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Acta Materialia 60 (2012) 2169–2177

Acta MATERIALIA

www.elsevier.com/locate/actamat

Microstructure and mechanical behavior of a Ti–24Nb–4Zr–8Sn alloy processed by warm swaging and warm rolling

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Received 1 September 2011; received in revised form 3 January 2012; accepted 3 January 2012
Available online 1 March 2012

Abstract

A combination processing technique of warm swaging and warm rolling is proposed to refine grains and improve the mechanical properties of a multifunctional β-type Ti–24Nb–4Zr–8Sn (wt.%) alloy. The results show that a highly swirled marble-like microstructure can be easily produced by warm swaging at an initial temperature of 573 K, whereas it has little effect on the nonlinear elastic deformation compared with the hot forged alloy with an equiaxed microstructure. Although the swirled microstructure has the limitation of an inhomogeneous distribution, swaging has the great advantage of refining the initial subgrains produced by hot forging with little loss of ductility. The following warm rolling at an initial temperature of 673 K results in a uniform microstructure comprising β phase with a size less than ~200 nm and the precipitation of nanosized α phase. Therefore, significant grain refinement was achieved through the formation and refinement of the subgrains. The ultrafine grained alloy exhibits large scale nonlinear deformation behavior with a recoverable strain of up to ~3.4% in combination with a high strength of ~1150 MPa, a low elastic modulus of ~56 GPa and good ductility of ~8%. Such an improvement in mechanical properties indicate great potential for biomedical applications.

Keywords: Titanium alloy; Grain refinement; Elastic behavior; Mechanical property; Martensitic transformation

1. Introduction

Titanium-based materials have become one of the most favored for biomedical applications due to their high strength, low elastic modulus, low density, good biocompatibility and corrosion resistance [1,2]. To alleviate the stress shielding effect caused by the stiffness mismatch between implants and the surrounding bone tissues [3] a series of metastable β-type titanium alloys have been developed with the advantages of a lower elastic modulus than Ti–6Al–4V alloys [4–6]. Significant grain refinement would be an effective way to optimize their mechanical properties, such as high strength, a low elastic modulus and good ductility.

It is well known that for metallic materials plastic deformation at large applied strains results in microstructure refinement. The mechanism governing grain refinement is generally accepted to be dominated by dislocation activities such as dislocation multiplication and interaction [7,8]. For materials with low stacking fault energies refinement can be significantly enhanced by a combination of mechanisms of deformation twining [9,10] and stress-induced phase transformation [11–14]. Since the evolution of the grain refinement process through the above mechanisms is sluggish conventional processing techniques such as rolling and swaging are incapable of refining the original coarse grains to the ultrafine grained (UFG) and nanostructured scales [12,15]. Several severe plastic deformation (SPD) techniques, such as equal channel angular pressing (ECAP) [16,17] and high pressure torsion (HPT) [18], have been developed.

The ECAP technique has the advantage of maintaining the cross-sectional area of the processed billets by repeated extrusion through a special die and has been widely used to

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doi:10.1016/j.actamat.2012.01.003

fabricate bulk UFG metallic materials [19–22]. Because titanium and its alloys are generally not sufficiently ductile at room temperature, high temperatures are needed for ECAP processing, for example 723 K for commercial pure titanium and 923 K for a Ti–6Al–4V alloy [23,24]. At high temperature recovery and recrystallization occur, compromising the efficiency of grain refinement [23,24]. On the other hand, titanium and its alloys are still hard to process by the ECAP technique at high temperatures, which in general limits the dimensions of the processed billets to lengths of less than 100 mm [19,25].

Ti2448 is a multifunctional β-type titanium alloy for biomedical applications with the chemical composition Ti–24Nb–4Zr–8Sn (wt.%) [26,27]. It exhibits large-scale nonlinear elastic deformation and highly localized plastic deformation, originating from the elastic and plastic instability of the bcc crystal [27,28]. The peculiar plastic deformation behavior leads to rapid grain refinement to tens of nanometers with high angle misorientation and even an amorphous transition during conventional cold processing [27]. Since the cold process decreases both ductility and reversible strain [29], a warm rolling technique at an initial temperature in the range 473–673 K has been developed to fabricate sheets of UFG Ti2448 alloy with a better balance between high strength, large recoverable strain and good ductility [30].

