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Research Paper

Increasing strength of a biomedical Ti-Nb-Ta-Zr alloy by alloying with Fe, Si and O

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A B S T R A C T

Low-modulus biomedical beta titanium alloys often suffer from low strength which limits their use as load-bearing orthopaedic implants. In this study, twelve different Ti-Nb-Zr-Ta based alloys alloyed with Fe, Si and O additions were prepared by arc melting and hot forging. The lowest elastic modulus (65 GPa) was achieved in the benchmark TNTZ alloy consisting only of pure β phase with low stability due to the 'proximity' to the β to α'' martensitic transformation. Alloying by Fe and O significantly increased elastic modulus, which correlates with the electrons per atom ratio (e/a). Sufficient amount of Fe/O leads to increased yield stress, increased elongation to fracture and also to work hardening during deformation. A 20% increase in strength and a 20% decrease in the elastic modulus when compared to the common Ti-6Al-4V alloy was achieved in TNTZ-Fe-Si-O alloys, which proved to be suitable for biomedical use due to their favorable mechanical properties.

1. Introduction

Replacement of large joints is considered as a major achievement in the orthopaedic surgery. However, an appropriate implant material is also a big challenge for material scientists. Along with knee arthroplasty, the hip endoprosthesis is the most demanded joint implant. One of the most delicate issues in hip implant design is the femoral stem that is crucial to prevent the implant from loosening. In fact, the loosening of the implant is one of the most frequent causes of implant failure (Chu et al., 2002). 152,000 hip joint replacements were performed in the US in 2000, thereof almost 13% were revisions and reoperations of previous hip replacement (Long and Rack, 1998). The percentage of reoperations will rise due to the longer life expectation and more active life-style. Therefore, the demand for implants with enhanced life-time will be increasing.

Development of orthopaedic implants is a complex and multi-field scientific issue. Titanium alloys have been extensively applied in orthopaedics for several decades due to their superior mechanical properties, excellent corrosion resistance and favourable biocompatibility (Geetha et al., 2009, Katti, 2004, Long and Rack, 1998, Rack and Qazi, 2006). Elastic modulus of the implant material determining its stiffness is currently a widely discussed topic. Typical elastic modulus of Ti and common Ti alloys is around 100 GPa, while elastic modulus of the cortical bone ranges from 20 to 30 GPa and elastic modulus of cancellous bone is even lower (7–15 GPa) (Niinomi et al., 2012, Rho et al., 1993, Zysset et al., 1999). This difference in stiffness of implant and surrounding bone leads to the transmission of the applied load through the implant stem and consequently, the surrounding bone is not loaded (so-called stress-shielding effect). The bone tissue that is not regularly loaded becomes atrophied and is prone to failure. Therefore, materials with reduced elastic modulus are being developed.

The relationship between the elastic moduli (E) of different phases in Ti can be expressed as follows $E_\beta \approx E_{\alpha''} \approx$ 60–85 GPa < $E_\alpha \approx$ 100 GPa < $E_\omega \approx$ 130 GPa (Niinomi, 1998, Nejezchlebová et al., 2016, Sun et al., 2007, Tane et al., 2013), which demonstrates the interest in β-Ti alloys.

Metastable β-Ti alloys have been developed since 1960s (Lütjering and Williams, 2007). The dominant area of application is the aerospace industry. However, two decades ago, specialized biocompatible alloys also emerged. The most used β stabilizing alloying elements are vanadium, chromium, iron, molybdenum and niobium. Nb and Zr are regarded as biocompatible alloying elements, whereas V, Cr and Co are considered inappropriate (Steinemann, 1998).

The design of biomedical alloys for orthopaedic use therefore faces several limitations. Firstly, only biotolerant elements can be used. Secondly, sufficient strength level must be achieved. And thirdly, elastic modulus should be reduced well below 100 GPa. Note that the latter two requirements are often in a trade-off relationship.

