Available online at www.sciencedirect.com

ScienceDirect

Ceramics International 41 (2015) 4544–4548

CERAMICS INTERNATIONAL

www.elsevier.com/locate/ceramint

Synthesis of highly crystalline multilayers structures of ^{10}BNNTs as a potential neutron sensing element

Pervaiz Ahmad, Mayeen Uddin Khandaker*, Yusoff Mohd Amin

Department of Physics, Faculty of Science, University of Malaya, 50603 Kuala Lumpur, Malaysia

Received 15 November 2014; received in revised form 26 November 2014; accepted 27 November 2014
Available online 6 December 2014

Abstract

Highly crystalline multilayers structures of ^{10}B-enriched boron nitride nanotubes (^{10}BNNTs) are synthesized via a simple technique at 1200 °C. Field emission scanning electron microscopy images show randomly aligned ^{10}BNNTs with some cotton like morphologies. Transmission electron microscopy indicates highly crystalline nature of the BNNTs with internal bamboo-like structures. X-ray photon spectroscopy spectrum confirms Boron and Nitrogen elemental components of ^{10}BNNTs, whereas Raman spectroscopy reports a peak at 1390 (cm^{-1}) that relates to E_{2g} mode of ^{10}BNNTs. The synthesized ^{10}BNNTs can effectively be used as a neutron sensing element in a solid state neutron detector with a 100% suggested efficiency.

Keywords: BNNTs; ^{10}B; ^{10}BNNTs; Synthesis

1. Introduction

Hexagonal boron nitride (h-BN) is an important material for different modern and microelectronic devices. It is a wide band gap semiconductor [1–3] with a direct band gap of 5.97 eV [4]. Structure-wise, h-BN is identical to graphite, however, the carbons atoms are replaced by alternative boron and nitrogen atoms in h-BN [4]. Natural boron (B) in h-BN is found to contain ~20% of ^{10}B and ~80% of ^{11}B [5]. Thus the isotopic ratio of ^{10}B and ^{11}B in h-BN and other B-based compounds can significantly alter their physical properties [6]. The rolled up sheets of h-BN with diameter in the nanoscale range constitute the tubular structures of h-BN called boron nitride nanotubes (BNNTs). Theoretically, BNNTs were predicted in 1994 [7] and experimentally synthesized in 1995 [8]. Unlike carbon nanotubes (CNTs), BNNTs are the large band gap semiconductor with properties independent of diameter or helicity [9,10]. These properties have made it a very important material for different applications in the field of biomedical [11–13], microelectronic mechanical system (MEMS) [14] and solid state neutron detectors [15].

BNNTs have been successfully synthesized by various researchers via their own designed experimental setup and precursors. On this basis, all the techniques used for the BNNTs synthesis were named as: arc-discharge, laser ablation, template synthesis, autoclave, ball milling and chemical vapor deposition (CVD) etc. [16]. Some of the above techniques have also been employed for the synthesis of ^{10}BNNTs. Han et al. [5], first synthesized ^{10}BNNTs via CNT-substitution reaction. Commercially available 99% ^{10}B enriched $^{10}B_2O_3$ is heated in the presence of N_2 flow at 1580 °C. During the reaction molybdenum oxide is used as a promoters with MW-CNTs and $^{10}B_2O_3$. The final product is then further heated at 650 °C to separate carbon impurities. Thus the overall process is not only a bit complicated, lengthly and required higher temperature but also contained impurities [5]. Tang et al. [17] and Zhi et al. [1] introduced their own designed experimental procedures and synthesized high quality BNNTs. Their developed technique [1,17] is further employed by Han et al. [18] for the synthesis of ^{10}BNNTs with ^{10}B, MgO

*Corresponding author. Tel.: +601115402880; fax: +60379674146.
E-mail addresses: mu_khandaker@yahoo.com, mu_khandaker@um.edu.my (M.U. Khandaker).

http://dx.doi.org/10.1016/j.ceramint.2014.11.150
0272-8842/

and SnO as precursors. Beside high temperature and impurities in the final product, the as-used methods were difficult to follow not only because of their complex, lengthy or time consuming procedures but also due to the high prices of as-used experimental setup and its other accessories.

Previously, we studied [19] some of the most prominently used experimental setup and analyzed it for further simplifications [20–25]. In the light of their work, the system designed is used for the synthesis of BNNTs in the presence of Ar gas as a reaction atmosphere and B, MgO and γ-Fe_2O_3 as precursors. By using this technique, high quality of BNNTs were easily synthesized at 1200 °C [19,26]. Han et al. [18] synthesized BNNTs from B, MgO and SnO as precursors. The same experimental conditions were used for the synthesis of ^{10}BNNTs except B in the precursors was replaced by ^{10}B [18]. The same idea has been utilized for the synthesis of ^{10}BNNTs in the present study. The detail of all the efforts done in this regard is fully mentioned in the next section.

