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Powder Metallurgy Processing of Ti–Nb Based Biomedical Alloys

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New Ti based alloys with non-cytotoxic and biocompatible elements, such as Nb, Zr, Ta, Sn and Mo, have recently attracted much attention for orthopedic applications. The aim of this study was to investigate the effects of Sn content (2 and 4 wt%) on microstructure and mechanical properties of Ti₁₆NbXSn sintered alloys. The results indicated that Ti₁₆Nb(0–4)Sn alloys were composed of $\alpha + \beta$ phases. Hardness and Young's modulus E of the alloys measured using nanoindentation technique. These results show that there is a relationship between the mechanical properties and Nb–Sn content. It is concluded that addition of Nb to the cp-Ti resulted to a decrease in Young's modulus.

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PACS/topics: powder metallurgy, TiNbSn alloys, implant

1. Introduction

Metallic biomaterials are widely used as implant materials under load-bearing conditions [1]. A crucial challenge for the development of metallic orthopedic implants is to obtain a material that combines high mechanical strength, unique fatigue behavior, enhanced biocompatibility and high biocorrosion resistance [2]. Ti alloys have been gradually replacing the stainless steels and Co–Cr alloys in clinical applications [3]. The Ti–6Al–4V ELI alloy is the most widely used titanium alloy to date. However, two main problems arise with the use of Ti–6Al–4V ELI Al and V have higher cytotoxicity. On the other hand, the substantially higher elastic modulus compared to that of natural bone. This gives rise to the so-called “stress shielding” effect that can cause bone resorption and loosening of implant. Recent research has attempted to overcome the long-term health problem caused by the release of the toxic ions from the alloys as well as the stress shielding effect. New titanium alloys containing only non-toxic metallic alloying elements (Nb, Ta, and Zr, Sn etc.), with excellent mechanical properties are being developed as potential implant material [4]. Using PM based approaches the traditionally high processing cost of titanium can be significantly reduced in several aspects. Sintering can achieve this by removing the melting step and lowering the potential for contamination at high temperature. The near-net-shape aspect of PM further minimises or eliminates the need for final machin-

ing [5]. The aim of this study was to investigate the effects of Sn content (2 and 4 wt%) on the microstructure and mechanical properties of Ti₁₆Nb(0–4)Sn wt% as-sintered alloy samples.

2. Materials and methods

Elemental metal powders of Ti, Nb, Sn were used as starting materials. The details of the equipment and general compacting, sintering procedure and microstructural characterization have been explained elsewhere [6]. After sintering, densities of sintered samples were measured according to Archimedes' water immersion method. Samples for metallographic examination were prepared. X-ray diffraction (XRD) patterns were obtained by means of a RIGAKU D/Max 2200 diffractometer. Hardness and Young's modulus of the alloys were measured by using a CSM Instruments Nanoindentation tester equipped with Berkovich-type diamond indenter at a maximum applied load of 80 mN.

3. Results and discussion

For all specimens, at the 400 MPa compaction pressure, the achieved density was $\approx 70\%$ of the theoretical value. Preparing samples at the highest pressure raises the risk of tooling wear and punch jamming. As a consequence, the compaction pressure could be practically limited to 400 MPa, which provides adequate green strength for handling and reduces the risk of die damage. After sintering (1500 °C), specimens with relative density up to 99% have been produced. In powder metallurgy works, increase in density with sintering is an expected result [2]. The sintering densities obtained in this study

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are compatible with the literature [5]. The porosity values obtained in the samples are compatible with the human cortical bone (1–16%), the porosity of the cortical bone increases with age [7]. Figure 1a–c presents the microstructural evolution of the samples sintered at temperatures 1500 °C. It is seen that as-sintered cp-Ti is formed as a single phase. Pure Nb and pure Sn cores were found completely dissolved into the $\alpha + \beta$ Widmanstätten matrix which is in accordance with XRD results (Fig. 2b). Morphology of sintered alloys is similar. Previous works on Ti-Nb based alloys showed that Nb

particles were not completely soluble in the matrix at low sintering temperatures (900–1200 °C) [8]. Also increasing in sintering temperature led to the dissolution of the niobium particles in the matrix and this event continues causes to an increment in the volume fraction of the Widmanstätten ($\alpha + \beta$) structure. Therefore, for mentioned alloys, sintering temperatures below the 1300 °C would not be sufficient to effectively sinter the particles. At 1500 °C, the Widmanstätten-like ($\alpha + \beta$) structure is distributed all over the alloys resulting in a homogeneous microstructure.

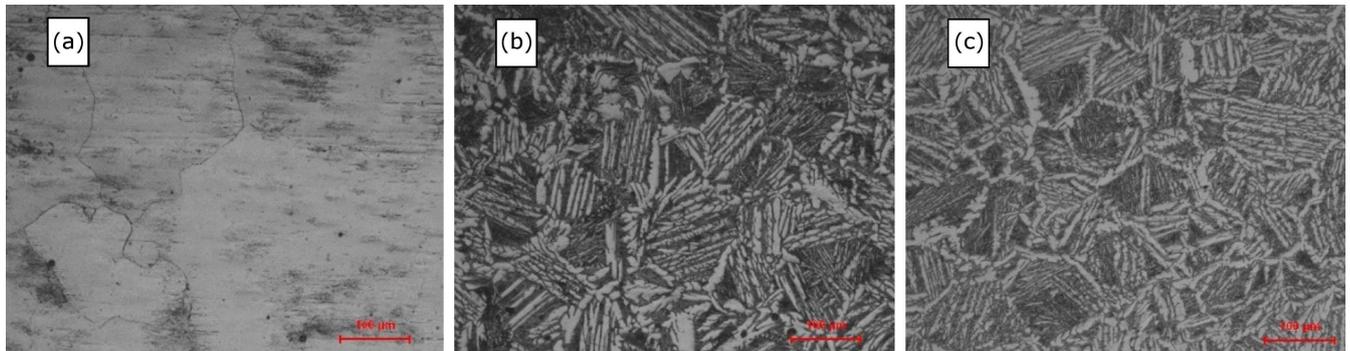


Fig. 1. Optical micrographs of sintered (a) cp-Ti, (b) Ti-16Nb, (c) Ti-16Nb-2Sn.

