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Effect of carbon nanotube addition on friction coefficient of nanotubes/hydroxyapatite composites

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ABSTRACT

Carbon nanotubes/hydroxyapatite (CNTs/HA) composites with different CNTs contents were synthesized by *in situ* method and were characterized by XRD, TEM and Raman spectroscopy. Friction coefficients of the composites were tested using UMT-2 friction tester. Effect of various factors including CNTs content, testing time and applied load on friction coefficient was carried out. Results show that the CNTs/HA composites exhibited lower friction coefficient than pure HA and their friction coefficients decreased with increase of CNTs content from 0 to 20 wt.%. Addition of CNTs in composite is beneficial to increase wear resistance of the CNTs/HA composite and to decrease its friction coefficient.

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1. Introduction

It is well known that hydroxyapatite (HA) is a non-toxicity bioactive material and it is also the model compound for the inorganic component of bone and teeth [1–3]. Recently, HA has attracted considerable attention in some fields like dentin, adsorption and bone due to its excellent bioactivity, biocompatibility and adsorption property [4–7]. However, the intrinsic properties of HA, such as low flexibility and toughness, lack sufficient strength and poor mechanical properties have restricted its potential application [6,8]. In order to overcome these obstacles, in particular, to improve the strength and mechanical property of HA, many efforts on fabrication of the HA-based composites have been carried out [9–14].

Carbon nanotubes (CNTs) with one-dimensional and hollow structure are one of the most fascinating nanomaterials. Since the discovery by Iijima [15], carbon nanotubes have attracted worldwide interests in some areas including materials science [16], electrochemical sensor [17,18], conductivity [19,20], polymer [21], assembly [22] and hydrogen storage [23,24] due to their high mechanical strength [25], excellent thermal conductivity, unique electronic properties [26,27] and thermal stability. These outstanding structural characters make CNTs ideal candidates as reinforcements in composite materials to enhance stiffness and strength [28]. Therefore, the addition of CNTs in various composites has been a hot topic. Recently, the composite materials containing HA in combination with CNTs has attracted worldwide attention [29–31]. Zhao et al. [32] synthesized carbon–hydroxyapatite–hemoglobin nanocomposites by self-assembling technique and the resulting composite exhibit high bioelectrocatalytic activity. The multi-walled carbon nanotube–hydroxyapatite composite prepared by Liu et al. [33] was used as an original adsorbent for Co(II) sorption from aqueous solutions and the experimental results show that the composite has high sorption capacity in Co(II) pollution cleanup. Meng et al. [34] have attempted carbon nanotubes as the reinforcement in HA-based composites to improve its mechanical strength.

Here, we reported the synthesis of the CNTs/HA composites with varying amount of CNTs by *in situ* method. The friction coefficients of the CNTs/HA composites were evaluated by UMT-2 friction tester. In particular, we discuss in detail the effect of several factors including CNTs content, testing time and applied load on friction coefficients of the CNTs/HA composites. For comparison, the friction coefficient of the pure HA was also investigated. We found that the addition of CNTs in composite can decrease the friction coefficient of the composite as compared with the pure HA. The friction coefficient of the composite gradually decreased with the increase of CNTs content in composite and increased with the increase in applied load.

2. Experimental

2.1. Material

Chemicals used in this work, such as anhydrous alcohol (C_2H_5OH), hydrofluoric acid (HF), concentrated nitric acid

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(HNO_3), calcium nitrate ($CaNO_3{\cdot}4H_2O$), ammonium monohydric phosphate {$(NH_4)_2HPO_3$}, ammonia, sodium dodecyl sulfate (SDS) and polyvinyl alcohol (PVA), were of analytical grade. They are purchased from Shanghai Chemical Reagent Corporation, PR China. Carbon nanotubes (CNTs) are made by our laboratory using the chemical vapor deposition (CVD) method with purity >95%.

2.2. Preparation and purification of carbon nanotubes

The details of the CNTs preparation can be found in our previous publication [35]. The purification of the as prepared carbon nanotubes need to be performed before they are used in the preparation of nanotubes/hydroxyapatite composites. In a typical acid treatment, the obtained carbon nanotubes samples were firstly treated by ultrasonication for 1 h with 40% hydrofluoric acid at ambient temperature, and then were refluxed using concentrated nitric acid at 120 °C, designated as CNTs.

2.3. Preparation of carbon nanotubes/hydroxyapatite composites

Preparation of carbon nanotubes/hydroxyapatite composites was carried out *via* an *in situ* method. In a typical preparation process, 0.0205 g of sodium dodecyl sulfate (SDS) was dissolved in 100 ml distilled water. Then, a required amount of CNTs was added into the SDS solution and was sonicated for 1 h to obtain a homogeneous mixture. Thereafter, 0.5 mol/L of $CaNO_3$ solution and 0.5 mol/L of $(NH_4)_2HPO_3$ solution according to the molar ratio of Ca/P = 5:3 were gradually added into the above homogeneous mixture through separate tube pumps at the speed rate of 60 ml/h with continuous stirring at 35 °C. The pH value of the mixture solution was adjusted to about 10 with ammonia solution. After continued stirring for another 6 h, the mixture was aged for 24 h, washed with deionized water for several times till neutral pH to remove the residual NO_3^- and NH_4^+. Finally, the resulting carbon nanotubes/hydroxyapatite composite samples were dried at 80 °C for 24 h in an oven. The obtained samples were denoted as CNTs (x)/HA, where x stands for the mass percentage of CNTs in composite from 2, 5, 10 and 15 to 20%. For comparison, the pure hydroxyapatite was also obtained according to the following reaction equation:

$$10Ca(NO_3)_2{\cdot}4H_2O + 6(NH_4)_2HPO_4 + 8NH_4OH \rightarrow Ca_{10}(PO_4)_6(OH)_2 + 46H_2O + 20NH_4NO_3$$

designated as HA. Then, the mass of HA was calculated according to the mass of calcium nitrate added in the reaction process.

