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Titanium/nanodiamond nanocomposites: Effect of nanodiamond on microstructure and mechanical properties of titanium

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ABSTRACT

Titanium (Ti)/nanodiamond (ND) nanocomposites with potential for biomedical applications were prepared by using spark plasma sintering technique. By means of X-ray diffraction, scanning electron microscopy, transmission electron microscopy and mechanical analysis, the Ti/ND nanocomposites were investigated, and thus the effect of ND on the microstructural and mechanical properties of Ti matrix was demonstrated. Experimental results showed that the Ti/ND nanocomposites exhibited pure α-Ti phase with ND concentrations from 0.1 to 0.35 wt% and with in-situ formed nano-TiC phase in 0.5–2.0 wt% NDs. The nanoindentation hardness, Young's modulus and compressive yield strength of the Ti/ND nanocomposites were significantly improved, as ND was incorporated into the Ti matrix. Improvements of hardness (60.2%), Young's modulus (27.4%) and compressive yield strength (24%) were achieved by doping of 0.5 wt% NDs in the Ti matrix but at an expense of ductility. The Ti/0.35 wt% NDs nanocomposites have the best integrated mechanical properties. These improvements could be ascribed to the outstanding mechanical properties of ND, homogeneous dispersion of ND nanoclusters, Orowan strengthening with ND/nano-TiC and carbon atom solid solution strengthening in the Ti/ND nanocomposites.

1. Introduction

Titanium (Ti) has found extensively applications in many engineering industries such as aerospace, chemical and biomedical fields due to their light weight, high specific strength, excellent chemical resistance and biocompatibility [1,2]. Pure Ti was once used as biomaterials, but the disadvantage for the use of pure Ti as implant materials is its low strength and insufficient hardness, which limit its application in artificial implants of hip and knee prostheses [3,4]. Ti alloys containing elements of vanadium (V), aluminum (Al), iron (Fe), niobium (Nb), zirconium (Zr), tantalum (Ta), molybdenum (Mo), nickel (Ni), gold (Au), silicon (Si), manganese (Mn) and so forth have been investigated for biomedical applications [5–14]. The mechanical properties of the Ti metal have been greatly enhanced by the alloying method. However one main problem for the Ti alloys is that metal ion release may induce cytotoxicity and damage human body [15,16].

Alternatively, Ti metal matrix composites (MMCs) with nanostructured reinforcement can offer superior mechanical properties and reduced weight. Carbon nanotubes (CNTs) and graphene make contributions to the development of new materials with outstanding mechanical properties because of its exceptional mechanical and high aspect ratio [17–19]. Many literatures showed significant achievements in Ti MMCs reinforced with carbon nanotubes (CNTs) and graphene [17–23]. However, the CNTs and graphene are both cytotoxic, which limit their applications in biomedical field [24–28]. Shvedova et al. [25] reported that exposure of human epidermal keratinocytes to CNTs produced oxidative stress and cellular toxicity, in addition it resulted in ultrastructural and morphological changes in cultured human cells. Graphene leads to significant interactions with membrane lipids leading to direct physical toxicity or adsorption of biological molecules leading to indirect toxicity, and it has a potential to induce foreign body tumors [26–28]. Another promising nanocarbon material, nanodiamonds (NDs) have excellent mechanical properties, high surface areas and tunable surface structures [29]. Especially, NDs are non-toxic and biocompatible comparing to CNTs and graphene, which make them very promising for biomedical application [30]. Diamond and diamond-like carbon films have been used for robust implant coatings [31,32] and NDs have been applied as stable cellular biomarkers [33], probe for biolabeling, and foundation for chemotherapeutic drug carriers and anti-inflammatory interfaces [34] as well as for localized cancer treatment [35]. By adding NDs into polymer, Cu and Al matrix, superior wear resistance, hardness and excellent bending strength can be

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obtained, which are much higher than that with CNTs [36–40]. Furthermore, owing to higher mechanical strength of NDs, the final particle size of 4–5 nm can be preserved without damaging its structures during milling process. To the best of our knowledge, there is just few work focusing on Ti MMCs reinforced with NDs. Melendez et al. [41] have reported that the bending strength of Ti MMCs reinforced with NDs (1.8% vol.) was higher than that with the same amount of CNTs, which were fabricated via normal powder metallurgy method. However, the optimal amount of NDs, the quantitative evaluation of mechanical properties and the strengthening mechanism of Ti MMCs reinforced with NDs have never been reported.

The purpose of this work is to study the preparation, effect of the ND on the microstructures and mechanical properties of the Ti matrix. The Ti/ND nanocomposites were prepared by spark plasma sintering (SPS) technique which can enable MMCs with favorable properties to be consolidated by shorter holding times and relatively lower temperatures [42–45]. The effects of ND amounts on the microstructure and mechanical properties of Ti MMCs are investigated. Additionally, the strengthening mechanisms of NDs in the Ti MMCs are explored. The possible biomedical applications of the Ti/ND nanocomposites is discussed.

2. Experimental procedure

2.1. Materials

Ti powders were prepared by hydride-dehydride method with purity of 99.5% and mean particle size of 10–44 μm (Nanjing Mingshan Advanced Materials Co. Ltd., China). Spherical NDs were fabricated by detonation technique and used as reinforcements with purity of > 98%, 5 nm in diameter and mean specific surface area of 350 m^2/g (Tianjin Qianyu Superhard Materials Co. Ltd. China).

