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Original Article

Enhancement in mechanical properties of a β-titanium alloy by high-pressure torsion☆

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ABSTRACT

Titanium alloys, mainly Ti–6Al–4V, are commonly used in biomedical applications as orthopedic implants. Due to the potential toxic influence of V and Al cations on health, a new alloy composition, Ti–24Nb–4Zr–8Sn, was introduced. However, Ti–24Nb–4Zr–8Sn has a much lower tensile strength by comparison with the Ti–6Al–4V alloy. The aim of this research was to determine whether high-pressure torsion (HPT) can be an efficient method for obtaining the desired properties in the case of the Ti–24Nb–4Zr–8Sn β-titanium alloy. This paper presents an analysis of the microstructural and mechanical properties of the Ti–24Nb–4Zr–8Sn alloy processed by HPT with various processing parameters. The obtained microstructures were examined using transmission electron microscopy (TEM). Mechanical properties, such as hardness and tensile strength, were also measured. The study demonstrates that HPT of the Ti–24Nb–4Zr–8Sn alloy leads to a significant reduction of grain size and this grain refinement gives a major improvement in tensile strength and hardness.

1. Introduction

Titanium alloys are commonly used in biomedical applications as orthopedic implants due to their good mechanical properties, low density and biocompatibility [1,2]. It has been reported that Al can cause neurological pathologies and V exhibits cytotoxic effects [3], therefore the most widely used Ti alloy in medicine, Ti–6Al–4V, is not fully biocompatible. Additionally, the value of the Young's modulus of this $\alpha+\beta$ microstructure alloy is significantly higher than that of the natural bone, which leads to a stress shielding effect [4].

There is a new generation of β-titanium alloys, aluminum and vanadium free, composed with elements stabilizing phase β, such us Ta, Nb and Zr. The Ti–24Nb–4Zr–8Sn alloy (wt%, abbreviated as Ti2448), a β-type titanium alloy with high

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strength and low Young's modulus, has been recently developed for biomedical applications [5,6]. The alloy exhibits good corrosion resistance by comparison with commercially pure Ti and the commonly used Ti–6Al–4V alloy. It has been reported that Nb, Zr and Sn are non-toxic and non-allergic alloying elements [7] and in vivo studies of the alloy show its excellent biocompatibility [8]. However, the tensile strength of the alloy is much lower in comparison to the commonly used Ti–6Al–4V alloy. Therefore, there is a necessity to improve the strength properties while maintaining the favorable low value of Young's modulus.

Severe plastic deformation processes (SPD) have been widely investigated and are recognized as valuable methods of obtaining an ultra-fine-grained or nanocrystalline microstructure [9,10]. Among several different SPD processes, high-pressure torsion (HPT) merits special attention due to the possibility of obtaining exceptionally small grains and high strength [11]. HPT process was first introduced over 70 years ago by Bridgman [12,13] but the technique has been widely researched only within the last twenty years. The schematic illustration of this technique is shown in Fig. 1: the disk is placed between two massive anvils and is subjected to a severe compressive pressure. HPT processing is then performed by imposing torsional straining through rotation of the lower anvil. It is worth mentioning that in this quasi-constrained method there is a small gap between the anvils so that a limited volume of material is forced outwards, as illustrated in Fig. 2 [14,15].

The range of literature covering the Ti–24Nb–4Zr–8Sn β-alloy is not very extensive and at present there are no papers describing or analyzing the high-pressure torsion process performed on this alloy. Taking into consideration the increasing interest in biomedical titanium β-alloys, this research area is well worth developing.

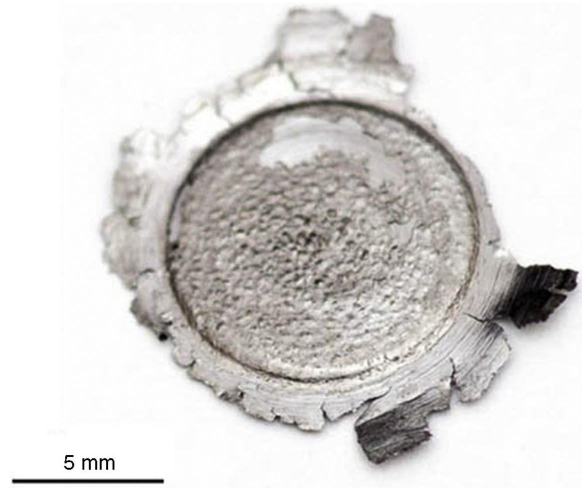

Fig. 2 – Sample processed by quasi-constrained high-pressure torsion.

