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Microstructure and strength of nano-/ultrafine-grained carbon nanotube-reinforced titanium composites processed by high-pressure torsion

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ABSTRACT

Nano-/ultrafine-grained carbon nanotube-reinforced titanium (CNT/Ti) composites were synthesized by ball milling and high pressure torsion (HPT) at room temperature. The effects of the number of HTP rotations and the weight fraction of CNTs on the microstructure, hardness and tensile properties of the CNT/Ti composites were investigated. Transmission electron microscopy (TEM) revealed that elongated grains with an average length of 100–250 nm parallel to the compression axis of HPT and a thickness of 10–25 nm are formed in CNT/Ti composites when CNT contents ranging from 0.3 wt% to 1.0 wt%. With increasing CNT contents, the grain size of Ti is refined, and the microhardness and tensile properties of the composites increase. Evaluating the mechanical properties of the CNT/Ti composites with 0.7 wt% CNTs after 20 rotations indicates a high tensile strength of 872 ± 5 MPa. C_s-corrected high resolution TEM of the interfaces between Ti and the CNTs after HPT reveals a gradual transition from the lattice planes of hexagonal Ti to those of the CNTs. The strengthening mechanisms are discussed from the aspects of matrix grain refinement, distribution of the CNTs and the CNT-Ti interfaces obtained during ball milling and HPT processing.

1. Introduction

Carbon nanotube-reinforced metal matrix (CNT/MMC) composites with excellent mechanical properties are of interest for many industrial applications and now have been developed for a variety of alloy systems, such as Al [1,2], Cu [3,4], Mg [5], Fe [6], Ni [7,8], and NiTi alloys [9]. However, the property improvement of CNT/MMCs has not reached the theoretical level. The major challenge to improve the mechanical properties of those composites is to obtain a homogeneous dispersion of CNTs and a strong interface between the reinforcement and the matrix [10,11].

Ti and its alloys are very popular as competitive metal matrix materials, but only a few studies on CNT/Ti composites are available [12]. There are different processing techniques to prepare CNT/Ti composites, i.e., hot pressing, spark plasma sintering (SPS) [13,14], hot extrusion [15–17] and so on. Jiang et al. reported that the mechanical properties of CNT/MMCs can be linked to the good structural integrity of the CNTs [16]. Wang et al. synthesized novel CNT/Ti composites by rapid low temperature SPS [17]. It can be noted that most of these composites are usually accomplished at high temperatures, which may result in a coarse-grained matrix or the formation of TiC via reaction of CNTs with titanium occurring at high processing temperatures. High pressure torsion (HPT), a severe plastic deformation (SPD) process, is capable of producing bulk nano-/ultrafine-grained composites with refined microstructure at low processing temperatures [18,19]. For example, Jenei et al. [18] fabricated CNT/Cu nanocomposites with high hardness by the HPT process. Li et al. [19] achieved a homogeneous dispersion of CNTs using HPT. Hence, the use of HPT seems to be appropriate to fabricate nano-/ultrafine-grained CNT/Ti composites with refined microstructure, good homogeneity and enhanced strength. Moreover, low processing temperatures may efficiently avoid the growth of the grains and the formation of TiC. However, detailed investigations on the strengthening contributions of CNTs and Ti matrix grain refinement during the HTP process, as well as the CNT-Ti interface conditions are still very scarce.

In this work, CNT-Ti powders were mixed by ball milling, which promotes homogeneous distribution of CNTs. Second, the mixed CNT-Ti powders were consolidated by HPT, which decreases the matrix grain size yielding nano-/ultrafine-grained CNT/Ti composites. HPT also helps to further increase the homogeneous distribution of CNTs and improves the interface bonding conditions between metal matrix and reinforcement. The microstructure, the microhardness, the tensile

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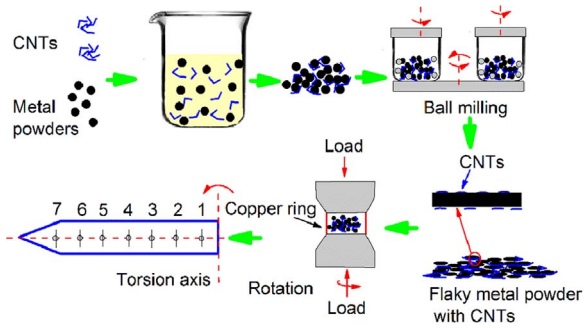

Fig. 1. Schematic illustration of the CNT/Ti composite production.

properties of the nano-/ultrafine-grained CNT/Ti composites, and especially the interface between Ti and CNTs were analyzed. Finally, their strengthening mechanisms were discussed.

2. Experimental details

Commercial Ti powders with a purity of 99.7% and an average particle size of 20 μm (Germany), and commercial multi-walled CNTs supplied by SIGMA-ALDRICH Co. USA with a purity higher than 98%, an average inner and outer diameter of 6–13 nm, and a length of 2.5–20 μm were used for the experiments. Fig. 1 illustrates the fabrication process of the samples.

