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Microstructural characterization of alumina-coated multi-walled carbon nanotubes synthesized by hydrothermal crystallization

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ARTICLE INFO

Article history:

Received 21 March 2010

Received in revised form

26 April 2010

Accepted 28 April 2010

Keywords:

Carbon nanotubes

Alumina

Hydrothermal crystallization

Microstructure

ABSTRACT

Multi-walled carbon nanotubes (MWCNTs) were coated with a nano-sized α -alumina polycrystalline layer by hydrothermal crystallization under autoclave conditions followed by manipulative calcination. These nanocomposites were characterized for their microstructure and composition by X-ray diffraction (XRD), energy-dispersive X-ray spectroscopy (EDX), transmission electron microscopy (TEM), high resolution transmission electron microscopy (HRTEM) and scanning electron microscopy (SEM). The results show that the microcrystalline α -alumina layer with the thickness of 1–3 nm was rough and compact, which profited from well adhesion by boehmite sol, the lower temperature for the conversion of the hydroxide to oxide, and the slower speed for the calcination process. In addition, the α -alumina-coated MWCNTs were dispersed uniformly in alumina particles.

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1. Introduction

Since discovered by Iijima [1], carbon nanotubes (CNTs) have been considered the most promising nano-sized materials due to their excellent Young's modulus, good flexibility, low density, very good electrical characteristics and excellent thermal performance [2–5], and have been investigated for devices such as advanced ceramic devices, field-emission displays, scanning probe microscopy tips and microelectronic devices [6,7]. In more recent years, the interest of using CNTs in ceramic composites arises from their formidable mechanical properties [8], and the preponderance of research efforts have focused on the development of nanotube-reinforced ceramic composites. Ideally, the inclusion of CNTs in a ceramic matrix is expected to pin or nail the ceramic particles for improving the mechanical properties compared to the single-phase ceramic material. Actually, the effect of CNTs is restricted by two main defects including lack of uniformity in the CNTs dispersion and weak interfacial connections between CNTs and ceramics [9].

Many attempts have been made to overcome the two main defects. Lupo et al. [10] reported that nanometric zirconia (ZrO_2) particles were grown on the CNTs to offer a ceramic layer by a hydrothermal process using zirconium hydroxide $[Zr(OH)_4nH_2O; n=8-16]$ in the presence of the CNTs. Hernadi et al. [11] prepared multi-walled carbon nanotubes (MWCNTs) decorated with alumina (Al_2O_3) nanoparticles by an impregnation method using

aluminum isopropoxide (AIP) as an inorganic source and decomposed by hydrolysis on the surface of carbon nanotubes. Yang et al. [12] showed that γ - Al_2O_3 layers with thickness of 1–3 nm covered the surface of MWCNTs, which were prepared by chemical precipitation using aluminum nitrate and ammonia as starting materials. Morisada et al. [13,14] showed that the MWCNTs were coated with a nanometric SiC polycrystalline layer by the reaction of SiO (g) and CO (g), which happened in vacuum at 1150 °C with SiO powders vaporized as the silicon source. Increases of microhardness by 20% and toughness by 12.5% were measured comparing SiC-coated MWCNTs-SiC composites with monolithic SiC ceramics. In these experiments the ceramic coatings were used to improve the weak adhesion between CNTs and various ceramics matrixes, and all the methods seemed to obtain a good dispersion of the CNTs.

In order to provide a favourable dispersibility and improve interfacial connections for CNTs in ceramic matrices, in this paper, we report a convenient route to produce α - Al_2O_3 , which coats on the surface of MWCNTs. AIP was used as an inorganic source and hydrolyzed into the aluminum hydroxide ($Al(OH)_3$) sol, which followed dehydration by a hydrothermal process in the presence of MWCNTs. The product was then calcined in a vacuum under control. The processing conditions and microstructure of the nanocomposites are described below.

2. Experimental

The schematic procedure for the fabrication process of α - Al_2O_3 /MWCNTs nanocomposites was shown in Fig. 1. The

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MWCNTs (Sun Nanotech Co Ltd., China) used in the present investigations were fabricated by chemical vapor deposition (CVD) and treated by acidification and activation, which have dimensions of 20–40 nm in diameter, 5–20 μm in length, and the density of 1.8 g/cm^3 . AlIP (Sinopharm Chemical Reagent Co Ltd., China) used as inorganic source was hydrolyzed and peptized in an open-top container at 90 $^\circ\text{C}$ for 6 h. Then nitric acid (HNO_3) (Xi'an Chemical Institute, China) was added to promote peptization. The mixture liquor was refluxed at 100 $^\circ\text{C}$ for 16 h to obtain a clear boehmite sol. The pretreated MWCNTs dispersed by ultrasonic stirring in the form of suspension within ethanol were added to the boehmite sol. The obtained suspension was sealed in a 316-grade stainless steel autoclave and this assembly was kept at 300 $^\circ\text{C}$ for 6 h for the hydrothermal reaction to occur. Finally, the slurry of Al_2O_3 and MWCNTs powders in the autoclave was obtained by exhaust process and dried at 50 $^\circ\text{C}$ in vacuum of 10 Pa. The resulting powders were heated up to 1300 $^\circ\text{C}$ at the calefactive speed of 5 $^\circ\text{C}/\text{min}$ and calcined at 1300 $^\circ\text{C}$ for 1 h in a vacuum of 10^{-3} Pa. The weight content of MWCNTs was controlled as 5%.