2. Methods

The researched material was biomedical Ti–24Nb–4Zr–8Sn β-alloy (Einsal East, Poland), heat-treated at 750 °C for 1 h followed by air cooling to obtain a coarse grain microstructure (see Fig. 3). The HPT process was performed on polished disks with diameters of 9.9 mm and thicknesses of 0.75–0.85 mm at room temperature under quasi-constrained conditions. Disks were processed under a pressure of 3.0 GPa, through 1, 5, 10, 20 and 50 turns, using a rotation speed of 1 rpm.

In order to perform microstructural characterization, disks of 3 mm diameter were cut from the center and from the half-radius position of HPT samples, parallel to the plane of the disks, and then electro-polished. Thin foils were examined using TEM JEOL JEM 1200 EX.

The Vickers microhardness measurements were conducted on ground and mirror-like polished samples, across the diameter of each disk in steps of 0.3 mm, under a load of 100 g, using a Zeiss microhardness tester equipped with a Vickers indenter tip.

The mechanical properties were investigated using tensile testing at room temperature. Two miniature tensile samples with gauge lengths of 2.5 mm were cut from each disk from an off-center position, as shown in Fig. 4. Electrical discharge machining (EDM) was used to cut the miniature tensile samples from the disk to obtain the required dimensions and to avoid damage during cutting. Tensile tests were conducted using a Zwick/Roell 005 testing machine at an initial strain rate of $1.0 \times 10^{-3}\,s^{-1}$, with optical non-contact displacement

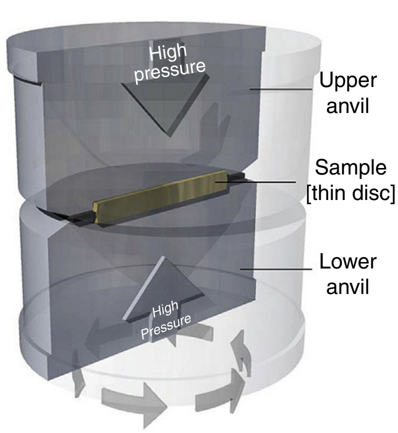

Fig. 1 – Schematic illustration of HPT process.

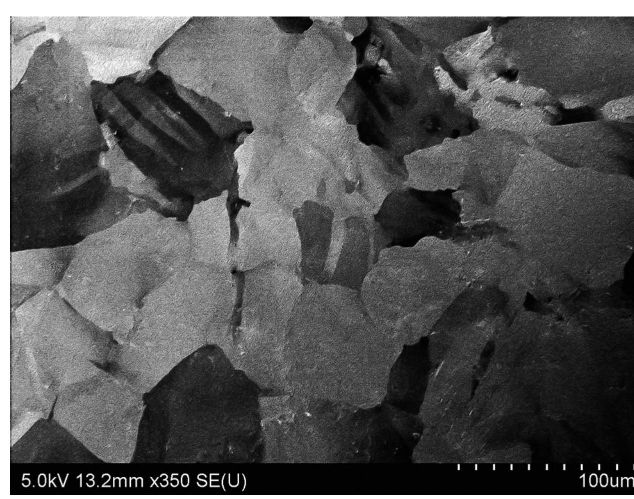

Fig. 3 – The initial annealed microstructure of the Ti–24Nb–4Zr–8Sn alloy prior to HPT.

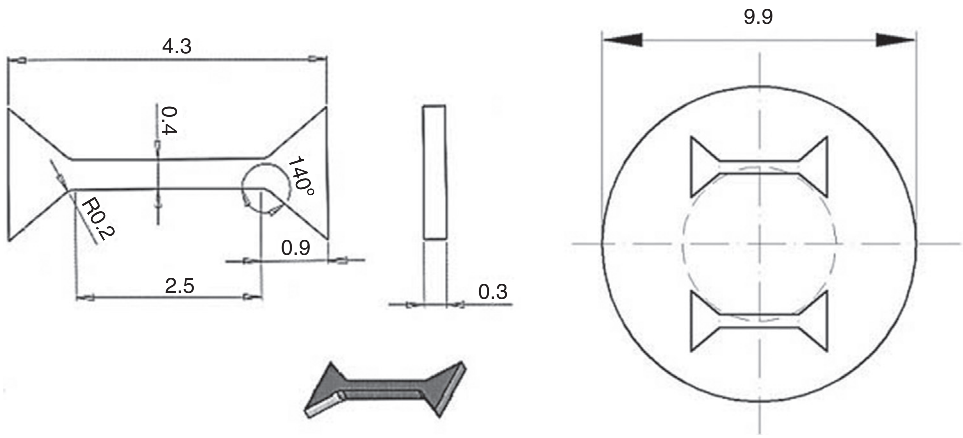

Fig. 4 – Schematic illustration of tensile specimen cut from the off-center area.

measurements by means of Digital Image Correlation (DIC) for engineering strain tracking [16]. All samples were pulled to failure in tension, at room temperature, at a constant rate of crosshead displacement.