First, the CNTs were washed in alcohol and dried in air at 350 K for 60 min. Then an isopropyl alcohol (IPA)-based solution [20,21] was used to obtain solutions with different contents of CNTs, i.e., 0.3 wt%, 0.5 wt%, 0.7 wt%, and 1.0 wt%. Second, 50 g Ti powder was added into the slurries of the prepared CNT solutions and dried in an oven at 373 K during the stirring process. Third, the composite powders were ball milled separately in an argon protected planetary ball mill for 3 h at 100 rpm using 10 mm steel balls and a 10:1 ball-to-powder weight ratio. Finally, the mixed powders were subjected to HPT processing at room temperature using a 400 kN device resulting in an applied pressure of 7.5 GPa, a rotation speed of 0.6 rpm and various numbers of rotations, i.e., 5, 10, 15, 20 and 30 rotations. The HPT consolidated samples had a diameter of 7.5 mm and a thickness of 1 mm.

The mass densities of the HPT-processed samples were measured using Archimedes' principle after the samples were polished with diamond paste. Microstructure characterization of the samples was performed using an optical microscope, a field emission scanning electron microscope (FE-SEM), a Philips CM12 transmission electron microscope and a Jeol JEM 2100 F C_s-corrected high-resolution transmission electron microscope (HR-TEM) operated at 200 kV. The TEM samples were cut from the HPT-disks at a position 3.0 mm and mechanically thinned to a thickness of about 50 μm, followed by mechanical dimpling. Subsequently the samples were ion-milled using a Gatan Precision Ion Polishing System until perforation using a voltage of 4 kV and an angle of 5°. Vickers microhardness was measured using a using a MATSUZAWA MMT-X instrument with a load of 500 g applied for 10 s on the cross-section of the HPT-processed disks, and the distance between the indents was set to 0.5 mm. Microtensile samples with a 1.5 mm gage length and 1 mm width were cut from the disks using electro-discharge machining at a position 2.5 mm from the center of the disk. During the tensile test, precise strains were measured using the digital image correlation (DIC) method.

3. Results and discussion

3.1. Ball milling process

Fig. 2 depicts representative SEM and TEM micrographs revealing the morphologies of the Ti–CNT/Ti powder mixtures before and after mechanical milling for 3 h. As shown in Fig. 2(a), the SEM images of the powder mixture surfaces after mechanical milling clearly show that the size of the particles is decreased. This can be attributed to the fact that the mechanical milling process involves deformation of the powders by a milling medium, i.e., the powder particles are repeatedly flattened and fractured. As shown in Fig. 2(b), mechanical milling also improves the dispersion of CNTs on the Ti powder particle surfaces due to the repeated collision of the CNT/Ti powder mixture with the steel balls, which helps to form a CNT/Ti composite at the surface of the Ti powders. Obviously, the CNTs in the powders after the ball milling process a good structure and are not severely distorted or crushed. These powders were consolidated into bulk samples using the HTP process.

3.2. HPT process

Fig. 3 presents characteristic micrographs of the CNT/Ti composite with 0.7 wt% CNTs synthesized by HPT after 10 rotations. The grains at the center of the HPT-processed sample are large (about 20 μm) and deformed along the loading direction, as shown in Fig. 3(a), and some pores can be observed. However, the grains at the edge of the sample in Fig. 3(b) are flattened and smaller, and the porosity is less, implying a higher level of deformation in this area compared with that at the center of the sample (Fig. 3(a)).

The morphology changes from the center of the disk towards the edge, and the grains are also more refined from the center to the edge, as it is expected for HPT-processed specimens after 10 rotations exhibiting a deformation gradient across the diameter of the disk. When the number of rotations was increased to 20 rotations, a homogeneous microstructure can be obtained.

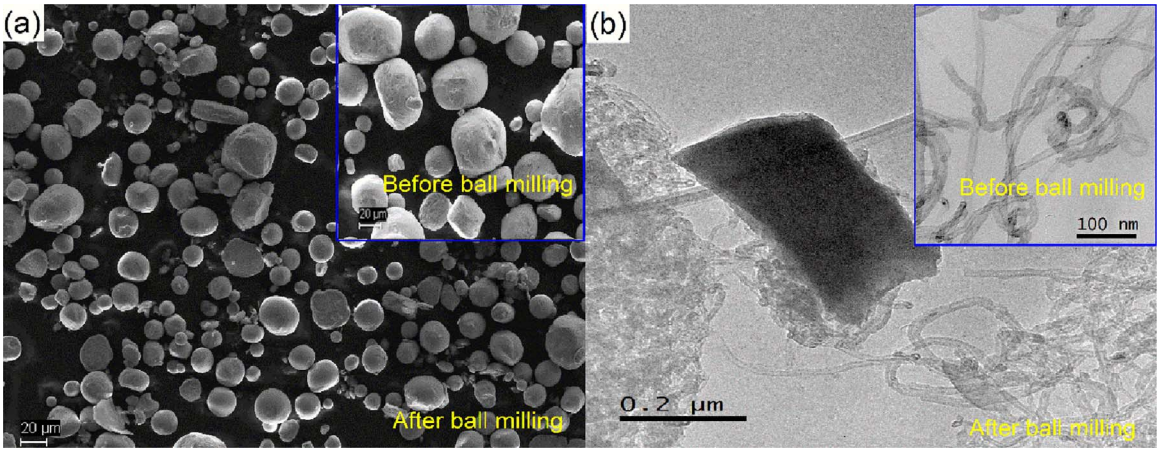

Fig. 2. Representative SEM (a) and TEM (b) micrographs depicting the morphologies of the Ti–CNTs mixed powders after ball milling for 3 h.