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Fatigue performance evaluation of a Nickel-free titanium-based alloy for biomedical application - Effect of thermomechanical treatments

G. Mussot-Hoinard[a,*], W. Elmay[a], L. Peltier[b], P. Laheurte[a]

[a] *Laboratoire d'Etude des Microstructures et de Mécanique des Matériaux (LEM3 UMR CNRS 7239), Université de Lorraine, île du Saulcy, 57045 Metz Cedex, France*
[b] *ENSAM, 4 Rue Augustin Fresnel, 57070 Metz, France*

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ABSTRACT

In the present work, structural fatigue experiments were performed on a Ti-26Nb alloy subjected to different thermomechanical treatments: a severe cold rolling, a solution treatment and two aging treatments at low-temperature conducted after cold rolling in order to optimize the kinetics of precipitation. The aim is to investigate the effect of microstructural refinement obtained by these processes on fatigue performances.

Preliminary tensile tests were performed on each state and analyzed in terms of the microstructure documented by using X-Ray diffraction and TEM analysis. These tests clearly promote the short-time-aged cold-rolled state with a fine α and ω phases precipitation. An interesting balance between mechanical properties such as high strength and low Young's modulus has been obtained.

Cyclic bending tests were carried out in air at 0.5%, 1%, 2% and 3% imposed strain amplitudes. At low straining amplitude, where the fatigue performances are at their best, the cold-rolled state does not break at 3×10^6 cycles and the long-time aged precipitation hardened state seems to be a good competitor compared to the cold-rolled state.

All failure characteristics are documented by Scanning Electron Microscopy (SEM) micrographs and analyzed in term of microstructure.

1. Introduction

Due to their attractive mechanical properties, corrosion resistance and excellent cold workability, biocompatible (Williams, 2008) metastable β-titanium alloys have attracted much attention for medical applications. During the last decade, Nickel-free low modulus β-titanium alloys were developed and characterized. Efforts were made through many studies to improve their properties in order to fulfill the application requirements (Hanada et al., 2005; Kim et al., 2006; Miyazaki et al., 2006; Geetha et al., 2009; Guo et al., 2010; Li et al., 2011; Niinomi et al., 2012). In particular, the refinement of the microstructure through thermomechanical treatments was given as an efficient way to improve the mechanical properties (Kim et al., 2006; Elmay et al., 2013; Elmay et al., 2014). Thus, low-temperature associated to short-time aging treatment performed on severe cold deformed material contributes to an interesting combination of high strength, large recoverable strain and low elastic modulus.

Furthermore, most devices used for biomedical applications are solicited under fatigue conditions. Therefore, the characterization of the fatigue behavior is nowadays a major investigation for biomaterials titanium alloys in order to evaluate their durability and their reliability. Many authors have contributed to a better understanding of fatigue behavior of titanium alloys. Some under rotating-bending mode (Miyazaki et al., 1999; Wagner et al., 2004; Young and Van Vliet, 2005; Bahia et al., 2006; Figueiredo et al., 2009; Pelton et al. 2013), others under three-point bending mode (Xue et al., 2007; Bourauel et al., 2008). Some authors have also worked on the fatigue performance of non-toxic Nickel-free Titanium alloys by performing cycling under tension mode (Akahori et al., 2005; Boehlert et al., 2005; Niinomi, 2003; Niinomi et al., 2007; Niinomi, 2007; Boehlert et al., 2008; Frotscher et al., 2009; Tahara et al., 2009; Zhang et al., 2009; Song et al., 2015; Sheremetyev et al., 2016). An evaluation of fatigue performance of aged materials beforehand solution treated has been the subject of the majority of these different studies. Few of these authors have considered the effect of aging treatments after cold-rolling (Akahori et al., 2005; Niinomi, 2007). However, the effect of aging time and more particularly of a short-treatment has not been taken into account. For instance, in these studies, long times aging are investigated (28.8 ks for Boehlert et al., 2005 and 259.2 ks for Akahori et al., 2005). The operating conditions and materials being different, a result

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* Corresponding author.
E-mail address: genevieve.mussot@univ-lorraine.fr (G. Mussot-Hoinard).

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comparison is not conceivable.

In the present study, the bending mode was chosen to characterize the fatigue life of a Ni-free biocompatible β-titanium alloy. This mode is appropriate for the fatigue of bone-plates and fixation devices commonly used for complicated fractures (Niinomi and Nakai, 2011). The selected Ni-free biocompatible β-titanium alloy was the Ti-26Nb alloy (at%). Bending straining was carried out on four metallurgical states: a severe hardening by cold rolling (CR), a solution treatment (ST) followed by water quenching (WQ) and two aging treatments conducted after severe hardening, at a low temperature of 573 K for two different aging times, a short one at 0.6 ks and a twelve time longer one at 7.2 ks. The low aging temperature associated to the short-time treatment has a nanostructuring effect of the ω and/or α phases precipitation occurring during aging of β-titanium alloys. These precipitates have an influence on the stability of the β phase; Limiting their coarsening leads to the obtaining of nanoscaled phases with a fine dispersion and a low volume fraction allowing the limitation of detrimental effects such as a loss of ductility and an increase in elastic modulus (Ohmori et al., 1998; Sun et al. 2011; Elmay et al., 2014).

Finally, this paper has to be considered as a contribution to a better understanding of the effect of microstructural refinement and nanoscaled precipitates obtained by thermomechanical processing on the fatigue life and the fracture characteristics of Ni-free β- titanium alloys.

