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Synthesis of Ti–Sn–Nb alloy by powder metallurgy

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Abstract

The microstructural evolution and characteristics of the Ti–16Sn–4Nb powder particles and bulk alloys sintered from the powders ball-milled for various periods of time were studied. Results indicated that ball milling to 8 h led to the development of a supersaturated hcp α-Ti and partial amorphous phase due to the solid solution of Sn and Nb into Ti lattice. The bulk Ti–16Sn–4Nb alloy made from the powders ball milled for a short time, up to 2 h, exhibited a primary α and a Widmanstätten structure consisting of interlaced secondary α and β. With an increase in ball milling time up to 10 h, the microstructure evolved into a fine β phase dispersed homogeneously within α phase matrix. The microhardness values of the bulk alloy in both α- and β-phases increased with the increasing of the ball milling time and reached a plateau value at 8 h and longer, i.e. 687 and 550 HV for α- and β-phases, respectively. Likewise, the microhardness of the α phases was always higher than that of the β phases in the bulk alloys made from the powders ball milled for the same milling time.

Keywords: Mechanical alloying; Titanium alloys; Microstructure; Powder metallurgy; Metallic biomaterials

1. Introduction

Titanium (Ti) and some of its alloys are widely used as dental and orthopaedic implant materials due to their excellent corrosion resistance, biocompatibility, high degree of strength-to-weight ratio, as well as the combination of high strength and low Young's modulus [1,2]. Pure titanium exhibits an allotropic changing from a body-centred cubic crystal structure at high temperatures (β phase) to a hexagonal close-packed crystal structure (α phase) at lower temperatures. The transformation temperature and microstructure of titanium are highly influenced by interstitial and substitutional elements resulted in a broad range of alloys with different properties and applications. This diversity is particularly highlighted in the α + β alloys [3]. Alloying elements like tin (Sn) and niobium (Nb) are biocompatible metals [4] and have complete solubility in the α and β phases or just in β phase of titanium, respectively [3]. In the present study, Sn was selected as the first additional element to increase strength and Nb was added to obtain a partial β-structure which contributes to decreasing the bulk elastic modulus of the alloy required for orthopaedic implants [5].

Using mechanical alloying (MA) to synthesize titanium-based alloys is a field of growing interest. This technique is a solid-state powder metallurgical process in which elemental powders are being alloyed by repeated deformation/cold-welding/fracture mechanisms under frequent mechanical impacts. It involves diffusion at atomic level and allows production of various non-equilibrium phases ranging from supersaturated solid solutions to nanocrystalline and/or amorphous phase with unique characteristics [6–9]. MA is capable of processing titanium alloys with specific microstructures and improved mechanical properties compared to the conventional powder metallurgy or casting techniques [10–12]. As the most important process variable of MA, the milling time is responsible for the final constitution of powders, microstructure and mechanical properties of a particular alloy.

Besides the chemical composition of mechanically alloyed materials, a full understanding of the microstructural evolu-

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tion of bulk alloys fabricated by sintering from the powders after different ball milling periods is a matter of importance. In the present study, a biocompatible Ti–16Sn–4Nb (wt.% hereafter) alloy was prepared from elemental powders by mechanical alloying for relatively short periods of ball milling. The morphological changes of powders and microstructural evolution of the sintered alloys were investigated using optical microscopy, scanning electron microscopy combined with energy dispersive spectrometer (SEM-EDS), and X-ray diffraction analysis (XRD). The microhardness measurement was performed using a Vickers hardness indenter.

2. Experimental procedure

Elemental powders of titanium (purity 99.9%, ≤45 μm), tin (purity 99.9%, 45 μm) and niobium (purity 99.8%, ≤45 μm) were prepared as starting materials. The powders were mixed together to obtain the targeted composition of Ti–16Sn–4Nb alloy. The MA process was conducted in a planetary ball mill (Retsch, PM400) with stainless steel containers and balls at room temperature. The weight ratio of ball to powder was maintained at 20:1. The ball milling was carried out at a rotation rate of 200 rpm. To minimize the contamination of oxygen and nitrogen, all the powders were handled in a glove box chamber under argon gas. The containers were also sealed and purged with argon before and during milling process. Due to the ductility of titanium and tin powders, the milling process must be carefully controlled by adding a certain amount of process control agent (PCA) to the ball milling environment which improves the milling efficiency by achieving a balance between cold welding and fracturing. About 2 wt.% of ethylene-bis-stearamide (EBS) was added as PCA and the powders were subsequently ball milled for different times from 20 min to 10 h.

