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A high Zr-containing Ti-based alloy with ultralow Young's modulus and ultrahigh strength and elastic admissible strain

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ABSTRACT

Ideal metallic biomaterials for use in implants are required to meet the mechanical compatibility, i.e., low Young's modulus combined with high mechanical strength, however, these constraints are generally contradictory. Here, an exceptional combination of ultralow Young′s modulus of $E \sim 45$ GPa, ultrahigh tensile strength of $\sigma_b \sim 1185$ MPa and ultrahigh elastic admissible strain of $\delta \sim 2.2\%$ is achieved in a new high Zr-containing Ti-based alloy (Ti-50Zr-5Al-4V (wt%)), which is subjected to solution treatment and cold rolling. The ultralow Young′s modulus is attributed to a mixture of bcc β and orthorhombic α″ phases while the ultrahigh strength results from the solution strengthening and work hardening. This study can be of significance for developing excellent removable implant materials.

1. Introduction

The demand for metallic biomaterials is increasing for improving the quality of life and longevity of an increasing aging population [1], and conventional metallic biomaterials such as stainless steels (SUS316L), cobalt chromium alloys, and Ti-6Al-4V extra low interstitials (ELI) alloy have been widely used in the biomedical field for many years [1,2]. However, in order to guarantee good osseointegration between implants and bone tissues and avoid revision surgeries, metallic biomaterials used as orthopedic implants should be of mechanical compatibility [1,2]. This implies that the elastic modulus of metallic biomaterials must be as close to that ($E \sim 20$–30 GPa) of natural bones as possible to eliminate the stress-shielding effect, which could cause osteoporosis or poor osseointegration [3]. Based on this scenario, many new near β and β titanium alloys have been recently developed [4–10], which exhibit lower Young′s modulus ($E \sim 55$–85 GPa) compared with conventional metallic biomaterials, e.g., Ti-6Al-4V ELI alloy with a Young's modulus of $E \sim 110$ GPa [11].

In addition to low elastic modulus, implant materials are also required to possess high mechanical strength [1,2], for example, the tensile strength should be approximately ∼1000 MPa [12], because the occurrence of yield behavior means the failure of implants. However, these constraints of low elastic modulus and high strength are usually contradictory and the variation trends of elastic modulus and strength are generally consistent [5,6,13]. To escape from this contradiction and achieve a combination of low elastic modulus and high strength, previous studies indicate that solid solution treatment [6], proper composition selection [14], and cold working [15,16] are possible strategies. For example, several high Zr-containing (25–40 wt%) Ti-based alloys with low Young′s modulus and high strength have been recently developed for temporary orthopedic devices [4,5,10]. This can be closely related with the effect of high content of Zr, which has been confirmed to be capable of enhancing the strength and reducing the modulus by theoretical studies [14] and of enhancing the cytocompatibility of Ti alloys due to the formation of Zr oxide by experimental studies [17]. Furthermore, some studies show that Zr can prevent calcium phosphate formation, which can accelerate the assimilation of implant materials and bone-tissues [18], on the surface of temporary implant materials [19,20]. Above studies hint that adding high content of Zr (e.g., 50 wt%) combined with cold working may enhance the mechanical compatibility of conventional Ti-6Al-4V ELI alloy, one of the most wide used metallic biomaterials, for use in removable orthopedic implants. In this study, a new near β Ti-50Zr-5Al-4V (wt%) alloy has been developed. The microstructure, Young's modulus and tensile properties of this alloy which is subjected to solution treatment and cold rolling have been investigated and an exceptional combination of ultralow Young′s modulus and ultrahigh strength and ultrahigh elastic admissible strain has been achieved.

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2. Materials and methods

2.1. Material preparation

The studied Ti-50Zr-5Al-4V (wt%) alloy (henceforth denoted as TZAV alloy) with a composition corresponding to a near β alloy was prepared by melting sponge Ti (99.7 wt%), sponge Zr (99.5 wt%), industrially pure Al (99.5 wt%) and industrially pure V (99.9 wt%) using a vacuum non-consumable electro-arc furnace. The ingot was then flipped and remelted three times to achieve compositional homogeneity. Subsequently, the ingot was held at 900 °C for 90 min and then hot rolled with a reduction of ~70%. The hot rolled ingot was β solution treated (ST) under vacuum at 850 °C (over β-transus temperature of $T_\beta \sim 735$ °C) for 1 h and then water quenched, and the as-quenched sample exhibited a coarse β microstructure with an average grain size of ~220 μm (data not shown here). Following solution treatment, cold rolling was conducted at room temperature (RT) with a cumulative reduction of ~35% at an average strain rate of ~1.5 s^{-1}.

2.2. Microstructural characterization

Microstructure and phase compositions in the rolling direction (RD)-transverse direction (RD) plane of TZAV samples subjected to solution treatment and cold rolling were characterized and determined using an optical microscope (OM), a transmission electron microscope (TEM) at an accelerating voltage of 200 kV and a current of 120 mA and an X-ray diffraction (XRD) with Cu Kα radiation at a diffraction angle (2θ) range of 30–80° and at an accelerating voltage of 40 kV and a current of 200 mA. Dislocation density of samples was calculated by means of quantitative X-ray-diffraction measurements using the Scherrer and Wilson equation [21,22]. For XRD analysis, the specimens were mechanically polished using SiC waterproof emery papers up to 2500 grit. For TEM, the specimens were mechanically polished with up to 2500 grit to a thickness of around 30 μm. Then, they were twin-jet electrochemically polished in a solution containing 10% perchloric acid and 90% methanol at 13 V and below −30 °C.

