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Research Paper

Effects of micro- and nano-scale wave-like structures on fatigue strength of a beta-type titanium alloy developed as a biomaterial

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ABSTRACT

Some newly developed β-type titanium alloys for biomedical applications exhibit distinctive heterogeneous structures. The formation mechanisms for these structures have not been completely revealed; however, understanding these mechanisms could lead to improving their properties. In this study, the heterogeneous structures of a Ti–29Nb–13Ta–4.6Zr alloy (TNTZ), which is a candidate for next-generation metallic biomaterials, were analyzed. Furthermore, the effects of such heterogeneous structures on the mechanical strength of this alloy, including fatigue strength, were revealed by comparing its strength to that of homogenous TNTZ. The heterogeneous structures were characterized micro-, submicro- and nano-scale wave-like structures. The formation mechanisms of these wave-like structures are found to be different from each other even though their morphologies are similar. It is revealed that the micro-, submicro- and nano-scale wave-like structures are caused by elemental segregation, crystal distortion related to kink band and phase separation into β and β′, respectively. However, these structures have no significant effect on both tensile properties and fatigue strength comparison with homogeneous structure in this study.

1. Introduction

A Ti–29Nb–13Ta–4.6Zr (mass%) alloy (referred to as "TNTZ" hereafter) is a multifunctional β-type titanium alloy with low Young's modulus, good mechanical properties, and favorable biocompatibilities (Kuroda et al., 1998; Niinomi, 2003, 2008a). TNTZ offers superior elastic behavior and Invar properties by changing the chemical composition (Niinomi et al., 2008b; Nakai et al., 2009). It was reported by the authors in a previous study that the micro-scale wave-like structure was caused by elemental segregation in TNTZ (Narita et al., 2011), which occurred during solidification because of the higher melting points of Nb and Ta. This segregation makes it difficult to fabricate large ingots of TNTZ with well-controlled compositions. Min et al. (2010) also reported this kind of micro-scale elemental segregation in Ti–15Mo. Biomedical beta-type titanium alloys with a low Young's modulus have high sensibilities for elemental compositions. The fluctuation of β-stabilizers results in differences in the microstructures and properties of these alloys (Hao et al., 2002; Niinomi, 2008a; Zhou et al., 2004; Zhao et al., 2012a, 2012b). Heterogeneous precipitation occurs due to micro-scale elemental

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segregation because the effects of heat treatment such as aging treatment differ depending on the Mo equivalent values (Min et al., 2011; Takesue et al., 2009). Although a homogeneous microstructure is preferred for fatigue strength in general, TNTZ with micro-scale elemental segregation subjected to aging treatment exhibits superior fatigue strength (Narita et al., 2011). However, the mechanism for this phenomenon has not been yet completely revealed. Therefore, TNTZ with the wave-like structure and TNTZ with a homogeneous structure were investigated in micro- and nano-scale using electron backscattered diffraction (EBSD) and a transmission electron microscope (TEM). Additionally, the effect of the wave-like structure on mechanical strength, including fatigue properties was evaluated in this study.

2. Materials and methods

2.1. *Materials preparation*

TNTZ with the wave-like structure, referred to as “WS” hereafter, and TNTZ with a homogeneous structure, referred to as “H” hereafter, were prepared by thermomechanical treatments, as shown in Fig. 1. An ingot of TNTZ weighing approximately 20 kg was fabricated by the levitation casting method. The ingot was subjected to hot-forging at 1273 K in air, forming a bar with a diameter of 25 mm. The WS samples were prepared without homogenization after hot-forging, whereas the H samples were subjected to long-term homogenization at 1273 K for 259.2 ks in vacuum, followed by water-quenching to eliminate microsegregation. Both samples were then subjected to the same thermomechanical treatment. The oxide layer of both samples was then removed,

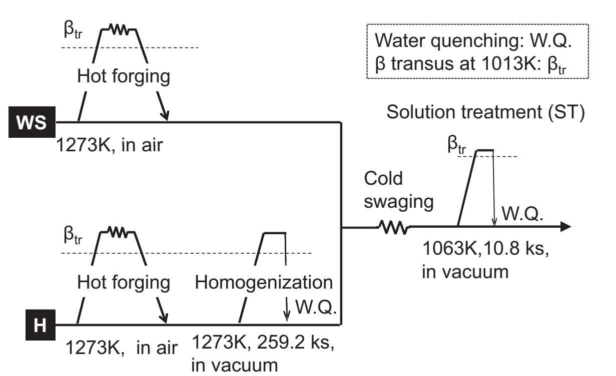

Fig. 1 – Thermo-mechanical treatments for TNTZ with micro-scale wave-like structure (WS) and long-term homogenized TNTZ (H).

leaving bars with a diameter of 20 mm. After this peeling procedure, the samples were subjected to cold swaging with a reduction ratio of approximately 90%, resulting in bars with a diameter of 6.5 mm. The samples were then solution treated (abbreviated to “ST”) at 1063 K for 10.8 ks in vacuum and then water-quenched for grain refinement. The chemical compositions obtained by inductively coupled plasma optical emission spectrometry (ICP-OES) and the carrier gas fusion method for the WS and H samples before solution treatment are shown in Table 1.

2.2. *Microstructural evaluation*

The microstructures of the samples were evaluated by field emission scanning electron microscopy (FESEM), electron probe microanalyzer (EPMA), EBSD and TEM. The sample was cut into a specimen with a thickness of 2 mm and a diameter of 6.5 mm. The cross-sectional surfaces of the specimens were wet-polished using emery papers of up to #4000 grit and buff-polished with a colloidal SiO_2 suspension. The X-ray diffraction (XRD) analysis was carried out on the longitudinal plane of the sample in scanning angles ranging from 20° to 110° with Cu Kα radiation at a voltage of 40 kV and 40 mA.

2.3. *Evaluation of mechanical properties*

Young's modulus, tensile properties, and fatigue properties were evaluated in this study. For the Young's modulus measurement, the samples were cut into specimens with a length of 50 mm and a diameter of 5.0 mm. The surfaces of the specimens were wet-polished using emery papers of up to #1500 grit. The Young's modulus measurement was carried out by the free resonance method. For the tensile test, the samples were machined into dog-bone-shaped specimens with a grip section diameter of 5.0 mm, a gauge section diameter of 3.0 mm, a gauge length of 11.0 mm, and a curvature radius of 4.0 mm at the shoulder. The surfaces of the specimens were wet-polished using emery papers of up to #1500 grit. For the fatigue test, the samples were machined into dog-bone-shaped specimens with a grip section diameter of 6.0 mm, a gauge section diameter of 3.0 mm, a gauge section length of 11.0 mm, and a curvature radius of 15.0 mm at the shoulder. The surfaces of the specimens were wet-polished using emery papers of up to #4000 grit and buff-polished with a colloidal SiO_2 suspension. The fatigue test was carried out using an electric servo machine at a frequency of 10 Hz and a stress ratio of $R=0.1$ in the tension–tension mode in air at room temperature. The fatigue limit was defined as the maximum cyclic stress over 10^7 cycles. After the fatigue test, the fractured surfaces were observed by SEM.

Table 1 – Chemical compositions obtained by ICP-OES and carrier gas fusion method for TNTZ with micro wave-like structure (WS) and long-term homogenized TNTZ (H) before solution treatment.

(mass%)	Ti	Nb	Ta	Zr	O	N	H
WS	Bal.	29.7	13.1	4.75	0.0776	0.007	0.0143
H	Bal.	29.6	13	4.89	0.0748	0.0077	0.0122