The mechanically alloyed powders were consolidated by a uniaxial cold press under the pressure of 750 MPa. The green compacts were then sintered in a high vacuum furnace at 1150 °C for 5 h with the heating/cooling rate of 10 °C/min.

Metallography of the as-sintered samples was carried out using conventional techniques and specimens were etched with Kroll solution: 3 ml HF, 6 ml HNO_3, 100 ml H_2O. The chemical analysis of the bulk samples after sintering was carried out using the inductively coupled plasma-atomic emission spectrometry (ICP-AES) and Leco combustion methods. Particle morphologies of the ball milled powders and the microstructure of bulk samples after sintering were observed by optical microscope, as well as scanning electron microscopy combined with secondary electron imaging (SEM-SEI). The distribution of elemental metals within the particles was analysed by scanning electron microscopy using backscattering electron imaging (SEM-BEI) combined with energy dispersive spectrometer (EDS) (Leica S440). Phase formation was characterised using XRD with Cu Kα radiation.

The Vickers microhardness of the sintered alloy samples was tested at a load of 100 g for 30 s. Average hardness values were obtained from at least six indents on each sintered sample.

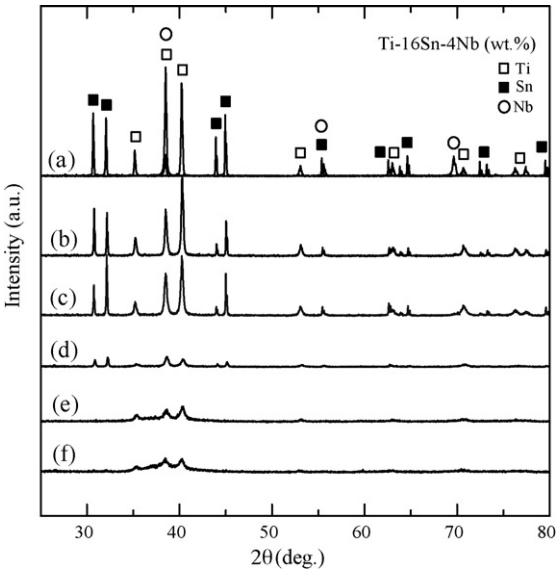

Fig. 1. XRD patterns of Ti–16Sn–4Nb powders ball milled for different times: (a) 0 h; (b) 20 min; (c) 2 h; (d) 5 h; (e) 8 h; and (f) 10 h.

3. Results and discussion

3.1. Phase formation

The XRD patterns of the Ti–16Sn–4Nb alloy powder mixtures and bulk samples made from the powders ball milled for different times (20 min to 10 h) are shown in Figs. 1 and 2, respectively. The original powder mixture comprised of all elemental metals of Ti, Sn and Nb (Fig. 1(a)). Irrespective of a slight change in the peak intensities of the elemental metals, there were

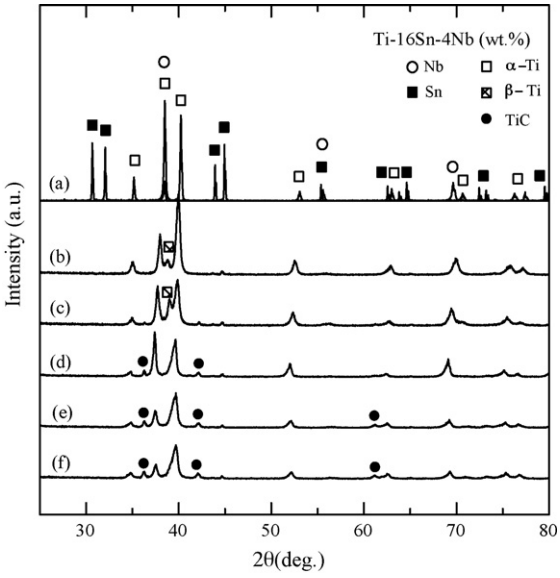

Fig. 2. XRD patterns of (a) elemental powders and the bulk Ti–16Sn–4Nb made from the powders ball milled for different times: (b) 20 min; (c) 2 h; (d) 5 h; (e) 8 h; and (f) 10 h.