2.4. Characterization

The XRD patterns of samples were recorded on a Rigaku D/MAX 2500PC powder X-ray diffraction instrument with Cu Kα radiation (λ = 0.15418 nm) over the scanning range 2θ = 10–80° for wide angle XRD at a voltage of 40 kV and 200 mA. Raman spectroscopy was recorded on a JYHR800 Raman spectrometer with a 488 nm argon ion laser (France JY Company). Laser wavelength: 514 nm. Transmission electron microscopy (TEM) morphologies of samples were observed with a Philips TEMCNAl-12 with an acceleration voltage of 100–120 kV. Microhardness was measured by HV-1000 Microhardness Tester at a load of 1 N for 15 s. The wear loss was measured using analytical balance.

2.5. Friction and wear test

Friction and wear tests of carbon nanotubes/hydroxyapatite composites were conducted on UMT-2 friction tester using ball-on-disk mode (CETR, USA) under dry conditions. The specimens were tested at normal load of 0.5, 1.0, 1.5, 2.0 and 2.5 N, accompanied with a constant rotor speed of 120 rpm and testing time of 600 s. The friction coefficient was recorded continuously by the tester. A stainless-steel ball (44OC) of ∅ 4 mm with a hardness of 62 HRC was used as a counterface friction material. The wear testing specimens were prepared prior to testing. Typically, 0.05 g of PVA was dissolved in 100 ml distilled water and then 1 g of composite sample was added into the PVA aqueous solution with stirring. After that, the mixed solution was dried at 80 °C for 4 h to obtain a dried composite powder. The powder was pressed to the flake-shaped solid by unidirectional axial compression method, about 4–5 mm in thickness and 10 mm in diameter. Then, the prepared solid sample was put into the tube furnace and was heated to 1000 °C at a heating rate of 5 °C/min in N_2 atmosphere and kept at 1000 °C for 2 h, denoted as HA-1000, CNTs (2%)/HA-1000, CNTs (5%)/HA-1000, CNTs (10%)/HA-1000, CNTs (15%)/HA-1000 and CNTs (20%)/HA-1000, respectively.

3. Results and discussion

3.1. Characterization of carbon nanotubes/hydroxyapatite composites

3.1.1. TEM analysis

Fig. 1 presents TME images of the pure HA and several CNT/HA composites. As shown in Fig. 1a, the synthesized HA sample exhibits the needlelike structure with uniform size, about 20–60 nm in length and 15 nm in diameter. As displayed in Fig. 1, when the CNTs content in composite was very low, it is noted that there are a number of free HA crystal grains in composite except that the surface of CNTs was uniformly covered with some of the HA crystal grains. With the increase of the CNTs content, the free HA crystal grains gradually decreased and the HA crystal grains covered on the surface of CNTs gradually decreased, which is probably attributed to the decrease of the HA content in the composite. Also, we found that when the CNTs content in composite was 15 wt.%, the HA uniformly covered on the surface of the CNTs and the obtained CNTs/HA composite exhibits good morphology.

3.1.2. XRD analysis of the pure HA, CNTs and CNTs/HA composites

Fig. 2 gives the XRD patterns of the pure HA, the CNTs and several CNTs/HA composite samples. From Fig. 2, The CNTs sample exhibits four diffraction peaks at *ca.* 2θ = 25.9°, 42.5°, 53.3° and 77.5°, which are assigned to the characteristic peaks of carbon nanotubes. Besides, it can be found that the pure HA sample exhibits several diffraction peaks at about 2θ = 25.8°, 31.8°, 32.2° and 33° corresponding to the (0 0 2), (2 1 1), (1 1 2) and (3 0 0) plane of hydroxyapatite, which is good agreement with the card (JCPDS No. 9-432). Further, several characteristic peaks of hydroxyapatite can be observed while the other impurity peaks was not noted, and the peaks are sharp and narrow, suggesting that the purity of the HA sample is high and the crystallinity is good. Additionally, according to Fig. 2, several CNTs/HA composite samples exhibit two kinds of diffraction peaks including the characteristic peaks of hydroxyapatite and carbons nanotubes. However, the intensities of diffraction peaks assigned to the CNTs are slight low and some of diffraction peaks relating to the CNTs can hardly been identified. This phenomenon is aroused by two reasons. One reason is probably the overlapping of the diffraction peak of CNTs with the diffraction peaks of HA. The other one may be due to the lower CNTs content in composite.

3.1.3. XRD analysis of CNTs (15%)/HA composite sample before and after calcination

Fig. 3 illustrates the XRD patterns of CNTs (15%)/HA and CNTs (15%)/HA-1000 samples. It is seen that the crystallinity of the CNTs

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