2. Experimental details

The methos used for the synthesis of ^{10}BNNTs is similar to our previously reported method for BNNTs [19,26] except B powder is replaced by ^{10}B powder. In short, a 2:1:1 ratio of ^{10}B, MgO and γ-Fe_2O_3 with a total weight of 100 mg is mixed in alumina boat [27] with a conventional stirrer. A few Si substrates are placed at the top of the boat. The boat is then slowly pushed inside one-end close quartz tube. Afterwards, the tube is moved into the chamber of horizontal quartz tube furnace for further processs. Before the experimental run, the ceal closed chamber is flushed with Ar gas to remove the oxygen and dust particles from the system. The Ar gas flow is maintained at a rate of 150–200 sccm and the system is heated up to 1200 °C. When the temperature is reached to 1200 °C, Ar gas flow is stopped and NH_3 gas is introduced in to the system with the same flow rate of 150–200 sccm. The system is maintained in such a condition for 1 h. At the end of 1 h, the flow of NH_3 gas is stopped. Subsequently, the system is slowly brought to room temperature. At room temperature white colour ^{10}BNNTs can be seen deposited at the walls of alumina boat and Si-substrate, as shown in Fig. S1 (Supporting materials). The ^{10}BNNTs sample thus synthesized is characterized by: (1) Scanning Electron Microscope (Model: Zeiss Supra 55 VP) to analyze the morphology and size of the tubes, (2) Transmission Electron Microscope (Model: Zeiss Libra 200FE), to analyze the tubular structure, interlayer spacing and crystallinity, (3) X-ray Photon Spectroscopy (Model: Thermo Scientific K-Alpha) to find out the elemental composition, (4) Raman Spectroscopy (Model: Horiba Jobin Yvon HR800) to find out the crystallographic structure and crystallinity.

3. Results and discussion

Fig. 1 shows lower magnification FESEM micrograph of the ^{10}BNNTs synthesized in the present study. The micrograph shows randomly aligned ^{10}BNNTs along with some cotton like morphologies. Most of these morphologies are stuck with the

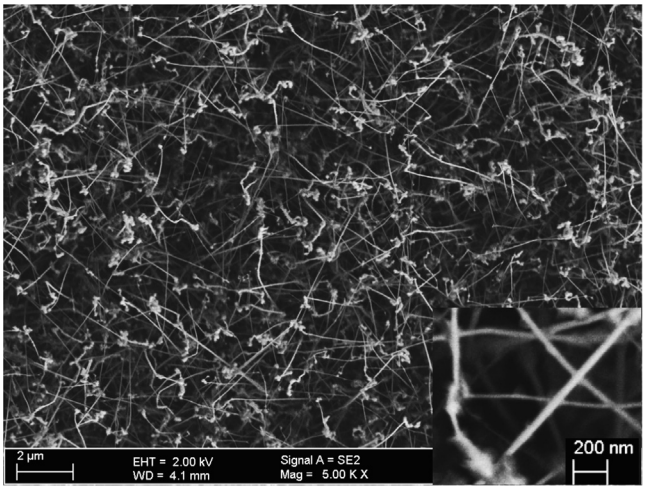

Fig. 1. Lower magnification FESEM micrograph shows randomly aligned ^{10}BNNTs along with some cotton like morphologies mostly stuck with their curves end. The inset magnified image gives a more clear view of the same ^{10}BNNTs.

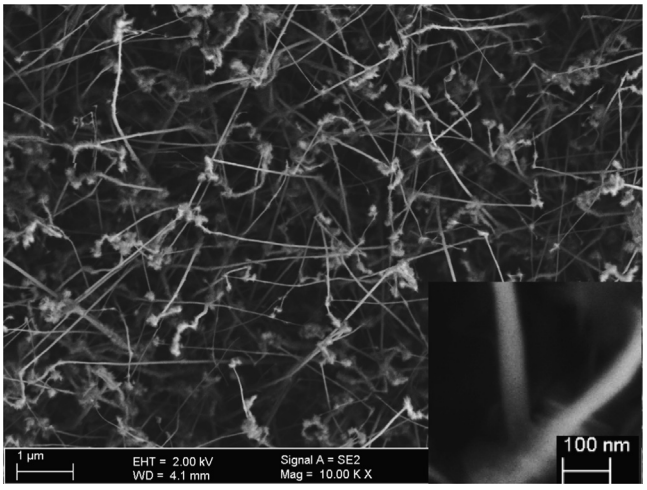

Fig. 2. High magnification FESEM micrograph displays randomly aligned ^{10}BNNTs along with some cotton like morphologies mostly stuck with their curves end. The inset highly magnified image gives a more clear view of the same ^{10}BNNTs.

curves end of the ^{10}BNNTs. These white species with ^{10}BNNTs look like raw cottons packs held with their plants. The inset magnified image on the bottom right hand corner gives a more clear view of the same ^{10}BNNTs.

Fig. 2 shows high magnification FESEM micrograph that further clarify the morphology of the randomly aligned ^{10}BNNTs and cotton like morphologies, previously shown in Fig. 1. The highly magnified inset image on the bottom right hand corner of Fig. 2 shows straight ^{10}BNNTs with cotton like species stuck at their bottom. It has been observed that the diameter of the tubes varies continuously throughout the sample. The scale in the inset image (on the bottom right hand corner of Fig. 2) indicates that the diameter of the tubes can be found in the range of 30–80 nm. The accurately calculated diameter of a few ^{10}BNNTs (in the range of 40 nm to 66 nm) with the help of an FESEM application is