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Fatigue and corrosion-fatigue strength of hot rolled $Ti_{35}Nb_{2.5}Sn$ alloy

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ABSTRACT

The $Ti_{35}Nb_{2.5}Sn$ alloy was obtained by vacuum arc melting. The alloy was homogenized and solubilized. It was then hot rolled with 40% reduction and quenched in water. Samples were also solubilized and quenched in water after hot rolling to check the procedure's effect on the formation of metastable phases. The microstructures were analyzed by microscopy and X-ray diffraction. Tensile tests were performed. Fatigue tests were also done to obtain the S–N curves in air and in environment containing 0.9% NaCl dissolved in water. The fracture micromechanics were analyzed in scanning electron microscope. The results were compared with published data from other beta titanium alloys. The fatigue limit is associated with critical stress by dislocations. The fatigue limit is higher than the stress required to drive the formation of the martensitic α″ phase. The study showed that the corrosion effect is higher in low cycle fatigue.

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1. Introduction

Biometals have considerable importance in the reconstruction of structural tissues. Materials for biomedical applications should have low cytotoxicity, high resistance to corrosion, wear and to fatigue. Materials must have also physical and mechanical compatibility with the tissue to be replaced [1–3].

Titanium and its alloys have been extensively used in biomedical applications, mainly due to their greater biocompatibility and lower elastic modulus compared to other metal alloys [4]. The Ti_6Al_4V alloy is one of the highest commercial productions among several other titanium alloys [5]. However, the Ti_6Al_4V alloy exhibits much higher elastic modulus than that of the human cortical bone. This alloy also presents cytotoxicity issues assigned to aluminum and vanadium [5,6].

β type titanium alloys are alternatives to overcome Ti_6Al_4V limitations. The β titanium alloys can exhibit lower elastic modulus and may be produced by elements that do not exhibit cytotoxicity, such as niobium, molybdenum and zirconium [7,8]. Niobium has been substitutionally dissolved in levels of approximately 40% in the titanium structure to retain the β phase [9–12]. Tin has been added in small amounts to TiNb alloys to suppress their martensitic transformations [13,14]. The thermomechanical processing control is crucial to the obtainment of stabilized phases in β Ti alloys as well as the alloy's final mechanical properties [15–17].

The current study investigated hot rolled and water quenched $Ti_{35}Nb_{2.5}Sn$ alloys. It aims to investigate hot rolling effects on phase transformations and to analyze the alloy's fatigue and corrosion-fatigue properties.

2. Materials and methods

2.1. Arc melting

The following raw materials were used to manufacture the alloy: 99.99% Ti, 99.50% Nb and 99.98% Sn. The titanium acid cleaning was conducted using a solution composed of water, nitric acid (NHO_3) and hydrofluoric acid (HF) in a 1:1:1 ratio. As for the Nb acid cleaning, a solution composed of water, sulfuric acid (H_2SO_4), nitric acid (NHO_3), and hydrofluoric acid (HF) was applied in a 1:1:1:1 ratio. Sn was obtained in the form of granules and it was not acid cleaned. The proper $Ti_{35}Nb_{2.5}Sn$ alloy composition was classified by analytical balance with 0.001 g resolution. Melting was carried out on arc furnace to obtain 140 g ingots. Three ingots were produced to enable microstructural analysis and the preparation of all specimens for mechanical tests. The ingots were re-melted six times to provide suitable chemical homogenization.

2.2. Thermomechanical process

Details of ingot heat treatments and the hot rolling procedure can be found in Griza et al. [18]. The parameters used in the processes are as follows: the ingots were subjected to homogenization at 1000 °C for 12 h and cooled in furnace. Subsequently, they were solubilized at 900° for 15 min and water quenched at 25 °C. The ingots were then hot rolled at 800 °C, followed by water quenching, in order to obtain plates with 40% strain and final thickness of 8 mm. The hot rolled plates were subjected to analysis and mechanical tests. Samples from the hot

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rolled material were solubilized again. This heat treatment was applied to verify microstructural changes in the alloy during hot rolling.

2.3. Alloy characterization

Five metallographic samples were obtained in the plane of thickness and rolling direction of the plates. The samples were prepared for microstructural analysis. They were embedded in resin and sanded using 240 up to 1200-grit water sandpaper. The polishing was performed using diamond paste of 6 μm, 3 μm and alumina suspension of 1 μm. The Kroll's etching (65 ml distilled water, 5 ml HF, 30 ml NHO_3) was used to reveal the microstructure of the samples. The rolled and thereafter solubilized samples were also prepared for analysis in the same way.

