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Research Paper

Effects of thermomechanical process on the microstructure and mechanical properties of a fully martensitic titanium-based biomedical alloy

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ABSTRACT

Thermomechanical treatments have been proved to be an efficient way to improve superelastic properties of metastable β type titanium alloys through several studies. In this paper, this treatment routes, already performed on superelastic alloys, are applied to the Ti–24Nb alloy (at%) consisting of a pure martensite α″ microstructure. By short-time annealing treatments performed on the heavily deformed material, an interesting combination of a large recoverable strain of about 2.5%, a low elastic modulus (35 GPa) and a high strength (900 MPa) was achieved. These properties are shown to be due to a complex microstructure consisting of the precipitation of nanoscale (α+ω) phases in ultra-fine β grains. This microstructure allows a superelastic behavior through stress-induced α″ martensitic transformation. In this study, the microstructures were characterized by X-ray diffraction and transmission electron microscopy and the evolution of the elastic modulus and the strain recovery as a function of the applied strain was investigated through loading–unloading tensile tests.

1. Introduction

During the last decade, Ti-based alloys have attracted much attention for medical applications as bio-implant materials due to their good mechanical properties, superior corrosion resistance and excellent cold workability (Long and Rack, 1998; Niinomi, 2008; Wang, 1996). The advantage of these alloys, especially the metastable β type alloys, is that they

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present a large span of properties which can be modulated and improved by controlling the thermomechanical treatment conditions (Laheurte et al., 2005; Prima et al., 2000). Researches on these β Ti-based alloys become rapidly intensified, especially with an aim to enhance their mechanical properties and superelastic performances. Several studies have been conducted on the characterization of the reversible stress-induced martensitic α'' transformation from the β microstructure in order to establish its influence on the mechanical properties and the superelastic performance (Elmay et al., 2010; Grosdidier and Philippe, 2000; Kim et al., 2005; Laheurte et al., 2010). Efforts have been made to improve mechanical functionality through the optimization of the microstructure such as controlling the constituent phases, size and morphology.

Refinement of structure by producing nanocrystalline materials is proved to be an effective way that provides an excellent combination between increased strength and good ductility (Koch, 2007; Valiev et al., 2003). Several methods have been developed and applied on Ti-based alloys such as the accumulative roll bonding technique (Kent et al., 2011), hot rolling (Geetha et al., 2004; Hao et al., 2007; Li et al., 2008) and equal channel angular extrusion (ECAE) (Arockiakumar and Park, 2010). It is well known that beta titanium alloys can be strengthened by the precipitation of nanoscale ω and/or α phase after aging treatments (Ivasishin et al., 2008; Sun et al., 2010a). Their presence results in a detrimental increase of elastic modulus. However, a low elastic modulus allows a reduction of the stress mismatch between the implant materials and human bone, a phenomenon responsible for the detrimental "stress shielding" effects (Niinomi, 2008; Sumitomo et al., 2008). Therefore, the thermomechanical route to reach a combination of low modulus and high strength simultaneously is a critical outcome.

Recently, Sun et al. have presented a thermomechanical strategy based on severe cold deformation followed by short-time heat treatment (Sun et al., 2010b, 2011a). This strategy is shown to be effective to optimize the balance between the high strength and the superelasticity through the combination of the microstructure refinement and the small volume fraction of α and ω precipitates.

However, such thermomechanical route has not yet been performed on an orthorhombic α'' titanium-based alloys. It will be interesting to expand the efficiency of such treatments on titanium alloys displaying a value of M_s temperature above RT (room temperature). In this study, we have investigated the effect of the combination of a severe plastic deformation by cold rolling and a short-time heat treatment on the Ti–24Nb alloy which contains only α'' orthorhombic phase at room temperature. The elastic properties and the engendered microstructure resulting from this thermomechanical route were investigated by cyclic tensile tests, XRD and transmission electron microscopy (TEM), respectively.

2. Experimental procedure

The Ti–24Nb (at%) ingot was melted by the cold crucible levitation melting method using pure metals. The ingot obtained was then subjected to the homogenizing treatment at 1223 K for 72 ks under high vacuum followed by water quenching. After that, the materials were cold rolled (CW) into thin sheets with 95% reduction of the original thickness. Tensile specimens were machined into rectangular 70 mm × 3 mm × 0.7 mm shape. Some specimens were subsequently encapsulated in quartz tubes under partial pressure of high-purity Ar and solution treated (ST) at 1173 K for 3.6 ks to undergo recrystallization and followed by water quenching. The chemical composition is given in Table 1. The titanium and niobium contents were measured by EDS analysis (Energy Dispersive X-Ray Spectrometer). The carbon and the oxygen were analyzed by the fusion method and infrared detection, the nitrogen and the hydrogen by fusion and conduction methods. The cold rolled (CW) specimens were subjected to a short-time annealing treatments at 873 K and 573 K (CWA_{873} and CWA_{573}) for 0.6 ks followed by water quenching. An X-Ray diffractometer equipped with curved position sensitive detector with Cu-Kα radiation was used to identify the different phases present in the samples at different treatment states. To minimize the texture effect on the intensity of the diffraction peaks, a two axis summation technique was used. It consists in rotating the specimen around the normal axis (ϕ) from 0° to 360° for different positions of declination angle (Ψ) between 0° and Ψ_{max} of 70°. Specimens for microstructural observation were mechanically polished and then etched in a Kroll solution consisting of 4% HF, 6% HNO_3 and 90% H_2O. Optical microstructure observation was performed using an Olympus BX61optical microscope equipped with a high-resolution digital camera. Cyclic loading–unloading tensile tests were performed at room temperature at a strain rate of 2×10^{-3} s^{-1}. Strain was measured using an extensometer with a 20 mm gage length. Thermal cycling tests under various constant stresses were performed using a tensile machine equipped with thermal enclosure. For each thermal cycle, the applied stress was increased stepwise, using the same specimen throughout the test. Transmission electron microscopy (TEM, Philips CM200) operating at 200 kV was used for microstructure observations.

3. Results

3.1. Characterization of the solution treated state (ST)

It is essential to obtain precise information about the transformation temperatures in order to characterize the martensitic

Table 1 – Chemical composition (at%) of Ti–24Nb alloy.

Element (at%)	Ti	Nb	C	N	H	O
Incertitude	($\pm0.3\times10^{-2}$)	($\pm0.3\times10^{-2}$)	($\pm0.25\times10^{-2}$)	($\pm0.15\times10^{-2}$)	($\pm0.5\times10^{-2}$)	($\pm0.25\times10^{-2}$)
Ti–24Nb	69.45	23.73	0.51	0.62	3.12	2.56