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Materials Science and Engineering C 25 (2005) 389 – 397
MATERIALS SCIENCE & ENGINEERING C
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Phase transformations in Ti–35Nb–7Zr–5Ta–(0.06–0.68)O alloys

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Received 26 November 2004; received in revised form 20 January 2005; accepted 24 January 2005
Available online 12 May 2005

Abstract

The phase transformations occurring in Ti–35Nb–7Zr–5Ta–(0.06–0.68)O β solution treated and aged between 427 and 593 °C for 8 h have been investigated. Aging at 427 °C resulted, respectively, in ω, ω+α and α phase formation for 0.06, 0.46 and 0.68 wt.% O. A modification in ω phase morphology, from near circular to ellipsoidal, was also observed with increasing O from 0.06 to 0.46 wt.%.

Aging at higher temperatures resulted in resolution of the ω phase in 0.06 wt.% O. Lenticular α precipitation was observed at higher O content, the volume fraction of α increasing with increasing O at a constant aging temperature and with increasing aging temperature at a constant O content. The latter also resulted in coarsening of the α precipitates and an increase in their aspect ratio. Finally aging of these alloys resulted in the formation of precipitate free zones (PFZs) along prior β grain boundaries, the width of these zones increasing with an increasing aging temperature. These observations are consistent with the ability of O to suppress ω phase formation through interruption of the ⟨111⟩ lattice displacement required for this phase's formation, while promoting α phase formation at higher O content, presumably through local ordering within the β phase.
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Keywords: Biomaterials; Aging; Omega phase; Phase transformations; Oxygen; Metastable β-titanium alloys

1. Introduction

Detailed examination of Ti–Nb alloys, Table 1, has shown that these materials after β solution treatment may, depending on composition and cooling rate, contain β and/or various quantities of orthorhombic martensite (α″), ω or primary α. For example, rapid cooling of Ti–(13–26)Nb–(22–38)Ta ternary alloys has been found to result in the formation of a complex β+α″+ω microstructure, the volume fraction of α″ decreasing with an increase in Nb+Ta content [2]. Slower cooling rates will result in formation of the α+β+ω phases. Furthermore, addition of 4.1–4.6 wt.% Zr has been observed to completely suppress α″ formation, β+ω being observed on rapid cooling [2]. Finally increasing the interstitial content tends to counteract this effect of Zr addition, α″ again being observed on rapid cooling [1].

Decomposition of β solution treated and rapidly cooled Ti–Nb alloys upon subsequent aging may additionally occur either by direct precipitation of the stable α phase, or may involve a two step process wherein the first stage includes formation of another metastable phase, hcp ω phase, subsequent aging for longer times resulting in precipitation of α [1–9].

Among the Ti–Nb alloys previously investigated, Ti–Nb–Zr–Ta alloys, because of their relatively low elastic modulus when compared for example to other titanium alloys, are attractive candidate materials for biomedical applications [1]. These studies have moreover shown that the elastic modulus may be altered through the addition of O, increasing O content resulting in an increase in the modulus, this effect being associated with local rearrangement of O as an interstitial solid solution within the β phase [10]. While normally used in the β solution treated

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doi:10.1016/j.msec.2005.01.022

Table 1
Phase formation in Ti–Nb alloys

Alloy	Cooling rate	Phases present
Ti–(16–23 at.%)Nb	WQ	β+α″+ω [1]
Ti–(23.4–26 at.%)Nb	WQ	β+ω [1][a]
Ti–(16–23 at.%)Nb	AC	α+β+ω [1]
Ti–(29–36)Nb–(5–12)Ta–(6.9–7.3)Zr	WQ	β+ω [2]
Ti–29Nb–13Ta–4.6Zr	WQ	β+α″ [3]
Ti–35Nb–7Zr–5Ta	WQ	β+ω [2]

WQ: water quench; AC: air cool.
[a] Interstitial dependent.

condition, where their elastic modulus is minimized, other applications for this alloy system are being considered where enhanced tensile and fatigue performance will be required. Improvement in the tensile properties achieved by increasing O content and/or aging have been reported elsewhere, however the phase transformations associated with it are not completely understood [11–13]. The current study reports the effect of O on the phase transformations in these alloys, particularly at the rather high levels previously examined following β solution treatment.