The Ti-Nb-Ta-Zr alloying system is a highly biocompatible material

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with favourable mechanical properties. The benchmark alloy for this study, Ti-35.3Nb-5.7Ta-7.3Zr (TNTZ), was developed in 1990s in the USA and patented in 1999 (Ahmed and Rack, 1999). The particular alloy composition was selected empirically aiming to minimize the elastic modulus which can be as low as 60 GPa in the solution treated condition (Tang et al., 2000). Ti-Nb-Ta-Zr alloy is predetermined for biomedical use also due to low metal release in vitro, which is advantageous especially for long-term implants (Okazaki and Gotoh, 2015). On the other hand, a considerable disadvantage of this alloy is its low strength. Despite both Zr and Ta provide some solution strengthening when compared to Ti-Nb binary alloys (Ferrandini et al., 2007, Sakaguchi et al., 2005), the ultimate tensile strength reaches only 550 MPa. The major issue for applicability of this alloy for manufacturing of large joint implants is increasing its strength.

The main hardening mechanism in metastable β-Ti alloys is the formation of α phase precipitates. However, the presence of α phase precipitates leads to an unfavourable increase of elastic modulus. Precipitation hardening can be achieved also by titanium carbides and titanium borides (Chen and Hwang, 2012, Du et al., 2014, Zhang et al., 2012) and by titanium silicides. Si has a very low solubility in both the α and β phase and contributes to hardening via creation of dispersed precipitates of Ti_5Si_3. Moreover, in alloys containing Zr even more stable $(Ti,Zr)_5Si_3$ compound is formed (Ankem et al., 1987, Headley and Rack, 1979). Si content of 0.2–0.4 wt% is often utilized in high-strength and high-temperature alloys in aerospace industry to increase the strength and to suppress excessive creep (Chaudhuri and Perepezko, 1994, Welsch et al., 1993).

Solid solution strengthening is the fundamental hardening mechanism in alloys. Among bio-tolerable elements, Fe and Ta are known to cause significant solid solution hardening (Kudrman et al., 2007), whereas the effect of Mo and Nb is low. However, the experimental results are often affected by undergoing phase transitions and changes in deformation mechanisms (Min et al., 2008, Min et al., 2010).

Considering interstitial hardening, enhanced hardness due to oxygen increase from 0.3 wt% to 0.5 wt% was reported in Ti-Nb-Ta-Zr single crystals (Takesue et al., 2009) and 0.46 wt% O content increases the strength of Ti-35Nb-7Zr-5Ta-0.46 O alloy to 1000 MPa in solution treated condition (Qazi et al., 2004). Nakai et al. (2009) reported the increase of the elastic modulus and the strength for similar Ti-29Nb-13Ta-4.6Zr alloy by increased oxygen content. Niinomi et al. (2016) recently discussed the effect of oxygen on phase transformations in the same alloy.

Ti-Nb-Ta-Zr-O alloys with various Nb and O content and comparatively low Ta and Zr are often referred to as gum-metal due to very low elastic modulus and unusual dislocation-free plastic deformation mechanism (Furuta et al., 2007, Nagasako et al., 2016, Saito et al., 2003, Tane et al., 2011). Enhanced publication activity in the last years illustrates high research interest in the biomedical Ti alloys with increased oxygen content.

The combined effect of Fe and/or Si on strengthening of the TNTZ alloy was investigated in detail in our previous study (Kopová et al., 2016). However, to our best knowledge, no study examining combined effect of oxygen and Fe/Si on the strength and the elastic modulus of biomedical β-Ti alloy has been reported yet.

2. Material and experimental methods

The material was prepared at the company UJP Praha, Czech Republic by arc melting of pure elements under low pressure of clean He atmosphere (350 mbar). Oxygen was introduced during melting by adding appropriate amount of TiO_2, which dissolved in the melt. Each part of the sample was remelted at least six times by electric arc to ensure the chemical homogeneity. Samples of an approximate weight of 200 g in the shape of small bricks were homogenized at 1400 °C in vacuum for two hours and furnace cooled. This condition is referred to as the as-cast condition. Despite slow furnace cooling, the alloys did not contain any α phase particles observable by scanning electron microscopy.

