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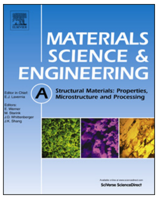

Fatigue behavior of ultrafine-grained Ti–24Nb–4Zr–8Sn multifunctional biomedical titanium alloy

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ABSTRACT

A multifunctional titanium alloy Ti–24Nb–4Zr–8Sn (wt%) was fabricated by warm swaging and rolling to get homogeneous microstructure with ultrafine-grained (UFG) β phase and nanostructured (NS) α phase, and their stress-controlled high cycle fatigue (HCF) and strain-controlled low cycle fatigue (LCF) behaviors were investigated in the study. The results showed that the UFG alloy exhibits much higher HCF strength than that of the hot-rolled alloy with coarse grains whereas its LCF endurance is worse slightly. This was explained by its stable microstructure originated from low homologous temperature being ~0.15 of melting temperature and the improvement of phase stability by grain refinement and NS precipitations. The former leads to dynamic recovery instead of dynamic recrystallization occurring in other UFG materials while the latter results in comparable cyclic stress stability with the hot-rolled alloy. The study also found that the shear bands have an identical angle of ~26° with the loading direction, which is independent of the strain ratios. Since the ductile UFG alloy has a nonlinear elastic deformation behavior with large recoverable strain, its LCF endurance is much better than those of other materials exhibiting linear elasticity and would be further improved by the depression of NS precipitation.

1. Introduction

Ultrafine-grained (UFG) metallic materials produced by severe plastic deformation (DPD) have attracted great attention due to the significant improvement of strength as compared with the corresponding coarse-grained (CG) materials [1–3]. Several typical SPD techniques such as equal channel angular pressing, high pressure torsion, accumulative rolling bonding and dynamic plastic deformation have been developed successfully to fabricate UFG metallic materials [1–7]. Extensive investigations have been conducted on their monotonic mechanical responses, deformation mechanisms and microstructure evolutions [8–14].

Since UFG materials have great potential for structural applications, their fatigue damage behaviors have been investigated systematically using UFG copper as a classical model metal [15–18]. The investigations have been performed also on other face-centered cubic (FCC) materials such as nickel [19] and aluminum and its alloys [20–22]. It is generally accepted that the grain refinement has considerable contribution on the stress-controlled high cycle fatigue (HCF) strength but negative effect on the strain-controlled low cycle fatigue (LCF) endurance [15–22]. Such influence can be explained by their higher strength and lower ductility on the basis of the total fatigue life diagram [23]. The deterioration of LCF endurance can also be explained by their microstructural instability, including dynamic recrystallization and its induced grain coarsening, and the formation of macroscopic shear bands which render cyclic softening and accelerate fatigue failure [23]. To alleviate the above mentioned negative effects, the influence of stress and microstructure on the cyclic behavior has been investigated in a wide range of metallic materials [24]. However, heretofore most of researches have been focused on UFG metallic materials with low melting temperature, corresponding to high homologous temperature when tested at room temperature. There is lack of knowledge on complex alloys with large amount of substitutional and interstitial elements as well as high melting temperature, in particular of the alloys with body-centered cubic (BCC) crystal structure.

Recently, a multifunctional titanium alloy, Ti–24Nb–4Zr–8Sn (wt%, hereafter abbreviated as Ti2448), has been developed for biomedical applications [25,26]. Its highly localized plastic deformation behavior enables the alloy to be easily refined to nanosize grains via conventional cold processing [25,27]. To overcome its low ductility, a combination technique of warm swaging and warm rolling has been developed to fabricate UFG Ti2448 alloy, which has advantages of better balanced biomechanical properties such as high strength, low elastic modulus, good ductility and large recoverable strain [28]. Considering its biomedical application, the fatigue properties are crucial in view of the requirements

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ensuring a safe performance during service, especially for hard tissue replacement to bear heavy loading. Furthermore, the UFG alloy satisfies the desirable features of metallic materials with good LCF endurance: they should have high content of solution elements and high melting temperature in order to realize lower homologous temperature [23].

In the study, both the stress-controlled high cycle fatigue (HCF) and the strain-controlled low cycle fatigue (LCF) behaviors of UFG Ti2448 alloy will be investigated and compared with the previous results of the hot-rolled alloy with coarse grains. On the basis of these experimental results, its fatigue behavior and damage mechanism will be discussed. The present study is helpful not only to realize the potential biomedical applications of the UFG Ti2448 alloy, but also to deepen the understanding on fatigue behavior of UFG materials and provide strategy to improve their fatigue properties.

2. Experimental

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. It was forged at 1123 K to a billet 55 mm in diameter, cold-swaged at an initial temperature of 573 K to a rod with a diameter of 25 mm (the warm swaging) and then cold-rolled at an initial temperature of 673 K to a diameter of 11 mm (the warm rolling). Uniaxial tensile tests were conducted in air at room temperature at an initial strain rate of $1.3 \times 10^{-4}\ s^{-1}$ using plate specimens with a gage length of 13 mm and a rectangular cross section of $3.0 \times 2.0\ mm^2$. In order to ensure accuracy of recoverable strain, a strain extensometer was used to record the stress–strain curves. Fatigue tests were conducted on an INSTRON 8862 testing machine at sinusoidal waveform cyclic stress in air at room temperature using rod specimens with a gage length of 20 mm and a gage diameter of 4.5 mm. The specimens were ground with SiC papers from 57 to 10 μm and then polished to get mirror surface. The stress-controlled HCF tests were carried out with stress ratio of $R=0.1$, and a frequency of 10 Hz. The total strain-controlled LCF tests were conducted at two different strain ratios. The tension–tension mode LCF tests with strain ratio $R=0.1$ were performed at a frequency of 1 HZ with the maximum strains ranging from 2% to 4.5%. The symmetric tension–compression LCF tests ($R=-1$) were carried out at a frequency of 0.5 HZ with the strain amplitudes up to 4%.

