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Microhardness and microstructure evolution of ultra-fine grained Ti-15Mo and TIMETAL LCB alloys prepared by high pressure torsion

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ABSTRACT

Two metastable β-Ti alloys, Ti-15Mo and Ti-6.8Mo-4.5Fe-1.5Al (TIMETAL LCB) were solution treated and subjected to severe plastic deformation by high pressure torsion. The evolution of microhardness, microstructure and elastic constants with increasing strain imposed by high pressure torsion was investigated.

Fragmentation of the microstructure with increasing strain was observed by scanning electron microscopy. Significant twinning in system {1 1 2} <111> after high pressure torsion was observed in both studied alloys by electron backscatter diffraction. Multiple twinning contributes significantly to the fragmentation of grains and consequently to the overall refinement of the microstructure.

Microhardness significantly increases with increasing strain and was fitted using the Hollomon and Voce laws. Hollomon's hardenability exponent is much higher for both studied β-Ti alloys than for the commonly used Ti-6Al-4V alloy. It reflects high capability of strengthening β-Ti alloys by intensive plastic deformation.

The measurement of elastic constants using resonant ultrasound spectroscopy showed that the deformation by high pressure torsion increases the Young's modulus as compared to solution treated material. On the other hand, further straining causes subsequent decrease of the Young's modulus.

1. Introduction

The importance of the β-titanium alloys in commercial practice has been increasing in the last few decades due to successful utilizing their unique properties such as high strength, low specific density, strengthening capability, high fracture toughness, and excellent corrosion resistance [1,2]. β-Ti alloys are extensively used in aircraft industry [3] and considered as prospective candidates for biomedical implants manufacturing due to their excellent biocompatibility and relatively low Young's modulus preventing the stress shielding [4–8]. However, a high strength condition is usually achieved by advanced thermomechanical treatment involving precipitation of α-phase particles, which significantly increases the Young's modulus [9,10].

Severe plastic deformation (SPD) methods strengthen metallic materials via reducing the grain size and increasing the dislocation density [11]. Furthermore, Young's modulus can be reduced by the microstructural refinement. It was also reported that the functional properties such as corrosion resistance and biocompatibility might be also improved by the microstructure refinement [12].

Ultra-fine grained (UFG) commercially pure titanium (CP Ti) was prepared by high pressure torsion (HPT) [13] and equal-channel angular pressing (ECAP) [14] almost two decades ago. Furthermore, UFG α+β Ti alloys such as Ti-6Al-4 V alloy and specialized biocompatible Ti-6Al-7Nb alloy were also studied in detail [15,16] and exhibited significantly improved strength and fatigue resistance [17,18].

On the other hand, there is only limited literature on the UFG metastable β-Ti alloys. Reports focused primarily on the study of the enhanced strength, fatigue performance [19,20] and microstructural refinement [21,22]. The elastic properties of UFG β-Ti alloy were studied only in alloys containing niobium as the main alloying element [23,24].

The mechanism of the grain refinement in the β-titanium alloys can vary widely, depending on the specific alloy composition, grain size, deformation mode, temperature and pressure [25]. The classical mechanism of the grain refinement is based on the movement of dislocations, formation of dislocation walls and sub-grain boundaries followed by lattice rotation forming high-angle grain boundaries [26]. However, the grain refinement can be also induced by twinning

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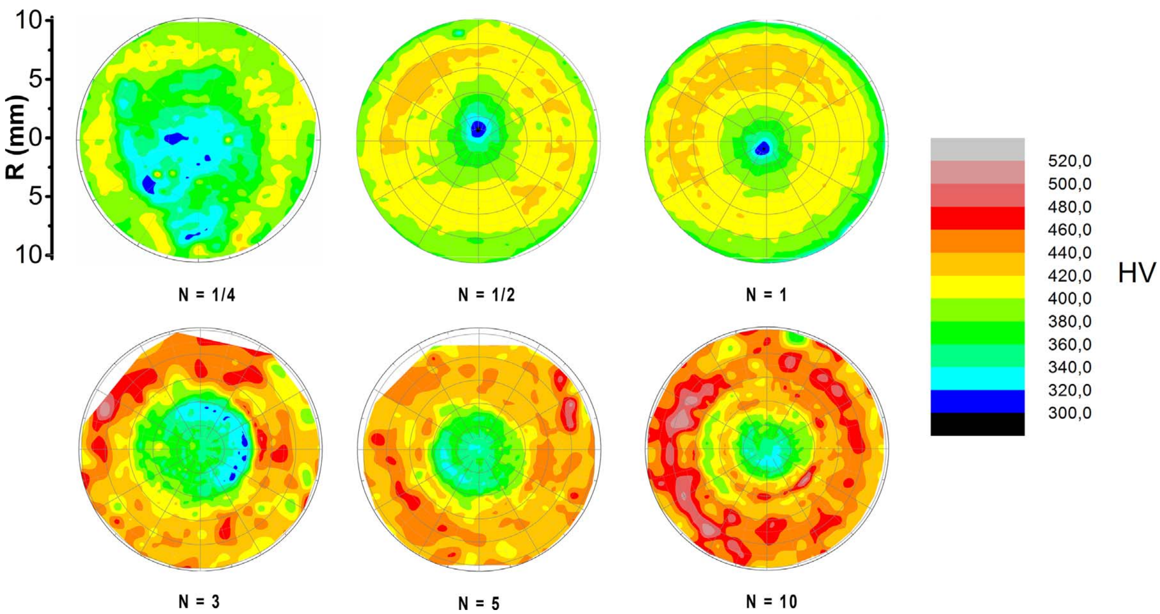

