

Full Length Article

Synthesis, structure and antioxidant performance of boron nitride (hexagonal) layers coating on carbon nanotubes (multi-walled)

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ABSTRACT

Carbon nanotube/Boron nitride (CNT/BN) nanocomposites were performed by a facile method with boric acid (H_3BO_3) and melamine ($C_3H_6N_6$). Uniform boron nitride (BN) precursor deposition films were deposited onto the surface of multiwall carbon nanotubes (MWCNTs), which involves attachment of the precursor and subsequent nitridation in ammonia atmosphere at 1050 °C. The qualitative and quantitative analysis of X-ray diffraction (XRD) on the crystal structure and phase composition. Scanning electron microscope (SEM) and transmission electron microscope (TEM) were used to characterize the material surface morphology and coating structure. Chemical composition analysis of surface and chemical bonds formed by X-ray photoelectron spectroscopy (XPS) and Fourier transform infrared Spectroscopy (FTIR) confirmed as well as the oxidation resistance under high temperature by thermal gravimetric analysis and differential thermal analysis (TG-DTA). The results indicate that the boron nitride coating on carbon nanotubes composite has been synthesized successfully by this method and greatly improves the antioxidant performance of carbon nanotubes compared with the original carbon nanotubes (690 °C, compared to 550 °C of original carbon nanotubes), which provides a potential application at high temperatures for carbon nanotube materials.

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1. Introduction

Due to its unique chirality, carbon nanotubes can be caused widely attention and application possessing the properties of metal and semiconductor, the excellent conduction, thermal conductivity of metal materials, good mechanical and wear resistance [1–4]. Thus, the material emerges as a good alternative for function-related materials has attracted widespread concerns and applications [5–8]. However, the oxidative properties of high temperature (above 500 °C) limits the practical application of carbon nanotubes in the process of development. The boron nitride has low density, high oxidation resistance, high thermal conductivity and stability, which is coated on the surface can make up the defects of carbon nanotubes. The composite possesses not only the advantages of carbon nanotubes, but also good mechanical properties of boron nitride materials, meanwhile avoiding the high temperature oxidation a good way [9–13]. Based on the following research progress, it is of great guiding significance for the research of the subject:

Chemical vapor deposition (CVD): BCl_3 and N_2 are sources of boron and nitrogen, respectively. The composite is synthesized

on a single CNT sample growing BN film to improve the field emission characteristics by plasma assisted chemical vapor deposition (PACVD) method under 650 °C [14–16].

Pulsed-DC magnetron sputtering: With ion assisted, rapid rate and high efficiency pulse DC magnetron sputtering technology, sputtering carbide boron (B_4C) target in argon and nitrogen plasma to deposit boron nitride (BN:C) film [17].

SiC (Si_3N_4) coating mechanism: The SiC was produced simultaneously in the SiO and B_2O_2 steam with boron and silicon dioxide as original material and Ni-C nanoparticles as the catalyst, which is by gas-liquid-solid growth mechanism of boron nitride coated sic nanowires [18–20].

Polyacrylonitrile (PAN) carbon fiber: Boron nitride (BN) films were formed on the surface of chemically activated polyacrylonitrile (PAN) carbon fibers by dip coating in a saturated boric acid solution and sintering at 1200 °C in nitrogen atmosphere [3].

However, the above methods are operated complicately, higher requirements for equipment, expensive and difficult to control the coating state of boron nitride, which are not conducive to the industrial application. It is a kind of simple technology, convenient operation and encapsulation effective method of boron nitride coating on carbon nanotubes that carbon nanotubes as material by boric acid (H_3BO_3) and melamine ($C_3H_6N_6$) in ammonia (NH_3)

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atmosphere under high temperature (1050 °C). The properties of the composite is analysed by XRD, SEM, TEM, XPS, FTIR, and TG-DTA. The antioxidant capacity of the composite has been greatly improved, which provides guidance for the application of carbon nanotubes at high temperature.

2. Experimental

2.1. Materials

As the original material of melamine and boric acid were supplied by Tianjin Guangfu Fine Chemical Research Institute and Tianjin Wind Boat Chemical Reagent Technology Co. Ltd respectively. Polyvinylpyrrolidone (PVP-K30) as a surfactant and agglomerant was purchased from Tianjin Bodi Chemical Co. Ltd. The industrial grade CNTs were purchased from Suzhou Carbon Technology Co., Ltd.

2.2. The synthesis of samples

The concentrated sulfuric acid and concentrated nitric acid are mixed in a ratio of 3:1. The carbon nanotubes is added by ultrasonicing, stirring and drying in vacuum. 1.236 g of boric acid and melamine 1.26 g were dissolved in 25 ml of ethanol. The pre-treated carbon nanotubes of 0.06 g was added in the solution. Then, 1.6 g of PVP-K30 was also added in it. The solution was ultrasoniced for 2 h, stirred for 12 h. filtered, dried in vacuum. The dried ground powder was sintered at a high temperature of 1050 °C for 2 h in an ammonia atmosphere to obtain a coated sample.

