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Mechanical properties of a medical β-type titanium alloy with specific microstructural evolution through high-pressure torsion

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ABSTRACT

The effect of high-pressure torsion (HPT) processing on the microstructure and mechanical biocompatibility includes Young's modulus, tensile strength, ductility, fatigue life, fretting fatigue, wear properties and other functionalities such as super elasticity and shape memory effect, etc. at levels suitable for structural biomaterials used in implants that replace hard tissue in the broad sense (Sumitomo et al., 2008 [4]). In particular, in this study, the mechanical biocompatibility implies a combination of great hardness and high strength with an adequate ductility while keeping low Young's modulus of a novel Ti–29Nb–13Ta–4.6Zr (TNTZ) for biomedical applications at rotation numbers (N) ranging from 1 to 60 under a pressure of 1.25 GPa at room temperature was systematically investigated in order to increase its mechanical strength with maintaining low Young's modulus and an adequate ductility.

TNTZ subjected to HPT processing ($TNTZ_{HPT}$) at low N exhibits a heterogeneous microstructure in micro-scale and nano-scale consisting of a matrix and a non-etched band, which has nanosized equiaxed and elongated single β grains, along its cross section. The grains exhibit high dislocation densities, consequently non-equilibrium grain boundaries, and non-uniform subgrains distorted by severe deformation. At high N which is $N>20$, $TNTZ_{HPT}$ has a more homogeneous microstructure in nano-scale with increasing equivalent strain, ε_{eq}. Therefore, $TNTZ_{HPT}$ at high N exhibits a more homogenous hardness distribution. The tensile strength and 0.2% proof stress of $TNTZ_{HPT}$ increase significantly with N over the range of $0 \le N \le 5$, and then become saturated at around 1100 MPa and 800 MPa at $N \ge 10$. However, the ductility of $TNTZ_{HPT}$ shows a reverse trend and a low-level elongation, at around 7%. And, Young's modulus of $TNTZ_{HPT}$ decreases slightly to 60 GPa with increasing N and then becomes saturated at $N \ge 10$. These obtained results confirm that the mechanical strength of TNTZ can be improved while maintaining a low Young's modulus in single β grain structures through severe plastic deformation.

1. Introduction

Mechanical biocompatibility, which includes Young's modulus, ductility, fatigue life, fretting fatigue, wear properties and other functionalities such as super elasticity and shape memory effect, etc. has been needed at suitable levels for structural biomaterials used in implants that replace hard tissue in the board sense [1,2]. In this study, the mechanical biocompatibility, which implies a combination of great hardness and high strength with an adequate ductility while keeping low Young's modulus close to that of bone (10–30 GPa), has gained attention to ensure the long-term stability of metallic biomaterials in surgical implantation [3]. In particular, the large mismatch of Young's moduli can cause "stress shielding" in which the stress transfer between an implant device and a bone is not homogeneous when Young's moduli of the implant and the bone are different. In such conditions, bone atrophy occurs and leads to the loosening of the implant and refracturing of the bone [4]. Therefore, the Young's modulus of metallic biomaterials should be low close to that of the bone.

A novel β-type titanium alloy, Ti–29Nb–13Ta–4.6Zr (TNTZ), which consists of non-toxic and non-allergic elements like Nb, Ta, and Zr, has been developed as an alternative of the conventional metallic biomaterials such as stainless steel, Co–Cr–Mo alloy, and the abovementioned titanium alloys [3]. A high degree of the mechanical biocompatibility in TNTZ is expected to be achieved by microstructural fine tuning [4]. The effects of conventional processes such as solution treatment, cold rolling and aging treatment on the microstructural and mechanical properties of this alloy have been extensively studied [5]. Although TNTZ exhibits a low Young's modulus (~60 GPA) in its metastable β, which is formed after solution treatment and cold rolling, its strength is less than that of Ti64 ELI [5]. Furthermore, aging treatment is a simple way to improve strength drastically because of the precipitation strengthening of α and/or ω secondary phases. However, Young's

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modulus increases simultaneously and becomes relatively high due to higher Young's moduli of the α and ω phases than that of the β phase, which is the matrix phase of TNTZ [5]. Therefore, new methods of achieving this high mechanical biocompatibility have been studied for TNTZ [6–8].

Recently, severe plastic deformation (SPD) techniques such as equal-channel angular pressing (ECAP) [9] and high-pressure torsion (HPT) [9,10] have been developed to produce ultrafine-grained (100–500 nm) [11] or nanostructured (<100 nm) [11,12] bulk materials. These materials are expected to exhibit relatively better mechanical properties than that of the materials having coarse grains [9–13]. The microstructures of metallic materials can be remarkably refined by dynamic re-crystallization through accumulating and rearranging of dislocations through SPD processing [9,10]. Among SPD techniques, the HPT processing developed by Bridgman [10] is the most effective to imparting especially nanostructured grains and extra-high dislocation density by severe torsional straining [10].