2. Experimental procedure

Three Ti–35Nb–7Zr–5Ta alloys containing 0.06, 0.46 and 0.68 wt.% O were examined in this investigation, Table 2. These were produced by vacuum arc melting, the differing O content being controlled through rutile addition. Following solidification the materials were hot forged and rolled to 16 mm diameter rod prior to β solution treatment for 1 h at 850 °C, 840 °C and 900 °C, respectively. After water quenching the alloys were aged at temperatures between 427 and 593 °C for 8 h and air-cooled.

Examination of the phase transformations occurring in the systems being considered utilizing X-ray diffraction, scanning and transmission electron microscopy analyses. X-ray diffraction utilized Cu-Kα radiation (45 kV/40 mA) at a scan rate of 0.06°/min, diffraction data being collected with a Peltier solid-state detector, samples having been prepared by standard mechanical polishing techniques. Scanning electron microscopy utilized samples etched in an aqueous solution of 8 vol.% HF and 15 vol.% HNO_3. Finally, transmission electron microscopy samples were mechanically ground, dimpled and ion milled prior to examination with an Hitachi HF 2000 field emission transmission electron (FE-TEM) microscope operated at 200 kV.

Table 2
Chemical composition of Ti–35Nb–7Zr–5Ta alloys (wt.%)

Alloys	Ti	Nb	Zr	Ta	H	C	N	O
Low O	Bal.	35.3	7.2	4.9	0.001	0.013	0.002	0.06
Medium O	Bal.	34.6	7.3	5.6	0.005	0.046	0.009	0.46
High O	Bal.	34.6	7.1	5.6	0.006	0.048	0.012	0.68

3. Results

X-ray diffraction examination of solution treated Ti–35Nb–7Zr–5Ta–(0.06–0.68)O alloys suggested all were single phase β, Fig. 1. Selected area diffraction indicated however that Ti–35Nb–7Zr–5Ta–0.06O also contained a low volume fraction of extremely small ω, Fig. 2. Although attempts to directly image the ω by dark field electron microscopy utilizing various β phase zone axis and ω reflections were unsuccessful these experiments did confirm that the ω/β habit plane and orientation relationship were in agreement with that previously reported $[111]_\beta//[0001]_\omega$, $(1\bar{1}0)_\beta//(11\bar{2}0)_\omega$ [14,15]. Additionally the present study indicates that not all variants of the ω phase were equally populated, in agreement with previous investigations of ω formation in similar alloys [16–19]. For example the ω phase diffraction intensities in Ti–35Nb–7Zr–5Ta–0.06O were strongest when the incident beam was parallel to $[102]_\beta$.

X-ray diffraction analysis of Ti–35Nb–7Zr–5Ta–0.06O and 0.46O after aging at 427 °C revealed the presence of $(2\bar{1}1)_\omega$, $(301)_\omega$ and $(2\bar{1}2)_\omega$ peaks in addition to those previously associated with the primary β, Fig. 3. Dark field imaging, Fig. 4, also suggested that the morphology of the ω particles present in Ti–35Nb–7Zr–5Ta–0.06O were almost circular in shape, similar studies of Ti–35Nb–7Zr–5Ta–0.46O indicated that this increase in O content resulted in a change in ω morphology, the latter tending to be ellipsoidal, Fig. 5. Finally no evidence of ω formation was observed by either X-ray or selected area diffraction studies of Ti–35Nb–7Zr–5Ta–0.68O aged at 427 °C. Indeed X-ray diffraction of Ti–35Nb–7Zr–5Ta–0.46O and 0.68O after aging at 427 °C indicated that the former contained a

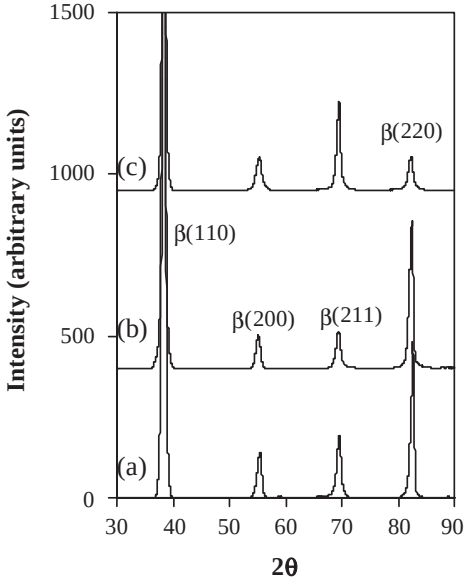

Fig. 1. X-ray diffraction patterns of solution treated Ti–35Nb–7Zr–5Ta containing (a) 0.06, (b) 0.46 and (c) 0.68 wt.% O.

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