3. Results and discussion

3.1. Microstructural evolution

Fig. 5 shows the microstructures at the half-radius position after (a) 1 and (b) 50 turns. HPT processing with only one turn results in a non-homogenous microstructure with a high density of dislocations, relatively large (about 500 nm) subgrains and a small fraction of fine grains (with a diameter of about 50 nm), as illustrated in Fig. 5a. After 50 turns, a highly refined and homogenous microstructure can be observed (Fig. 5b) and dark field images reveal very small grains with diameters ranging from 20 to 50 nm. The diffraction patterns (presented as inserts) also indicate more pronounced microstructure refinement for the sample processed with 50 turns as well as large misorientation angles between grains (continuous diffraction rings).

3.2. Microhardness measurements

The values of the Vickers microhardness measured across the diameter of the samples are shown in Fig. 6. Each point on

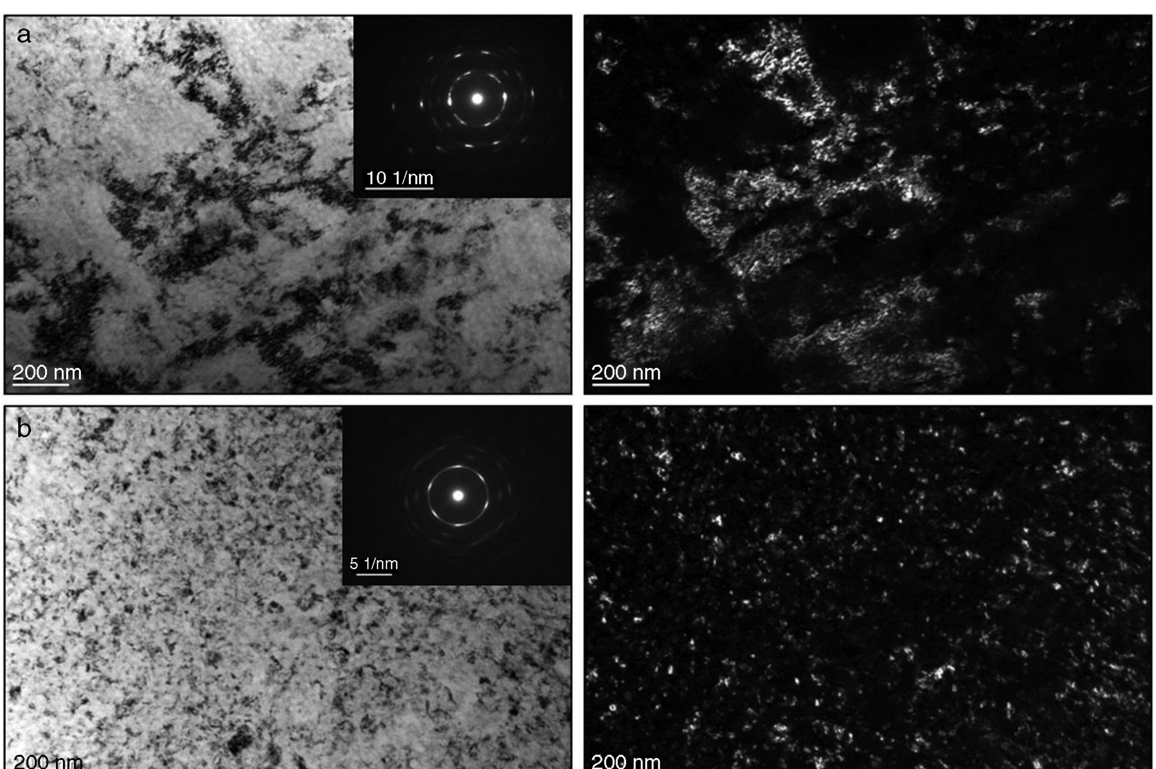

Fig. 5 – Transmission electron micrographs illustrating the microstructures after HPT at the 1/2 radius position for the samples processed with: 3 GPa and 1 turn (a) and 3 GPa and 50 turns (b).