The HPT processing is effective for producing ultrafine-grained TNTZ having dislocation density in single β structure [14] even though BCC metals, for example molybdenum [15], chromium [16], and tungsten [17], exhibit a slower refinement progress than most of the FCC metals such as pure aluminum, copper, and nickel [9,10] due to their lower stacking fault energies. However, TNTZ subjected to HPT processing ($TNTZ_{HPT}$) at rotation numbers (N) up to 20 exhibits a heterogeneous microstructure and consequently a heterogeneous hardness distribution on the surface and the cross section of coin-shaped specimens [14]. Furthermore, the HPT processing may be applied repeatedly to impose exceptionally high straining, since the dimensions of TNTZ sample practically do not change in HPT processing [10].

The aim of this study is to improve the mechanical biocompatibility of TNTZ by imparting to it high strength while maintaining a low Young's modulus through homogeneous grain refinement and dislocation accumulation. Also, a homogeneous microstructure is strongly needed to achieve the same mechanical properties in every area of metallic specimen. Consequently, a higher imposed straining, which means very high N ($N>20$), is needed in order to achieve homogeneous microstructure for TNTZ due to its slow refinement behavior. Therefore, the microstructural evolution and its effect on mechanical properties of TNTZ through HPT processing at very high N are investigated systematically in this study.

2. Experimental procedures

2.1. Materials

The material used in this study was a hot-forged TNTZ bar with a diameter of 25 mm and a length of 50 mm. Its chemical composition is listed in Table 1. The bar was subjected to solution treatment at 1063 K for 3.6 ks in vacuum followed by water quenching ($TNTZ_{ST}$). Then the resultant $TNTZ_{ST}$ was cold-rolled into a plate with a thickness of 0.8 mm (reduction ratio > 80%) ($TNTZ_{CR}$). Finally, the $TNTZ_{CR}$ was machined into coin-shaped specimens with a diameter, R, of 20 mm for HPT processing.

2.2. HPT processing

The coin-shaped specimens of $TNTZ_{CR}$ subjected to HPT processing between the two anvils in opposition vertically by rotating the lower anvil at 0.2 rotations per minute (rpm) for the rotation numbers, N, of 1, 5, 10, 20, and 60 at room temperature under a pressure of 1.25 GPa.

Table 1
Chemical composition of hot-forged TNTZ (mass %).

Nb	Ta	Zr	C	N	O	H	Fe	Ti
28.6	12.3	4.75	0.02	0.01	0.09	0.04	0.22	bal.

Herein, the coin-shaped specimens of TNTZ subjected to HPT processing are referred to as $TNTZ_{HPT}$.

The equivalent strain (ε_{eq}) in the coin-shaped specimen subjected to HPT processing is given by the following equation (10, 18):

$$\varepsilon_{eq} = ((2\pi rN)/(t\sqrt{3})) \quad (1)$$

where r is the distance from the specimen center, π is the ratio of the circumference of a circle to its diameter, and t is the specimen thickness. Therefore, the ε_{eq} that results from HPT processing changes as a function of r and N, and t for the coin-shaped specimen.

2.3. Material characterization

The microstructure of each specimen was evaluated using an optical microscopy (OM), an X-ray diffractometer (XRD), and a transmission electron microscope (TEM). The specimens, which were examined through the OM observation, were wet-polished using waterproof emery papers of up to #4000 and were then buff-polished to obtain a mirror surface by colloidal SiO_2 suspension. Subsequently, they were etched by a 5% HF etching solution. Fig. 1(a) shows a schematic illustration of the areas that were used for the microstructural evaluation. XRD analysis and TEM observations were carried out at a half radius, r_h, position, $r=r_h=5$ mm, along a cross section of each specimen. Fig. 1(c) shows an illustration of a final TEM specimen on cross section whose preparation is detailed in a previous study [14]. TEM observations were carried out with an accelerating voltage of 200 kV. The grain diameter for equiaxed grains, and length and width for elongated grains were quantitatively measured by approximating every grain by counting 100 grains taken randomly from TEM bright field images. The error bars represent the standard deviation of grain-diameter, length and width measurements.

The phase constitution and the texture of each specimen were analyzed by XRD using a Cu–Kα radiation tube with a voltage of 40 kV and a current of 40 mA. The specimens, which were examined by XRD analysis, were polished up to a mirror surface. For texture analysis, the distribution of the diffraction intensities from two crystal planes β {110} and β {200} were analyzed for the surfaces of the coin-shaped specimens at the r_h position.

2.4. Mechanical tests

Hardness (HV) measurements were carried out using a micro-Vickers hardness tester with a load of 500 g for a dwell time of 15 s along the surface and cross section of a coin-shaped specimen as shown in Fig. 1(a). The measurements along the surface of the coin-shaped specimen were performed at the intervals of 1 mm and 22.5° between the measurement positions in the radial direction, respectively. Measurements along the cross section of the specimen were performed at intervals of 1 mm between the measurement positions in the radial direction, respectively. The tensile properties of the specimens, which were machined at the r_h position of the coin-shaped specimens shown in Fig. 1(b) were evaluated using an Instron-type testing machine with a cross-head speed of 8.33×10^{-6} m·s^{-1} at room temperature. The fracture surface was analyzed using a scanning electron microscope (SEM). In order to improve the accuracy of the measurements, Young's modulus was determined from the stress–strain curves that were recorded using a strain gage.

3. Experimental results

3.1. Microstructure

3.1.1. Microstructure observed by OM

Optical micrographs on the cross section of $TNTZ_{HPT}$ at $N=60$ are shown in Fig. 2. After HPT processing, the microstructure has a unique