The previous studies have shown that the hot processed Ti2448 alloy contains lots of subgrains of less than 1 μm in size [26–31]. It is therefore expected that refinement of these subgrains would be helpful in achieving ultrafine grains. In this study a combination processing technique of warm swaging and warm rolling was developed to fabricate a UFG Ti2448 alloy. Evolution of the microstructure and its effect on mechanical properties after each process were investigated. It will show that the combined process has the merit of not only enhancing the biomechanical properties of the Ti2448 alloy and but also producing UFG rods with lengths and diameters large enough for biomedical applications.

2. Materials and experiments

An ingot with a diameter of 380 mm was made by vacuum arc melting using a Ti–Sn master alloy and pure Ti, Nb and Zr as raw materials and forged at 1123 K to form a billet 55 mm in diameter. The chemical composition of the ingot obtained by wet chemical and gas analysis is given in Table 1. It was swaged at an initial temperature of 573 K to form a rod with a diameter of 25 mm (warm swaging) and then rolled at an initial temperature of 673 K to a diameter of 11 mm (warm rolling). The above two-step warm process has a total reduction in area of ~95%.

Table 1
Chemical composition of Ti2448 alloy (wt.%).

Nb	Zr	Sn	O	Ti
24.3	3.85	8.20	0.16	Balance

Uniaxial tensile tests were conducted in air at room temperature (~295 K) at an initial strain rate of $1.3 \times 10^{-4}\ s^{-1}$ using specimens with a gage length of 15 mm and a diameter of 3 mm. Ductility was calculated from the change in gage length before and after tensile testing to failure. In order to ensure accuracy of the recoverable strain a strain extensometer was used to record the stress–strain curves. Young's moduli (E) were measured by the free resonant vibration method (JE-RT Young's modulus measurement apparatus) using cylinder specimens 6 mm in diameter and 50 mm in length. X-ray diffraction (XRD) and transmission electron microscopy (TEM) analyses were carried out to investigate the phase constitution and microstructure evolution during the two-step warm process along the transverse direction of the bars. XRD analysis was conducted using a Cu $K\alpha$ radiation source with an accelerating voltage of 40 kV and a current of 250 mA. TEM analysis was carried out in a JEOL 200 CX-II operated at a voltage of 200 kV. TEM specimens were prepared by twin jet electropolishing in a solution of 590 ml of CH_3OH, 350 ml of $CH_3(CH_2)_3OH$ and 60 ml of $HClO_4$ at a temperature of ~250 K. Specimens were also etched at the boiling temperature of an aqueous solution of 40 vol.% HCl for optical microscopy observation.

3. Results

3.1. Microstructure of the warm swaged alloy

The initial microstructures of the hot forged billet with a diameter of 55 mm are shown in Fig. 1. Optical observation showed that the hot forged alloy had an equiaxed microstructure with grain sizes of ~100 μm, whereas TEM analysis found that the equiaxed grains consist of subgrains with sizes of ~1 μm. Neither the α″ martensite nor the ω phase could be detected by selected area diffraction (SAD) analysis (see, for example, the inset in Fig. 1b showing a single β phase). The above results are in agreement with previous reports [26,31].

Fig. 2 shows the optical microstructures of the warm swaged bars with a diameter of 25 mm. Observation along the transverse direction found gradual changes in the microstructure: a swirled marble-like microstructure becomes more significant with increasing depth from the surface to the core (Fig. 2a–c). Detailed observation showed a fine and weakly swirled microstructure near the surface with a thickness of up to ~2.5 mm (Fig. 2a and b), which changed to a highly swirled microstructure near the core (Fig. 2c). The above analysis suggests that swaging with a reduction in area of ~80% cannot produce a uniform microstructure. Such a depth dependence of the optical microstructure is much weaker in longitudinal sections, for example Fig. 2d, showing a uniform fibrous structure. Consistent with the previously reported fibrous microstructure of bcc crystals, the quantitative XRD analysis