2.2. Nanocomposite fabrication

NDs were mixed with Ti powders with various fractions of 0.1, 0.25, 0.35, 0.5 wt% and up to 2.0 wt%. They were mixed through a two-stage process. The schematic illustration of fabrication process of the Ti MMCs reinforced with NDs is presented in Fig. 1. In stage I, the NDs were dispersed in ethanol solution using ultrasonication for 30 min. The Ti powders were dispersed in another glass cup in the same way. Afterwards the two kinds of powder solutions were mixed together and stirred using ultrasonication for 30 min. In addition, they were mixed by using a high energy planetary ball milling machine (QM-3SP2) with ball to powder ratio of 10:1 for 5 h at 250 RPM. The powder mixtures were dried in a vacuum oven, loaded into a cylindrical graphite die (Φ10 mm) and consolidated using spark plasma sintering (SPS) in stage II. The SPS experiments were conducted in a spark plasma sintering system (FCT-HP-D5, FCT Systeme GmbH, Germany) installed at Southeast University. The sintering temperature was measured by a thermocouple (TC). The sintering parameter was set as 900 °C for 10 min at a pressure of 60 MPa in vacuum with a heating rate of 100 °C/min. The resulting sintered specimen have diameters of 10.0 mm and heights of 12.0 mm.

2.3. Characterization techniques

The relative densities of all the sintered Ti MMC samples were determined using Archimedes principle in water at room temperature. The phase compositions of the samples were identified by X-ray diffraction system (XRD, D8 discovery, Bruker) with Cukα monochromatic radiation. The microstructures of these sintered samples were characterized using optical microscopy (OM, Olympus, BX60M), field-emission scanning electronic microscopy (FESEM, Sirion, FEI) equipped with energy-dispersive X-ray spectrometer (EDS), and transmission electron microscopy (TEM, Tecnai, FEI) with selected area electron diffractions (SAD). The hardness and Young's modulus of the samples were measured by a nanoindentation test system (Micro Materials-NanoTest). A calibrated diamond Berkovich indenter tip was used for indentation at the maximum load of 5 mN and loading-unloading rate of 0.25 mN/s. Compression tests were conducted on a universal testing machine (CMT5305, MTS) with a strain rate of 0.5 mm/min. Five samples of each group was used for statistical analysis of the mechanical properties.

3. Results and discussions

3.1. Powder morphology

Fig. 2(a) shows the SEM micrograph of pure Ti powders. The raw Ti powders exhibit irregular shape with particle sizes of 10–44 μm. The TEM image of NDs is shown in Fig. 2(b). The round nanometer sized

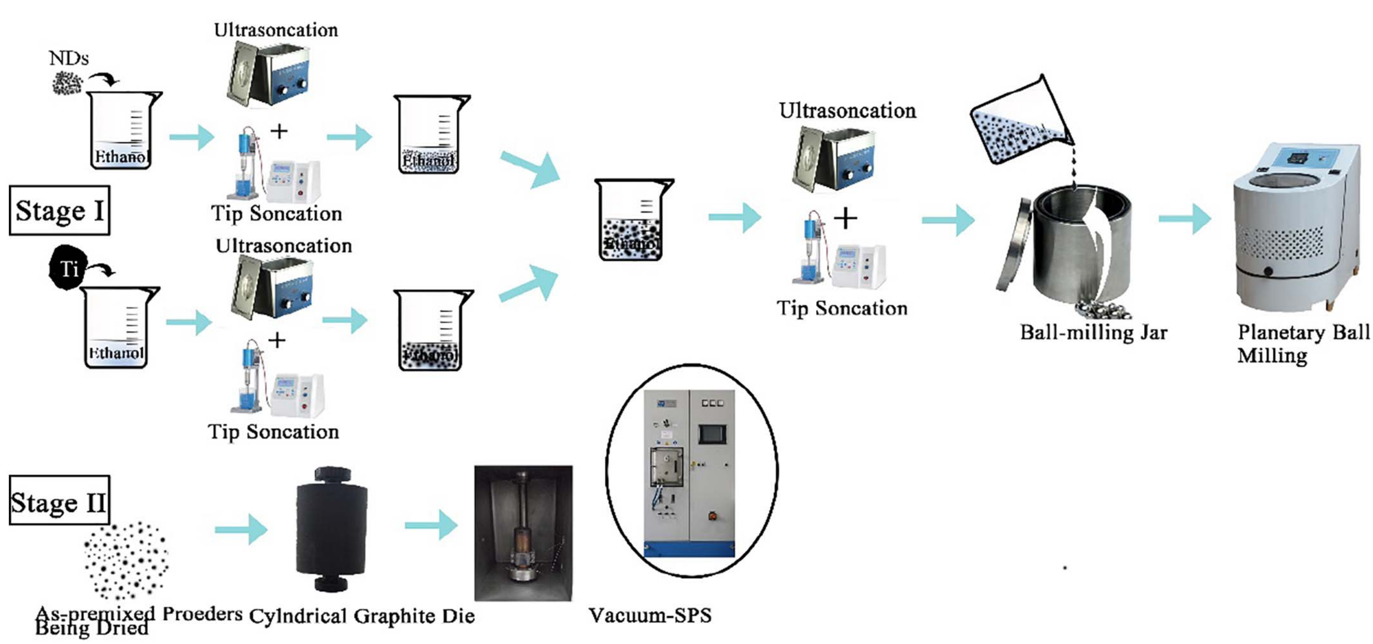

Fig. 1. Schematic illustration of processing procedure for the Ti/NDs nanocomposites.