2.3. Young's modulus and mechanical property testing

Young's modulus and mechanical properties of TZAV samples were evaluated by tensile tests at RT using an Instron 5948 machine under a constant cross-head speed (i.e., 0.33 mm/min) with an initial strain rate of 1.1×10^{-3} s^{-1}. The strain was measured with a non-contacting video extensometer. The resolution of the video extensometer was 0.5 μm, and the frame rate of one data-point per 20 millisecond was used to capture data-points both in the elastic region and in the plastic region. The extensometer can be calibrated by placing a known distance piece adjacent to the specimen face or measuring the standard specimen with width/diameter of a micrometer. These targets points are then selected using the computer mouse and their known values entered, this results in a recalculation and display of the size of the monitor's field of view. Provided the camera is not moved or the lens adjusted between tests, subsequent specimens will be automatically measured without recalibration. Rectangular plate samples with a gauge cross section of 2.2 mm × 0.35 mm and a gauge length of 5 mm (see the inset in Fig. 1) were cut using wire electric discharge machining from the RD-TD plane. The tensile tests were guided by the standard ISO 6892, 1998. The samples were non-standard, however, the sizes complied with $L_0/\sqrt{S_0} = 5.65$, where L_0 and S_0 were the gauge length and cross-section area of tensile samples, respectively. For each condition, four specimens were used for tests, and the standard deviations of Young's modulus, tensile strength and elongation to failure were less than 5 GPa, 30 MPa and 1%, respectively. The tensile direction was parallel to the rolling direction of the samples.

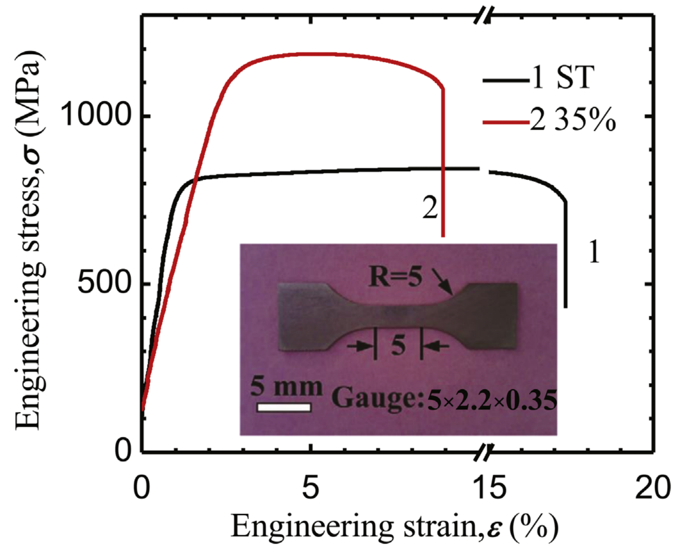

Fig. 1. Tensile engineering stress-strain curves of TZAV alloy subjected to solution treatment (ST) and cold rolling with a reduction of ~35%, and the inset is the tensile specimen geometry.

3. Results and discussion

3.1. Young's modulus and tensile properties

Fig. 1 shows the tensile engineering stress-strain curves of TZAV alloy subjected to solution treatment and cold rolling. A primary stress is imposed before the tensile testing in order to ensure the fixation and alignment of tensile specimens and avoid loosening between tensile specimens and fixtures during tensile testing. As a result, the stress-strain curves do not start from the origin. After solution treatment (TZAV-ST) (see curve 1), the Young's modulus (E), yield strength (σ_y), tensile strength (σ_b) and elongation to failure (ε_f) of TZAV alloy are ~70 GPa, ~680 MPa, ~850 MPa and ~17.3%, respectively. After cold rolling with a reduction of ~35% (TZAV-35%) (see curve 2), an extraordinary decrease in Young's modulus combined with an increase in strength is observed, and the Young's modulus, yield strength, tensile strength and elongation to failure are $E \sim 45$ GPa, $\sigma_y \sim 1000$ MPa, $\sigma_b \sim 1185$ MPa and $\varepsilon_f \sim 9.0\%$, respectively. In general, low elastic modulus and high mechanical strength are usually contradictory for most of Ti alloys, as shown by the gray region in Fig. 2. However, the TZAV-35% sample (indicated by symbol ♦ in Fig. 2) is fundamentally separated from the general trend (the gray region) and exhibits an exceptional combination of ultralow elastic modulus ($E \sim 45$ GPa) and ultrahigh tensile strength ($\sigma_b \sim 1185$ MPa).

3.2. Elastic admissible strain

The mechanically compatible performance of an orthopedic implant material can be evaluated by the elastic admissible strain [15], which is defined as the ratio of yield strength to Young's modulus of the material [23]. Fig. 3 presents the comparison of elastic admissible strain values of TZAV alloy and some commercial Ti alloys as well as recently developed β Ti alloys. As can be seen, the highest elastic admissible strain values for commercial Ti alloys and recently developed β Ti alloys are $\delta \sim 0.99\%$ (Ti-35.3Nb-5.1Ta-7.1Zr annealed) and $\delta \sim 1.31\%$ (Ti-34Nb-25Zr as-cast),