All samples were analyzed by X-ray diffraction (Shimadzu XRD 6000) according to the following specifications: 30 mA, 40 kV, scan 1.2°/min, 2θ ranging from 30 to 90°. The samples were also analyzed by optical microscopy (Leica DM 2500 M) and scanning electron microscopy (SEM-Jeol JCM-5700 Carry Scope).

2.4. Mechanical tests

Three specimens were machined for the tensile tests, whereas 17 specimens were machined for the fatigue and corrosion-fatigue cyclic tests (Fig. 1). The axis of all the specimens matches the rolling direction of the plates. Tensile tests were performed using the universal testing machine (Instron 3367) with 30 kN load cell, according to the ASTM E8/E8M standard. The tensile samples were sanded up to 600 mesh.

The cyclic test specimens were polished using a rotary mandrel and polishing cloth tapes with diamond paste. The specimens were polished up to 0.15 μm roughness and measured by profilometer (Mitutoyo SJ-410). Cyclic tests were performed according to the ASTM E466 standard, using a servohydraulic testing machine (MTS Landmark 3070.10). The tests were conducted with load ratio R = 0.1 and frequency of 30 Hz at room temperature. S–N curves were achieved in both non-aggressive environment (air) and in aggressive medium (0.9% NaCl aqueous solution). As for the last case, we used the lower frequency of 1 Hz [19]. The low frequency was applied in corrosion-fatigue tests since time is necessary for the corrosive medium to work on the front side of the propagating crack.

An acrylic chamber was used in the corrosion-fatigue tests to allow the suitable attachment of the specimens immersed in the solution and the controlled aeration of the solution (Fig. 2). The solution was kept at room temperature. The solution's pH was controlled by litmus paper and kept between 6 and 7.

3. Results

3.1. Microstructural analysis

Fig. 3 shows the optical microscopy of the alloy after homogenization, solubilization and quenching. It also shows the alloy's X-ray diffractogram. The solubilization product is the matrix composed of β-phase coarse equiaxed grains 1 ASTM (ASTM E112-13). Beta phase formation was expected after the solubilization due to niobium and tin contents, which are recognized as beta-stabilizing elements [9,17].

Fig. 4 shows the optical microscopy and X-ray diffraction results of a hot rolled and water quenched sample. The microstructure consists of β-phase coarse grains. Grain boundaries (GBs) showed irregular lines. Needles of α″ phase and slip bands were also observed on the β matrix. The X-ray diffraction matched the microscopic analysis. The α″ phase was verified by means of two low intensity peaks (between 40° and 45°) in the diffractogram. The martensitic α″ phase was produced during the thermomechanical processing.

The literature reports that the α″ phase can be formed by mechanical stress at room temperature [20], or even by transformation induced by the coordinated movement of atoms, thus causing microscopic and homogeneous shear during rapid cooling [21].

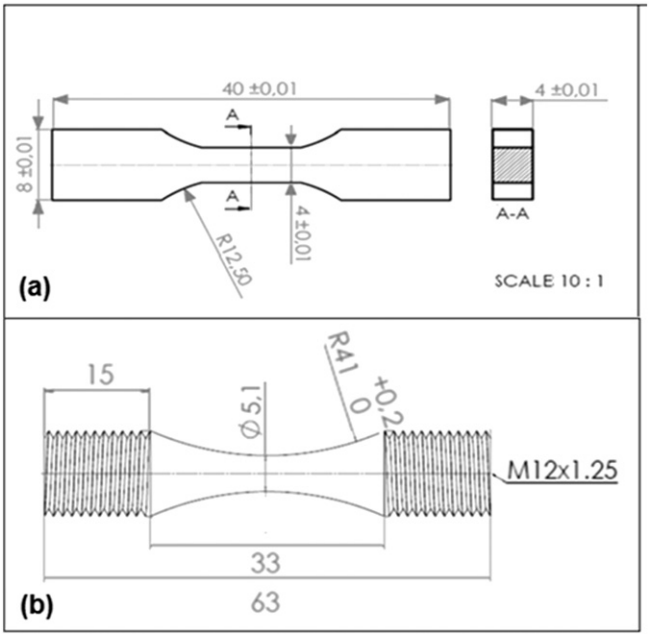

Fig. 1. Dimensions (mm) of the specimens for tensile test (a) and cyclic tests (b).

Fig. 2. Chamber assembled for the corrosion-fatigue test.

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