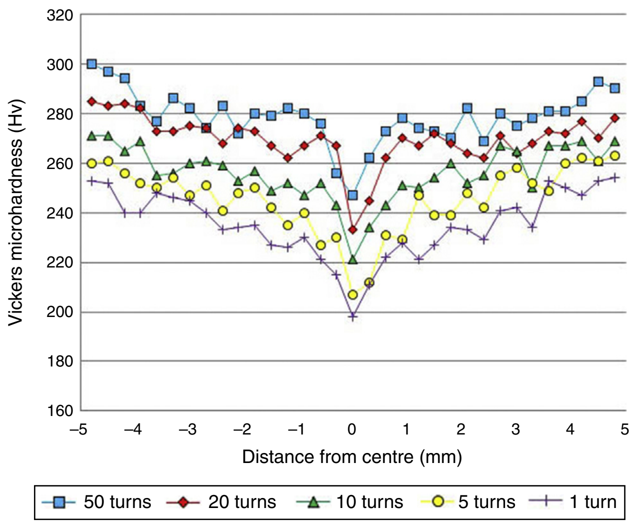

Fig. 6 – Distribution of the average Vickers microhardness across the diameter of the HPT samples processed for various numbers of turns.

the plot is an average of three measurements recorded at the same distance from the center. The broken line illustrates the value for the annealed and unprocessed alloy.

After one turn, the microhardness increases significantly at the edges of the disk (by 60 Hv) and gradually decreases toward the center, which is due to the strain distribution along the radius. For higher number of turns, a characteristic plateau of microhardness distribution along the radius is observed. It should be noted that there is no saturation of microhardness even after 50 turns.

3.3. *Mechanical properties*

Fig. 7 shows tensile engineering stress–strain curves before and after HPT. The material in the initial state exhibits high elongation and relatively low tensile strength, typical for the coarse-grain microstructure. HPT induces major changes in mechanical properties of the material. After only 1 turn under

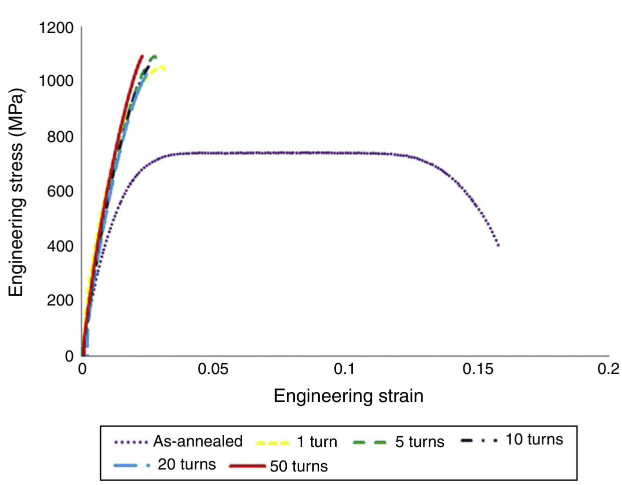

Fig. 7 – Tensile engineering stress–strain curves of Ti–24Nb–4Zr–8Sn alloy before and after HPT.

3 GPa, the tensile strength increases significantly to a value of 1050 MPa. However, the elongation to failure decreases drastically in comparison to the as-annealed alloy. Further straining does not cause substantial changes and the tensile strength increases only slightly. Samples processed by HPT display typical brittle fracture behavior.

The value of Young's modulus of the alloy before processing equals to 52 GPa. Processing by HPT leads to a slight increase in the Young's modulus value by approximately 10% with no evident correlation to the number of turns.

4. Conclusions

Disks of the Ti–24Nb–4Zr–8Sn β-alloy were processed by high-pressure torsion at room temperature using an applied pressure of 3 GPa through up to 50 turns. The microstructural evolution and mechanical properties were studied.

(1) Processing by HPT brings about a significant microstructural refinement, gradually progressing with increasing numbers of turns.
(2) Grain size refinement induces major changes in the mechanical properties. The tensile strength increases from about 700 MPa in the initial state to over 1000 MPa after processing while the Young's modulus remains at a relatively low level (less than 60 GPa).
(3) Vickers microhardness measurements revealed that HPT leads to a significant growth of this value. Further straining introduces a continuous increase, but with no saturation.

The combination of the mechanical properties (high strength and low Young's modulus) obtained via HPT processing makes the Ti–24Nb–4Zr–8Sn β-alloy promising for biomedical applications as an implant material.

Conflicts of interest

The authors declare no conflicts of interest.

Acknowledgements

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