absorption by the test samples [13]. The intense B 1s component at 190 eV (Fig. 2b) can be fitted to two sub-peaks of 190.3 and 191.6 eV; these peaks may be assigned to B–N [14] and B–O [15] bonds formed from the small amount of available Na₃B₃O₅. The N 1s peak can also be fitted to two sub peaks at 397.9 and 398.8 eV (Fig. 2c), which could be assigned to N–B [13,16] and N–H [17] bonds obtained from the incomplete decomposition of urea. These results indicate the presence of BN on the MWNT surface.

Addition of SDBS not only helped disperse MWNTs in the aqueous suspension but also contributed to BO₃[−] and NH₄⁺ adsorption on the MWNT surface. The major mechanism for adsorption is shown in Fig. 3. Several papers have reported that SDBS is more effective than other surfactants, such as SDS, for dispersing MWNTs [11,18,19] because of the conjugate structure formed between the benzene ring of the former and the sp²-hybridized carbon hexagonal ring of the MWNTs. As SDBS is stably absorbed on the MWNT surface, more BO₃[−] and NH₄⁺ molecules can adhere to the surface of the MWNTs. In other words, the

Table 1
Composition of the BN@MWNT hybrids.

Samples	H ₃ BO ₃ /CO(NH ₂) ₂ –(mol/mol)	PVP (mg)	SDBS (mg)	MWNT-COOH (mg)
BN@MWNT-1	1/3	–	–	200
BN@MWNT-2	1/6	–	–	200
BN@MWNT-3	1/12	–	–	200
BN@MWNT-4	1/6	50	50	200
BN@MWNT-5	1/6	25	75	200
BN@MWNT-6	1/6	–	100	200

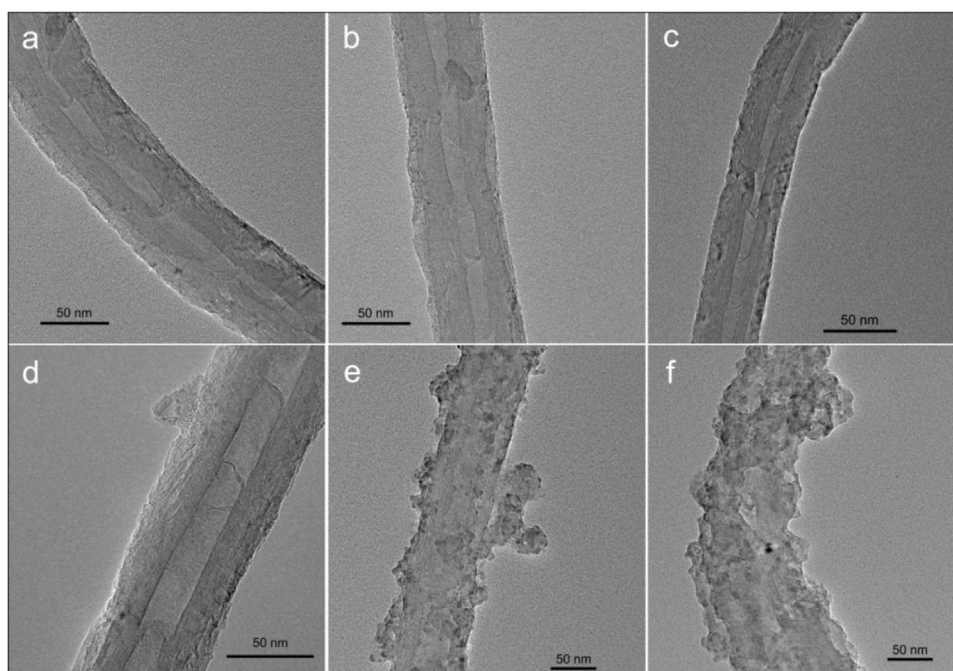


Fig. 1. TEM images of (a) BN@MWNT-1, (b) BN@MWNT-2, (c) BN@MWNT-3, (d) BN@MWNT-4, (e) BN@MWNT-5, and (f) BN@MWNT-6.

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