Table 1
Chemical composition of investigated alloys. TNTZ refers to Ti-35.3Nb-5.7Ta-7.3Zr. Contents of alloying elements are given in wt%. Fe, Si and O are added at the extent of Ti. An asterisk (*) marks the alloys which could not be successfully forged.

Ti-35.3Nb-5.7Ta-7.3Zr (TNTZ)	TNTZ-2Fe
TNTZ-0.25Si	TNTZ-2Fe-0.25Si*
TNTZ-0.4O	TNTZ-2Fe-0.4O
TNTZ-0.25Si-0.4O	TNTZ-2Fe-0.25Si-0.4O*
TNTZ-0.7O	TNTZ-2Fe-0.7O*
TNTZ-0.25Si-0.7O	TNTZ-2Fe-0.25Si-0.7O*

The as-cast material was subsequently forged using forging hammer into the shape of rods by company Comtes FHT, Czech Republic. Prior to the forging and between the forging steps the material was heated to approximately 1100 °C in argon atmosphere to avoid excessive oxidation. The forging process was performed in air with cold tools and the temperature of the workpiece was not further controlled. Since no α phase was observed in the interior of the rods, the forging temperature of bulk material did not fall below β-transus temperature. The forged rods were machined to the diameter of 8–10 mm depending on surface damage. This condition is referred to as the as-forged condition.

The nominal composition of twelve alloys investigated in this study is summarized in Table 1. The oxygen content was checked by carrier-gas-hot-extraction (CGHE) method. The resulting oxygen content was 0.06, 0.35 and 0.66 wt% of O for alloys with the nominal oxygen content of 0, 0.4 and 0.7 wt%, respectively. The differences in the measured oxygen content between the alloys with the same nominal oxygen content were below 0.01 wt%. Nitrogen contamination was below 0.03 wt%. The forgeability of alloys with high content of Fe, Si and O was generally poor. Alloys marked by asterisk in Table 1 could not be successfully forged.

SEM observations were performed using scanning electron microscopes FEI Quanta 200 F and Tescan LYRA 3GMU both equipped with field emission gun (FEG) operated at the accelerating voltage of 20 kV. Microhardness was measured using automatic micro-hardness tester Qness Q10a according to Vickers with load of 0.5 kg and indentation time 10 s.

A computer-controlled DAKEL-CONTI-4 acoustic emission system was used to monitor acoustic emission (AE) signal during tensile tests. Four channels with different amplification (0–20-30–40 dB) and 2 MHz sampling frequency were used to detect and store data. More details on the method and the data analysis are reported elsewhere (Bohlen et al., 2004, Dobroň et al., 2012).

Tensile tests were performed at room temperature employing the computer controlled Instron 5882 machine using the strain rate of $10^{-4}\ s^{-1}$. Round samples with the diameter of 3 mm and the gauge length of 15 mm were used for the tensile tests.

Elastic constants were evaluated by the ultrasonic pulse-echo method (Papadakis and Lerch, 2000). Elastic constants are determined from velocities of propagation of quasi-longitudinal (qL) and quasi-transverse (qT) acoustic waves. Calculation of elastic coefficients from the set of velocities is relatively simple for materials with cubic crystal symmetry, which is the case of studied β-Ti alloys. Two sets of delayed broadband transducers for generating and receiving acoustic waves (10 MHz or 30 MHz for qL waves and 5 MHz or 20 MHz for qT-waves) were used with a pulse/receiver system DPR50+ (JSR Ultrasonics). Time of flight measurements were carried out by a pulse overlapping technique implemented in a digital storage oscilloscope LT264M (LeCroy) (Landa and Plešek, 2002).

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