Both X-ray diffraction (XRD) and transmission electron microscopy (TEM) analyses were employed to characterize specimens prior to and after fatigue tests. X-ray data was gained from a diffraction meter with a CuKα radiation source using an accelerating voltage of 40 kV and a current of 250 mA. TEM observation was carried out on a JEOL 2010 CX-II machine using specimens cut from the cross section of the tested specimens. Thin foils were prepared by twin-jet electropolishing in a solution of 590 ml CH_3OH, 350 ml $CH_3(CH_2)_2CH_2OCH_2OH$ and 60 ml $HClO_4$ at −25 °C. Fracture surfaces after the fatigue test were analyzed by a LEO SUPRA35 scanning electron microscope (SEM).

3. Results

3.1. Microstructure

TEM microstructure of Ti2448 alloy after the warm swaging and rolling is shown in Fig. 1. It is clear from a pair of bright and dark field images that the average grain size of the β matrix is less than 200 nm. This was further supported by the inset selected area electron diffraction (SAD) pattern in Fig. 1a, showing the continual diffraction rings of the β phase and a little amount of nanostructured α phase precipitations. These results are consistent with the previous investigation [28]. Higher magnification observation found that the grains contain lots of tangled dislocations.

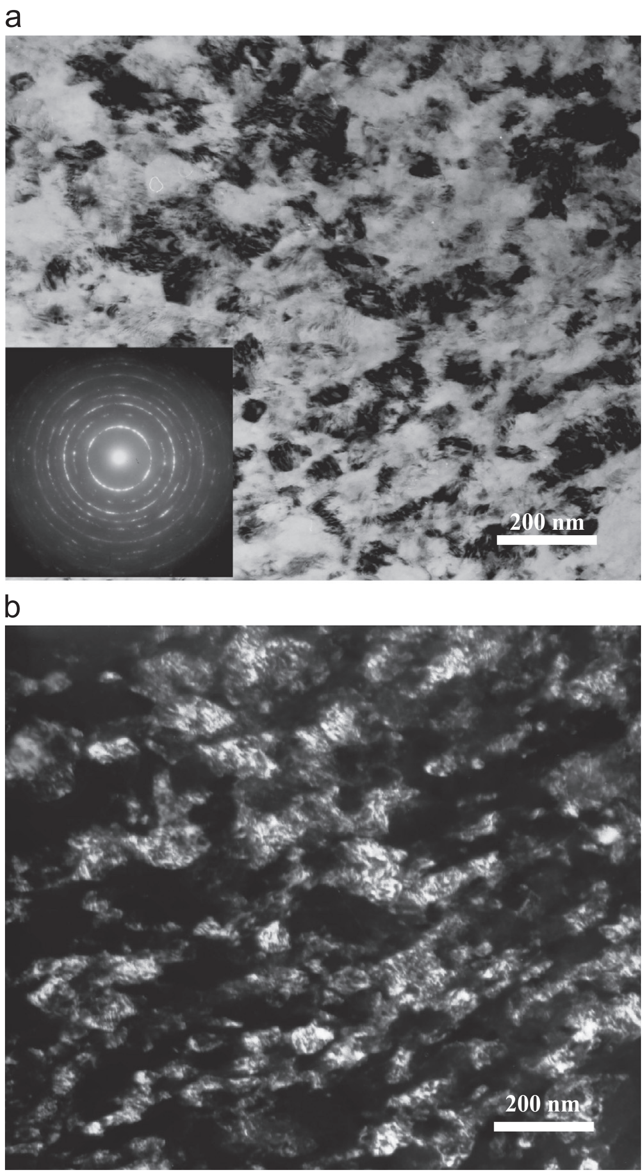

Fig. 1. A pair of bright field (a) and dark field (b) TEM microstructures of UFG Ti2448, in which the inset in (a) is the corresponding selected area electron diffraction (SAD) pattern.

The hot-rolled Ti2448 has equiaxed single β phase microstructure with grain sizes of about several micrometers and subgrains with sizes of about 600 nm [29]. Since these subgrains have slight difference in orientations less than 2° in general, the hot-rolled Ti2448 was named as the coarse-grained (CG) alloy in the study. For comparison, the warm-swaged and warm-rolled Ti2448 alloy with ultrafine-grained (UFG) microstructure and nanostructured (NS) precipitations was named as UFG alloy.

3.2. Cyclic tension and compression behaviors

The stress–strain curves of the UFG alloy subjected to cyclic tension with the total strain of 5% and an interval of 1% (Fig. 2a) showed that it exhibits nonlinear elasticity, and the loading and unloading curves at strains of 1%, 2% and 3% are overlapped, fully