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Development of a new metastable beta titanium alloy for biomedical applications

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Abstract

The paper presents experimental results of the effect of the multi-pass rolling process in different temperatures and deformation percents on the microstructure, mechanical and corrosion characteristics of a new titanium (Ti) alloy. The results of mechanical and corrosion properties were optimum through deforming the Ti alloy in warm conditions owing to the formation of ultrafined structure. This paper demonstrated that the investigated Ti alloy is a prospective candidate for biomedical applications due to its better mechanical and corrosion properties.

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Keywords: Titanium; Rolling; Mechanical properties; Corrosion; Biomedical applications

1. Introduction

Among metallic materials, Ti alloys are receiving much interest for various applications, especially in medicine owing to their relatively low density, excellent biocompatibility, better mechanical performance, low Young's moduli and superior electrochemical behavior [1–3]. Commercial pure titanium (CP–Ti) and the most commonly alloy used ($\alpha + \beta$) Ti– 6Al–4V are the main Ti materials used in biomedical applications as orthopedic implants. However, from the point of view of mechanical performance, biocompatible CP–Ti is poor for some applications where high strength and wear resistance are required [4]. On the other hand, the main alloy elements in Ti-6Al-4V alloy (Al and V) can be released from the alloy during service causing neurological disorders, toxicity in human tissues [5] and delay in bone integration after implantation [6]. Moreover, CP-Ti and $(\alpha + \beta)$ Ti6Al4V alloy have higher elastic modulus (at around 105 and 110 GPa respectively), comparing with the cortical bone (10-30 GPa) [2,3,7], which leads to stress shielding effect and absolutely bone loss and implant's failure [1,2,8]. Therefore, it is essential to design novel alloys from biocompatible elements with appropriate micro-structural features and higher biomedical characteristics. Recently, metastable β-Ti alloys which compose of non-toxic elements such as Zr, Nb, etc. are successfully developed as implantable materials because of the best combination of the above requisite characteristics [2,7,8]. In addition, the

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presence of β phase in these alloys provides excellent cold workability with an isotropic mechanical behavior due to of body centered cubic (bcc) structure [9]. In this regard, Ti-Zr-Nb alloy system (ASTM F1713-08) is developed originally from biocompatible and non-toxic elements as new implantable Ti alloy [10]. Zirconium and niobium is the main β -phase stabilizers in this alloy. It is important to mention here that Zr and Ti have unlimited solubility in each other since they have the same crystal structure [11]. On the other hand, Nb has an ability to form a homogenous solid solution with all types of Ti [12]. Therefore, in recent years, brilliant attempts are being devoted to develop a number of biomedical β-type Ti-Zr-Nb alloy systems with high mechanical properties and superior electrochemical behavior [13-16]. However, the strength of Ti-Zr-Nb alloy system is still low in comparison with α or $(\alpha + \beta)$ -type Ti alloys, thus it needs to increase with maintaining lower Young's modulus and corrosion rate. In this study, the microstructure of a new alloy Ti-15Zr-12Nb (TZN) (in wt.%) was modified through warm rolling (WR) and hot rolling (HR) processes. The influence of the microstructure on the mechanical and electrochemical performances of this alloy was also evaluated.

2. Material and methods

In this study, Ti-15Zr-12Nb (TZN) alloy was manufactured using non-consumable vacuum arc melting method. The mixture of sponge Ti, zirconium chips and niobium powder is used as raw materials. The melting process was carried out in copper crucible under ultra-high purity argon gas. The casting operation was repeated three times to get high homogeneity in chemical composition.

In a dynamic argon condition, the alloy samples were solution treated at 880 °C for 1 h followed by iced water quenching. The alloy samples were then deformed in two different thermal conditions. Some samples were deformed by warm rolling (WR) at 660 °C for 1 h up to the final thickness reduction ratio of 95% using a multi-pass rolling process. Afterward, the rest samples were deformed by hot rolling (HR) from 880 °C to 660 °C up to the final thickness reduction ratio of 70% using same rolling process.

Micro-structural investigation of the samples was carried out using NOVA NANO SEM 450 FEI, Netherlands at 2 kV. The energy dispersive spectroscopy (EDS) mapping was also obtained. The metallographic rules were made on the samples using silicon carbide grinding up to 1200 grit, and a mirror polishing via 0.5 μ m diamond paste. Kroll's reagent (5 vol% HF and 5 vol% HNO₃ in water) was used for etching the polished samples. X-ray diffraction analysis at room temperature was achieved using an X-ray Diffractometer, Bruker, German, D8 Advance with Cu K α radiation at 40 kV and 30 mA.

Vickers micro-hardness test was evaluated using a standard tester (model: MicroWhizHard, Mitutoyo, Japan) with an applied load of 300 gf and a dwell time of 5 s for each indents. For this test, mirror-polished samples were achieved using a diamond paste of 0.5 μ m. The average value of five measurements was taken for each sample.

Mechanical properties were estimated through tensile test (ASTM E8 M) with a constant strain performed at ambient temperature using a computerized universal testing machine (Model: UTE-60, FIE). The elastic modulus (E) was achieved via determining the slope of the straight line of stress—strain curve. The tensile samples with 10 mm width, 4 mm thickness and a gage length of 25 mm were precisely prepared. Mechanical grinding was made for tensile samples using SiC papers of up to #2500 grit and the gage length was polished using 0.5 μ m diamond paste.

The electrochemical properties of the investigated TZN samples were evaluated using computerized three-electrode cell potentiostat (model: WPG100e, Korea). Open circuit potential (OCP), corrosion current density (I_{corr}), and corrosion rate were used for estimating the performance of the corrosion. The samples were ultrasonically cleaned after standard grinding and polishing processes using waterproof SiC papers of up to #2500 grit and diamond paste of 0.5 µm respectively. For each experiment, 0.126 cm² surface area was exposed to fresh and naturally aerated solution of 0.9% NaCl at 7.4 pH and 37 \pm 1 °C to simulate physiological environment. The OCP was determined as a function of time until the steady state was attained. Moreover, corrosion potential (Ecorr) and Icorr of the TZN samples were estimated from the polarization curve of potential vs. current density via the extrapolation technique. A scan rate of 0.166 mV/s in the range from -750 to 2500 mV(Ag/AgCl) was used through the test.

3. Results and discussion

3.1. Chemical composition analysis

The results of the chemical composition (in wt.%) of the Ti-15Zr-12Nb alloy were obtained using an EDS spectrometer as shown in Table 1.

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