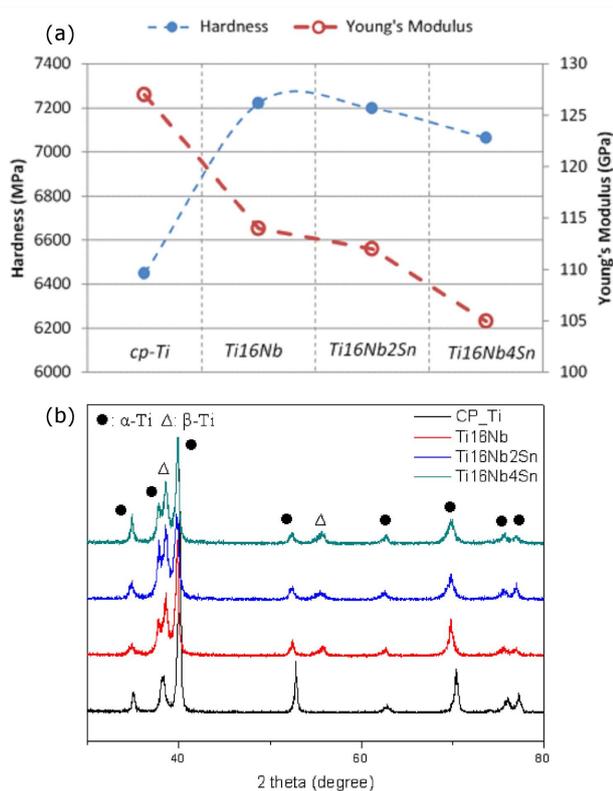


Fig. 2. (a) Mechanical properties, (b) XRD patterns of as-sintered alloys.

The mechanical properties of cp-Ti and Ti16Nb(0–4)Sn alloys are presented in Fig. 2a. The

hardness of cp-Ti is measured lower than Ti16Nb(0–4)Sn alloys, but Young's modulus of cp-Ti is measured higher than Ti16Nb(0–4)Sn alloys. The β -type Ti-based alloys possess more excellent combination of high strength and low modulus as well as higher plasticity compared with α type Ti-based alloys [3]. For this reason it is an expected result. With addition of 16 wt% Nb into cp-Ti, Young's modulus decreased from 127 GPa to 114 GPa. In β -type Ti alloys, the increase of β stabilizing element such as Nb, reduces the elastic modulus of the alloy by reducing the lattice bond strength [9]. In previous works on the addition of Nb to $\alpha + \beta/\beta$ type Ti alloys, Nb has been found to be effective in reducing Young's modulus [1]. As seen in Fig. 2a, as the Sn was added to the Ti-16Nb alloy, there was decrease in the hardness and Young's modulus. In this respect, Sn β stabilizing effect and changes in crystal structure size were effective. Hardness and Young's modulus depend on the interatomic forces. Planar distance in the crystal structure of β phase was increased from 2.3284 Å to 2.3320 Å by the addition of 4 wt% Sn to Ti16Nb alloy. An increase in the interatomic distance results in a drop in Young's modulus. Produced this Ti16Nb4Sn alloy can better improve the stress transmission between bone and the implant than cp-Ti. It is very important for inhibiting bone resorption and enhancing the remodelling of bones.

4. Conclusions

The effect of Sn addition to the Ti16Nb alloy was investigated. For this purpose, microstructure analyzes and mechanical tests were performed. Relative density over

99% was obtained for alloys sintered at 1500 °C. Microstructures of the sintered alloys are composed of Widmanstätten ($\alpha + \beta$) structure. Nb and Sn were found to be effective in β stabilizers. The hardness of Sn alloyed materials is measured as higher than that of cp-Ti, while Young's modulus of cp-Ti is measured as highest one. It is thought that β stabilizing effect of Sn and changes in the crystal structure size of β phase resulted to a decrease in Young's modulus.

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References

- [1] S. Ehtemam-Haghighi, Y. Liu, G. Cao, L.C. Zhang, *Mater. Sci. Eng. C* **60**, 503 (2016).
- [2] E.B. Taddei, V.A.R. Henriques, C.R.M. Silva, C.A.A. Cairo, *Mater. Sci. Eng. C* **24**, 683 (2004).
- [3] Y. Li, C. Yang, H. Zhao, S. Qu, X. Li, Y. Li, *Materials (Basel)* **7**, 1709 (2014).
- [4] A.T. Sidambe, *Materials (Basel)* **7**, 8168 (2014).
- [5] H.-W. Liu, D.P. Bishop, K.P. Plucknett, *Can. Metall. Q.* **52**, 39 (2013).
- [6] E. Yılmaz, A. Gökçe, F. Findik, H. Ö. Gülsoy, *Pamukkale Univ. J. Eng. Sci.* **23**, 945 (2017).
- [7] V. Bousson, C. Bergot, A. Meunier, *Radiology* **217**, 179 (2000).
- [8] D. Zhao, K. Chang, T. Ebel, H. Nie, R. Willumeit, F. Pyczak, *J. Alloys Comp.* **640**, 393 (2015).
- [9] K.A. Nazari, A. Nouri, T. Hilditch, *Mater. Des.* **88**, 1164 (2015).