2.3. Characterization

X-ray powder diffraction (XRD, Vinci, Germany) patterns were obtained on a Rigaku smartlab diffractometer with Ni-filtered Cu K α radiation at a scanning rate of 8°/min in the 2 θ range from 10° to 90°. The presence of various chemical bonds was measured by Fourier transform infrared Spectroscopy (FTIR, Vector 22, Germany) in the range of 500–4000 cm⁻¹. X-ray photoelectron spectroscopy (XPS, ESCALAB 250Xi, America) was used to characterize its surface chemical composition from the Thermo Fisher Scientific Co. Ltd with a source of monochromatized AlK α . Surface morphology and structure in the films were investigated by scanning electron microscope (SEM, Nova Nano SEM450 and Verious460L, American FEI Company) and transmission electron microscope (TEM, JEM-2010FEF, Japan JEOL). The quantitative analysis of elements was examined by X-ray energy spectrometer (EDAX) simultaneously. The layers on the CNT surface with their uniformity of the product were also characterized by STEM-HAADF (Talos F200X, American FEI Company) with the corresponding elemental maps of C, B and N. Oxidation mechanism of original materials and synthetic samples were measured using thermal gravimetric analysis (TGA) and differential thermal analysis (DTA) instrument (TG-DTA, SDTQ-600, America TA Instruments-water LLC) during rising from room temperature to 900 °C at a rate of 5 °C/min in air atmosphere.

3. Results and discussions

Crystal structure and phase analysis were performed using XRD. Fig. 1 shows the XRD patterns of the (a) h-BN, (b) CNT and (c) CNT/BN. Pattern (a) is obtained from the product of the h-BN, showcasing that the characteristic diffraction peaks of 2 θ = 25.62°, 42.39°, 54.39° and 76.85° correspond to the (0 0 2), (1 0 0), (0 0 4) and (2 2 0) crystallographic planes of h-BN, respectively. Carbon nanotubes (Fig. 1(b)) possesses obvious characteristic diffraction peaks

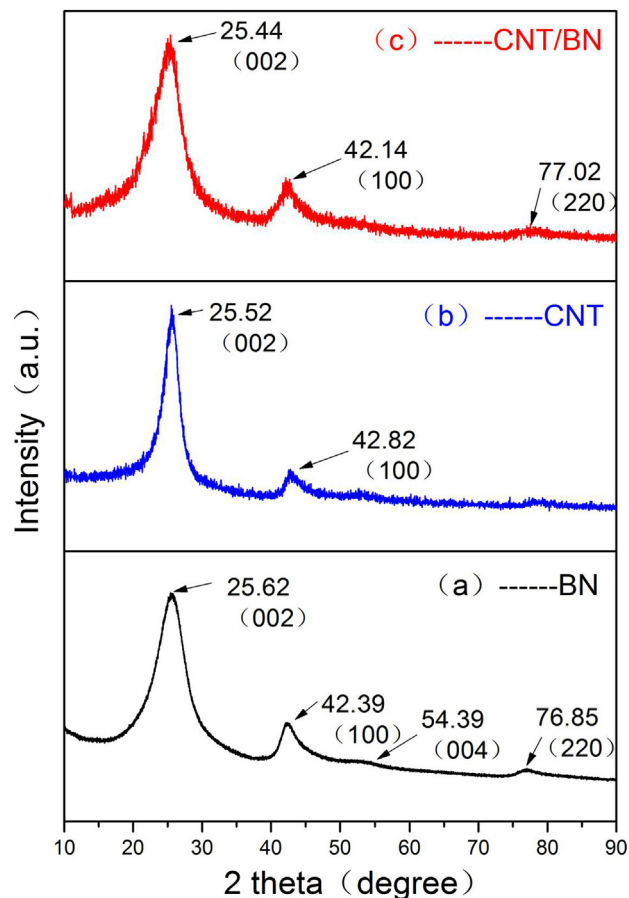


Fig. 1. XRD diffraction pattern of (a) h-BN powder, (b) CNT and (c) CNT/BN.

at 2 θ = 25.52° and 42.82°, corresponding to the (0 0 2) and (1 0 0) crystal plane, respectively. Notably, there are two distinct characteristic diffraction peak at 25.44° and 42.14° from the crystal properties of the coated sample in Fig. 1(c), indicating a little offset of the peak through the comparison. The Pattern (c) also shows a weak characteristic diffraction peak at 2 θ = 77.02°, which can mainly assignable to the crystallographic plane of (2 2 0) [18,21]. The structural similarities between h-BN and carbon enables them integrated with each other so well that it is hardly to identify the diffraction peak between them [10]. Furthermore, the atomic ratio of boron nitride is 1:1 and atomic number adjacent to each other, which also leads to the two characteristics of h-BN diffraction peak close to the carbon nanotubes. As the diffraction peak of mobility and reduction of compound crystallization performance, which can be illustrated the presence of boron nitride coating film in the carbon nanotube surface.

Fig. 2 shows the FTIR spectrum of (a) CNT/BN and (b) h-BN. It shows that both of them have strong absorption peaks around 1380 cm⁻¹ and 800 cm⁻¹, corresponding to the presence of B–N stretching vibration and B–N–B bending vibrations, respectively, which are in good agreement with typical h-BN vibration characteristics. The sharp and strong absorption peak around 3400 cm⁻¹ is derived from the sample surface absorption of the typical –OH stretching vibration. However, it should be noted that a significant decrease in absorption peak intensity in B–N and B–N–B bending vibration of CNT/BN, which can be attributed to the existence of a boron nitride coating [22,23]. In addition, due to the existence of the reaction gas oxygen that a weak absorption peak around 2360 cm⁻¹ indicates the presence of the C=O stretching mode of CO₂, which is consistent with that reported by Bi et al.

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