Fig. 1. Microhardness of Ti-15Mo alloy after various numbers of HPT turns represented by color-coded polar diagram. (For interpretation of the references to color in this figure, the reader is referred to the web version of this article.)

followed by lattice rotation. The two main mechanical twinning systems in bcc materials including β-Ti alloys are {1 1 2} ⟨1 1 1⟩ [27–30] and {3 3 2} ⟨1 1 3⟩ [31,32], the latter one being activated especially at low temperatures and at high strain rates.

Two metastable β-Ti alloys were used in this study: Ti-15Mo alloy and Ti-6.8Mo-4.5Fe-1.5Al alloy (TIMETAL LCB). Ti-15Mo alloy exhibits good mechanical properties, does not contain any toxic elements such as vanadium and therefore is suitable for a medical use. The TIMETAL LCB (low-cost beta) alloy is characterized by lower production costs due to the partial replacement of the relatively expensive β stabilizing alloying elements such as vanadium or molybdenum by iron. The TIMETAL LCB alloy was commercially used for suspension springs manufacturing, since the weight of a suspension spring can be reduced by 60% thanks to the high strength, low density and reduced Young's modulus of the alloy [9] when compared to previously used steels.

The objective of the present study is to examine the evolution of the microstructure, mechanical and elastic properties of two metastable β-Ti alloys with imposed equivalent strain.

2. Experimental

Ti-15Mo alloy was supplied by Carpenter Technology Corp. in a form of a rod with the diameter of 10.5 mm. Ti-6.8Mo-4.5Fe-1.5Al alloy was produced on demand by Huizhou Top Metals Ltd. using magnetic levitation melting and finally wire-cut to the diameter of 20 mm. The as-delivered material was solution treated (1083 K, 20 min) in a protective Ar atmosphere and water quenched. Ti-15Mo alloy was further cut to cylinders (diameter 10.5 mm, height approx. 5 mm) and pressed in HPT machine at room temperature to obtain the desired diameter of 20 mm. The principle of HPT method is described in detail in [33].

Samples with the diameter of 20 mm and the thickness of 1 mm were prepared by HPT at Ufa State Aviation Technical University (USATU) Ufa, Russia at room temperature and the pressure of 2 GPa. A series of samples after N=¼, ½, 1, 3, 5 and 10 turns and N =¼, ½, 1 and 5 turns of HPT was prepared from Ti-15Mo alloy and TIMETAL LCB alloy, respectively. The total equivalent strain imposed in the sample by HPT can be expressed by the von Mises approach, which utilizes a simple torsion, and the strain is then expressed by the linear relation [34]:

$$\varepsilon_{vonMises}=\frac{2\pi Nr}{\sqrt{3}\,h}, \qquad (1)$$

where N is the number of rotations, r represents the distance from the sample centre and h is the final thickness of the specimen. The equivalent strain imposed by pressing is about 1.5, while equivalent strains imposed by the torsion are by two decades higher. The thickness is therefore neglected.

Microhardness measurements were carried out using the automatic microhardness tester Qness Q10a by Vickers method; 1 kg load and dwell time of 10 s were applied. More than 1000 indents were automatically evaluated along concentric circles, which allows a detailed investigation of microhardness variations on both the surface and the cross-section of the disc.

The scanning electron microscope FEI Quanta 200 FX operated at 10 kV was used for microstructural observations and electron backscatter diffraction (EBSD) analysis.

Young's modulus and Poisson's ratio were evaluated by the resonant ultrasound spectroscopy (RUS) [35] using a fully contactless laser-based RUS set-up described in detail in [36]. This set-up utilizes focused laser pulses for generating the vibrations in the examined sample and the scanning laser beam for the interferometric detection of the modal response. Five different conditions after different stages of straining were used for RUS measurements for each alloy. All samples were rectangular parallelepipeds with the approximate dimensions of 2×2×1 mm^3. For the RUS measurements, the materials of all samples were considered as elastically isotropic, with only two independent elastic constants: Young’s modulus E and Poisson’s ratio ν.

3. Results

3.1. Microhardness

3.1.1. Ti-15Mo

The microhardness evolution with the increasing number of HPT turns on the specimen's surface is depicted as a series of color-coded images in the Fig. 1. The variations of the microhardness in the cross-section of the specimens are shown in the Fig. 2. The microhardness increases with the increasing distance r from the centre and with the increasing number N of HPT turns. In each image in the Fig. 1. two distinct regions are clearly visible – a central region with a low