



Sintering behavior and mechanical properties of a metal injection molded Ti–Nb binary alloy as biomaterial



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ARTICLE INFO

Article history:

Received 25 January 2015

Received in revised form 25 March 2015

Accepted 5 April 2015

Available online 9 April 2015

Keywords:

Metal injection molding

Ti–Nb alloy

Sintering behavior

Titanium carbide

Mechanical properties

ABSTRACT

Sintering behavior, microstructure and mechanical properties of a Ti–16Nb alloy processed by metal injection molding (MIM) technology using elemental powders were investigated in this work by optical microscopy, X-ray diffraction (XRD), dilatometer, scanning electron microscopy (SEM) and energy-dispersive spectroscopy (EDS). It was found that from 700 °C to 1500 °C the homogenization and densification process of MIM Ti–16Nb alloy consisted of three steps, i.e., Ti-diffusion-controlled step, Ti–Nb-diffusion step and matrix-diffusion step. Titanium carbide formation was observed in the samples sintered at 1300 °C and 1500 °C, but not in the ones sintered at 900 °C and 1100 °C. The MIM Ti–16Nb specimens sintered at 1500 °C exhibited a good combination of high tensile strength and low Young's modulus. However, the titanium carbide particles led to poor ductility.

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1. Introduction

Titanium (Ti) and its alloys are excellent materials for medical applications owing to their excellent corrosion resistance, good fatigue strength and high strength-to-weight ratios [1]. They up to now mainly used in the form of commercially pure Ti (CP-Ti) and Ti–6Al–4V alloy [2]. However, the stress shielding effect generated by the mismatch of Young's modulus between them and bone and the potential cytotoxic effect resulting from aluminum (Al) and vanadium (V) ions eventually released from Ti–6Al–4V are disadvantageous for biomedical applications in the human body [3]. Niobium (Nb) is a strong β -stabilizer in Ti alloys and thus contributes significantly to a decrease of the Young's modulus of Ti alloys [4]. Furthermore, Nb is a non-toxic element and is not associated with any tissue reaction [5]. Therefore, increasing attention has been paid on Ti–Nb alloys for biomedical applications [4]. Hanada et al. [6] found that in Ti–Nb binary alloys, there is a minimum Young's modulus when the Nb content is around 16 wt.%.

Artificial implants usually exhibit complex shapes with sophisticated details, however, the conventional processing of titanium implants is limited due to the costly, multi-step process of fabrication and associated geometry design constrains [7]. Metal

injection molding (MIM) is a net-shape powder metallurgy technique with the potential to avoid such problems and can therefore contribute to the development of Ti implants with higher functionality and significant cost reduction [8]. German and Bose [9] stated that there are three stages during the sintering of MIM specimens – (a) in the initial stage, the shrinkage of the powder compact starts when neck growth takes place; (b) the intermediate stage determines the densification of the final compact and shows the highest shrinkage rate among the three stages of the sintering process; (c) due to the formation of the closed spherical pores, further shrinkage becomes very difficult in the final stage.

In powder metallurgy techniques (e.g., MIM), the advantages of using blended elemental instead of the pre-alloyed powders include: (1) feasibility to change composition [10]; (2) lower cost [11]; (3) possible formation of unique microstructures [12]; (4) enhanced diffusional fluxes resulting from compositional gradients during sintering densification [13]. However, the sintering parameters should be carefully controlled to avoid compositional inhomogeneity of final products [14], because the sintering of multicomponent systems includes two processes – densification and homogenization [15–17]. Both processes are dependent on mass diffusion [18].

There have been several attempts in the MIM of Ti–Nb alloys in recent years. The first report of MIM Ti–Nb alloy focused on the fabrication of a MIM Ti–17Nb alloy using elemental Ti and Nb powders [19]. Good shape preservation and reproducibility has been

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observed in the as-sintered MIM Ti–17Nb samples. Zhao et al. [20] investigated the microstructure and mechanical properties of a series of MIM Ti–Nb alloys. After sintered at 1500 °C for 4 h, the MIM Ti–22Nb alloy exhibited a good combination of high yield strength (about 650 MPa) and low Young's modulus (about 70 GPa). However, the densification and homogenization mechanism, microstructural evolution during sintering, and their correlations with sintered mechanical properties of MIM Ti–Nb alloys fabricated using elemental powders have not been thoroughly analyzed. Hence there is a fundamental need to understand the sintering behavior of the MIM Ti–Nb alloys. In this study, the MIM technique was applied to fabricate Ti–16Nb alloys using gas-atomized Ti and hydride/dehydride (HDH) Nb powders. The purpose was to understand the homogenization and densification behavior of a MIM Ti–Nb alloy during sintering, and to evaluate the mechanical properties of Ti–Nb samples fabricated by MIM.

2. Experimental

The Ti powder (–325 mesh, average particle size: 21 µm; supplied by TLS Technik GmbH, Germany) and Nb powder (–120 mesh, average particle size: 75 µm; supplied by MHC Industrial Corporation, China) were used to fabricate Ti–16Nb (wt.%) alloys. Fig. 1 presents the morphology of the Ti and Nb powders.