The resulting material was a black powder. The processing conditions and microstructural characterization of the powders were described by means of XRD, EDX, TEM, HRTEM and SEM. SEM studies were carried out using a JEOL-JSM6700F operated at low energies between 2 and 5 kV. EDX, TEM and HRTEM

observations were performed with a JEOL-JEM3010 equipped with an energy-dispersive X-ray spectrometer, employing an accelerating voltage of 300 kV. X-ray diffraction (XRD) was recorded on a D/MAX 2250 V X-ray powder diffractometer with $\text{CuK}\alpha$ radiation in the range of $20^\circ < 2\theta < 80^\circ$.

3. Results and discussion

SEM micrographs taken at low magnification give overviews of the composite powders' microstructures which include the powders before the calcination process (Fig. 2a) and after the calcination process (Fig. 2d). The uniformity of these composite powders was revealed from the two micrographs. A close-up of the material in Fig. 2(a,b) shows that there are few eyeable nanotubes which are denoted by arrows. Fig. 2(c) which is an amplificatory micrograph of Fig. 2(b) shows that few nanotubes (showed by arrows) are inserted into the nano-sized particles and the substances (showed by circles) seem to be on one side of the MWCNTs. This guesswork is validated rather from Fig. 2(e,f) which are the feature of Fig. 2(d). Comparing with Fig. 2(b,c), the volume of the particles is shrunk a little and the further nanotubes which are homogeneously distributed within the particles are observed. From Fig. 2(f), the surfaces of the nanotubes seem to have a wrapping layer which is observed using TEM and HRTEM below.

The TEM and HRTEM micrographs of the sample which was prepared by dispersing the particles undergoing calcination process in acetone and then transferring onto a holey carbon are shown in Fig. 3(a,b), respectively. Fig. 3(a) shows that the surface of the nanotube is coated by a rough and compact solid layer, which is testified to include the C, O and Al elements using EDX spectrum, as shown in Fig. 3(c). Combining with the XRD pattern of the sample (Fig. 4, curve b), the coating layer is revealed to be $\alpha\text{-Al}_2\text{O}_3$ crystal layer. Fig. 3(b) shows a representative HRTEM micrograph of the composites, illustrates clearly the interface of the nanotube and the $\alpha\text{-Al}_2\text{O}_3$ crystal layer, and the thickness of the layer is about 1–3 nm. The inner wall of the nanotube which is near the broken opening is also coated by the $\alpha\text{-Al}_2\text{O}_3$ crystal layer. It suggests an alternative route for filling ceramics inside the MWCNTs.

The XRD patterns of the composite powders before the calcination process (curve a) and after the calcination process

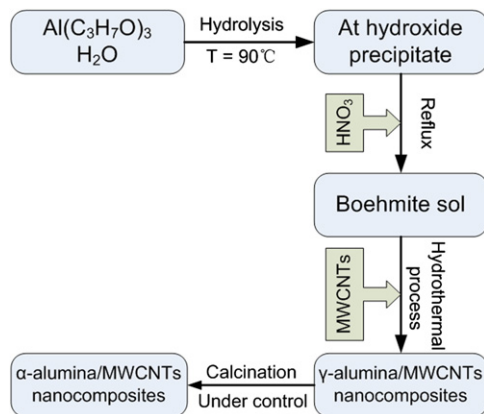


Fig. 1. Schematic diagram for fabrication of α -alumina/MWCNTs nanocomposites.

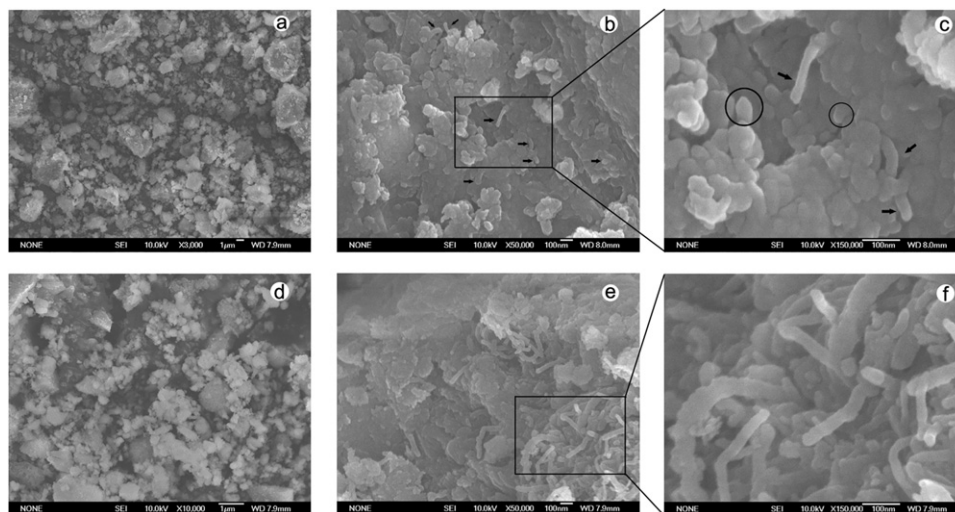


Fig. 2. The SEM micrographs of the alumina/MWCNTs composite powders. (a–c) the powders before the calcination process and (d–f) the powders after the calcination process. (a) and (d) are the overviews with the features (b,c) and (e,f), respectively.

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