2. Experimental procedure

2.1. Material

A Ti-26Nb (at%) ingot was obtained using the cold crucible levitation melting technique (CCLM) under high purity Ar atmosphere using ultra-pure titanium and niobium raw materials (Morita et al., 2000). This ingot was then subjected to a thermal homogenization treatment at 1223 K during 72 ks under high purity Ar atmosphere followed by water quenching. A severe cold rolling (CR) process was performed on the Ti-26Nb homogenized ingot. The achieved cold rolling thickness reduction rate was about 95% nominal strain. The material was flat bars with a rectangular cross section of width and height (*b* and *h*) respectively equal to 1.88 mm and 0.8 mm ± 0.04 mm. The longitudinal direction of the bars was parallel to the rolling direction.

Four states were investigated for the present study: a cold rolled state issued from the severe cold rolling process previously described, a solution treated state and two cold rolled aging states. The solution treated state was obtained by a solution treatment (ST) performed above the β transus at 1173 K for 3.6 ks on the cold rolled sample and then water quenched. Concerning aging treatments, most authors make the choice to apply them after a ST state (Akahori et al., 2005; Boehlert et al., 2005; Niinomi, 2007; Niinomi et al., 2007; Boehlert et al., 2008; Frotscher et al., 2009; Tahara et al., 2009; Zhang et al., 2009; Song et al., 2015). In the present study, the aging treatments were applied after the CR process in order to get maximum benefit from the hyperdeformed structure in which nucleation sites promote the kinetic precipitation of new phases (Furuhara et al., 2001) leading to improve hardening. Aging treatments were conducted under ultra pure Ar atmosphere at a low temperature of 573 K for either a short time period of 0.6 ks or a twelve time longer period of 7.2 ks. These treatments were followed by water quenching (WQ). These four states were respectively called CR, CR/ST/WQ, CR/573K-0.6ks/WQ and CR/573K-7.2ks/WQ.

Surface roughness of samples was measured by a Leica confocal and interferometry 3D microscope. For each of the four states, the mean transverse value, determined from three different regions on samples, is less than 0.5 µm. The samples surface was therefore left without polishing. An X-Ray diffractometer equipped with curved position sensitive detector with Cu-Ka radiation was used to identify the different phases present in the samples after the different thermomechanical treatments. Thin foils were extracted from flat bars using a FIB Zeiss Auriga 40 equipped with both electronic and ionic columns (Gemini and Orsay Physics Cobra, repectively). An ionic Ga beam of 30 kV was used. Thin foils were finished under a beam of 5 kV before extraction in order to eliminate amorphous layers induced by the high energy abrasion. Microstructure observations were done using a Philips transmission electron microscopy (TEM) operating at 200 kV.

The failure characteristics were analyzed using a Scanning Electron Microscopy (SEM) operating at an acceleration voltage of 30 kV.

2.2. Experimental testing

Bending fatigue tests complemented by preliminary common tensile tests were undertaken on the four CR, CR/ST/WQ, CR/573K-0.6ks/WQ and CR/573K-7.2ks/WQ states in order to identify the best candidate to medical applications.

2.2.1. Preliminary tensile testing

Room temperature uniaxial tensile tests up to 3% strain were performed at a strain rate of $2{\times}10^{-3}\ \mathrm{s}^{-1}$ using a Zwick testing machine. Tensile direction was parallel to the rolling direction and strain was measured with an extensometer.

2.2.2. Bending fatigue testing

Bending testing is appropriate for the study of the fatigue life of bone-plates and fixation devices commonly used for complicated fractures.

Fatigue tests were conducted at room temperature using a bending set-up and an automated servo-hydraulic MTS mechanical testing machine equipped with a 0.25 kN force transducer adapted to the specimens answer. The cyclic mechanical strain were applied to the specimen by cyclical compressive displacements using a sinusoidal profile at a frequency of 5 Hz. Fatigue tests were undertaken at 3%, 2%, 1% and 0.5% maximum strain amplitudes on sample surfaces by using respectively, according to relation (1), loading cylinders of 13.3 mm, 20 mm, 40 mm, and 80 mm radii of curvature R. The values of the compressive displacements necessary to obtain the required strain amplitude have been experimentally verified for each cylinder using a strain gauge placed on the specimen surface.

$$\varepsilon = h/2R \tag{1}$$

(Eggeler et al.,2004; Wagner et al.,2004; Pelton et al., 2013)

The maximum stress $\sigma_{xx\max}$ (relation 2) due to the bending moment M in the central section of the sample never exceed 250 MPa. I_z represents the second moment of area with respect to the vertical axis.

$$|\sigma_{xx}|_{\max} = h\ M/2\ I_z \tag{2}$$

The fatigue limit, at which the specimen did not break, was set at $3{\times}10^{6}$ cycles in this study. A standard hip-joint should be able to support about $2{\times}10^{6}$ cycles per year (Windler and Klabunde, 2001). For each state, all fatigue tests were performed twice or more in case of scattering of the results.

Some fatigue tests were instrumented with a FLIR infrared thermal camera in order to verify the adiabatic condition during the fatigue tests. No representative variation in temperature (below 1 K) has been registered.

All tests were instrumented with a Microsoft HD camera taking views at 1 min shooting interval to detect precisely the failure moment of samples. Therefore, this approach allowing real-time monitoring of the test can be considered as an in-situ method compared to other methods commonly used such as low current detection.