The samples were produced by blending elemental Ti powder and Nb powder. The powders were mixed with the polymer binder consisting of 5 wt.% stearic acid, 35 wt.% polyethylene vinyl acetate and 60 wt.% paraffin wax by means of a Z-blade kneader at a temperature of 120 °C for 2 h to form a feedstock. All feedstocks used in the experiments had the same binder system fraction of 31 vol.%. An Arburg 320S machine was used for the injection molding of the feedstocks after granulation. The feedstocks were injection molded as standard “dog bone” shape tensile specimens with a nominal length of 89 mm and gauge thickness of 4.9 mm. The paraffin removal was performed by solvent extraction, i.e., immersing the green (after injection) components into hexane at 40 °C for 20 h in a LÖMI EBA 50 debinding device. Thermal debinding and the sintering of specimens were carried out in one cycle. Thermal debinding was under argon (Ar) flow (500 Pa), while, the sintering process was under vacuum (10^{-3} Pa). Sintering was conducted in a XERION XVAC 1600 furnace with tungsten heating elements and shield packs of molybdenum. In order to investigate the sintering behavior of the MIM Ti–Nb alloys, an identical thermal debinding cycle was used for all specimens and only sintering temperature was varied. Sintering temperatures ranged between 900 °C and 1500 °C with a heating rate of 5 °C/min. After reaching the sintering temperature, the samples were held for 2 h and then furnace cooled to room temperature with a cooling rate of 10 °C/min. In the following, the as-sintered Ti–16Nb samples are referred to as Ti–16Nb (900), Ti–16Nb (1100), Ti–16Nb (1300) and Ti–16Nb (1500) indicating sintering temperatures of 900 °C, 1100 °C, 1300 °C and 1500 °C, respectively.

A conventional LECO melt extraction system was used to determine the impurity levels of oxygen (O), carbon (C) and nitrogen (N). Constituent phases of the samples were characterized by X-ray diffraction (XRD), which was conducted on a Siemens D5000 diffractometer at 40 kV and 40 mA at room temperature using the copper K α wavelength (1.5418 nm). Optical microscope (Olympus PMG3), scanning electron microscope (SEM) (Zeiss-DSM962) and energy-dispersive spectroscopy (EDS) in the SEM (EDAX LEO 1530) were used for microstructural examination and compositional analysis. The porosity of each sample was measured using an image analysis system (Olympus Soft Image Solution, analysis pro).

Dilatometry measurements were performed on a vertical configuration dilatometer fabricated by LINSEIS (L70/2171) under Ar atmosphere. MIM cylinders (diameter: 5 mm, length: 10 mm) made from Ti–16Nb were chemically and thermally debound followed by pre-sintering at 700 °C for 1 h. The microstructure of the pre-sintered MIM Ti–16Nb sample is shown in Fig. 2. The same fabrication process was also applied to produce MIM CP-Ti cylinders as a reference in the

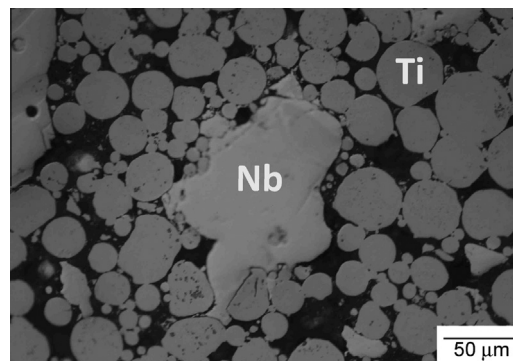


Fig. 2. Optical micrograph of the pre-sintered MIM Ti–16Nb sample.

dilatometry measurement. The two specimens for dilatometry are referred to as Ti–16Nb (DIL) and CP-Ti (DIL), respectively. A heating rate of 3 °C/min and an isothermal holding at 1500 °C for two hours were used. The linear shrinkage (LS) and linear shrinkage rate (LSR) of the samples were calculated by using

$$LS = (l_0 - l_t) / l_0 \times 100\% \quad (1)$$

and

$$LSR = \frac{d}{dt} \left(\frac{l_0 - l_t}{l_0} \right) \quad (2)$$

where l_0 and l_t are the sample length before the dilatometry measurement and the length at time t during the measurement, respectively.

The as-sintered “dog bone” shape tensile specimens were directly used in the tensile tests without any preparation. Tensile tests were performed on a servohydraulic test machine (RM-100, Schenk-Trebel, United States) modernized with a Zwick DUPS electronic testing system, and equipped with a 100 kN load cell using a strain rate of $1.2 \times 10^{-5} \text{ s}^{-1}$ to measure tensile strength, elongation and Young's modulus. The elongation was measured by a laser noncontact extensometer (WS-160, Fiedler Optoelektronik GmbH, Germany). At least three samples of each configuration were tested.

3. Results and discussion

3.1. Impurity levels

The Ti powder, Nb powder and the as-sintered MIM Ti–16Nb samples described in the following sections were analyzed with respect to their content of O, C and N (Table 1). The deviation in these impurity levels of the as-sintered alloys was not significant.

3.2. XRD study

The XRD spectra of the Ti–16Nb (900), Ti–16Nb (1100), Ti–16Nb (1300) and Ti–16Nb (1500) samples are displayed in Fig. 3. Body centered cubic (BCC) α -Nb phase was observed in the Ti–16Nb (900) and Ti–16Nb (1100) samples, but not in the Ti–16Nb (1300) and Ti–16Nb (1500) samples. Such a result indicates that the dissolution of Nb particles into the matrix had finished in the latter two samples, but not in the former two. It is important to

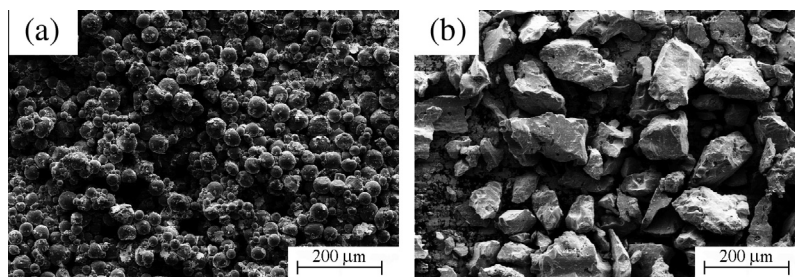


Fig. 1. Scanning electron micrographs of (a) Ti powder and (b) Nb powder.

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