Available online at www.sciencedirect.com
SCIENCE DIRECT®
Biomaterials 26 (2005) 2899–2907
Biomaterials
www.elsevier.com/locate/biomaterials

A comparison of the fatigue behavior of cast Ti–7.5Mo with c.p. titanium, Ti–6Al–4V and Ti–13Nb–13Zr alloys

Chia-Wei Lin, Chien-Ping Ju, Jiin-Huey Chern Lin*

Department of Materials Science and Engineering, National Cheng-Kung University, Tainan, Taiwan, ROC

Received 10 May 2004; accepted 3 September 2004
Available online 22 October 2004

Abstract

The purpose of the present study is to compare the high-cycle fatigue behavior of newly developed Ti–7.5Mo alloy with that of c.p. Ti, Ti–13Nb–13Zr and Ti–6Al–4V alloys in their as-cast state. Experimental results indicate that Ti–6Al–4V and c.p. Ti have higher stress-controlled fatigue resistance but lower strain-controlled fatigue resistance than Ti–7.5Mo and Ti–13Nb–13Zr. Among four materials Ti–7.5Mo demonstrates the best strain-controlled fatigue performance. The fracture surfaces of the present materials are comprised of three morphologically distinct zones: crack initiation zone, crack propagation zone, and the final-stage overload zone. The fatigue cracks almost always initiate from casting-induced surface/subsurface pores. A river pattern is observed in the propagation zone. In the overload zone dimples are typically observed. Three factors most significantly affecting the fatigue performance of the present materials are the presence of the casting-induced surface/subsurface pores; the location of the pores; and the inherent mechanical properties of the materials.

Keywords: Titanium alloy; Ti–7.5Mo; Fatigue; Casting

1. Introduction

Although Ti–6Al–4V alloy is widely used as an orthopaedic implant material due to its excellent corrosion resistance and mechanical properties, studies have shown that the release of aluminum and particularly vanadium ions from the alloy might cause such long-term health problems as peripheral neuropathy, osteomalacia and alzheimer diseases [1,2]. Other negative considerations for Ti–6Al–4V alloy include its relatively low wear resistance [3] and high elastic modulus that is considered to potentially cause "stress shielding effect" [4].

Fatigue has often been observed in Ti and Ti alloys and considered as an important cause to the failure of the materials. For example, fatigue failure has been reported in dental implants, removable partial denture (RPD) framework, plates, pedicle screws and cables [5–7]. The cyclic loading applied to orthopaedic implants during body motion results in alternating plastic deformation of microscopically small zones of stress concentration produced by notches or microstructural inhomogeneities [3].

Fatigue environments may be divided into two distinct groups: strain-controlled and stress-controlled. Although practically most fatigue environments in human body are a combination of the two, the very compliant nature of biological materials tends to place them toward the direction of strain-controlled fatigue [8]. Examples for strain-controlled application include pacemaker leads which require a conductive metal that can survive very high numbers of flexing motions without breaking and clasps of RPD which require adequate retention for insertion and removal of the device [9].

It has been suggested that higher implant stiffness is associated with a higher fusion rate, while lower stiffness

*Corresponding author. Tel./fax: +886 6 274 8086.
E-mail address: chernlin9@yahoo.com (J.-H. Chern Lin).

0142-9612/$ - see front matter © 2004 Elsevier Ltd. All rights reserved.
doi:10.1016/j.biomaterials.2004.09.007

is correlated to higher bone density [10]. According to the studies, assuming concentric cylindrical geometry for the bone and prosthesis, the stress shielding effect could be biomechanically reduced by using a lower modulus device [11]. Much research effort has been devoted to the study of more biocompatible and lower modulus β or near-β Ti alloys. For example, the water-quenched and aged near-β Ti–13Nb–13Zr alloy was reported to have a modulus 30–40% lower than that of mill-annealed Ti–6Al–4V alloy [3].

A binary Ti–7.5Mo alloy with an α'' phase has recently been developed in the present authors' laboratory [12]. In the as-cast state, this low-modulus alloy has a strength similar to that of as-cast Ti–15Mo and Ti–13Nb–13Zr. Since fatigue resistance is always one of the most important mechanical properties for implant materials, it is the purpose of the present study to evaluate the fatigue (particularly high-cycle fatigue) behavior of the newly developed Ti–7.5Mo alloy. The results are compared with those of as-cast commercially pure titanium (c.p. Ti), Ti–13Nb–13Zr and Ti–6Al–4V alloys.

2. Experimental procedure

The materials used for the study include c.p. Ti, Ti–6Al–4V, Ti–13Nb–13Zr and Ti–7.5Mo alloys. (Note: All compositions in this study are presented by weight.) All the materials have been prepared from raw titanium (99.8% in purity), molybdenum (99.95% in purity), niobium (99.8% in purity) and zirconium (99.8% in purity) using a commercial arc-melting vacuum-pressure-type casting system (Castmatic, Iwatani Corp., Japan). The ingots of approximately 30 g each were re-melted three times to improve chemical homogeneity. Prior to casting, the ingots were re-melted again. The difference in pressure between the two chambers allowed the molten alloys to instantly drop into a graphite mold at room temperature. The scanning electron microscopy (SEM)/energy dispersive spectroscopy (EDS) analysis indicates that, except Ti–6Al–4V, the average chemical compositions of three cast ingots (Ti–6.5Al–3.0 V, Ti–13.4Nb–12.8Zr and Ti–7.3Mo, respectively) were quite close to those designed.

X-ray diffraction (XRD) for phase analysis was conducted using a Rigaku diffractometer (Rigaku D-max IIIV, Rigaku Co., Tokyo, Japan) operated at 30 kV and 20 mA. An Ni-filtered CuKα radiation was used for the study. Surfaces of the cast alloys for microstructural study were mechanically polished via a standard metallographic procedure to a final level of 0.05 μm alumina powder, followed by etching in a Kroll's reagent containing water, nitric acid, and hydrofluoric acid (80:15:5 in volume). Morphology of the etched alloys was examined using an optical microscope (Leitz Labrorlux 12 Pols, Leica Co., Germany).

Smooth plate specimens with a rectangular cross section of 3 mm × 1.5 mm and a gage length of 6 mm for tensile and fatigue tests (ASTM E466) were discharge-machined from as-cast plates. The surfaces of the specimens were ground parallel to the specimen length axis using waterproof emery paper to a final grade of 1000.

A servo-hydraulic-type testing machine (EHF-EG, Shimadzu Co., Tokyo, Japan) was used for tensile and fatigue tests. The tensile testing was performed at room temperature at a constant crosshead speed of $8.33 \times 10^{-6}\,\mathrm{m\,s^{-1}}$. The average ultimate tensile strength (UTS), yield strength (YS) at 0.2% offset, modulus of elasticity and elongation to failure were taken from five tests under each condition. One-way ANOVA followed by Student-Newman-Kuels test [13] was used to evaluate statistical significance of the data. In all cases the statistical differences were considered significant at $p<0.05$.

A tension-to-tension stress mode was used for fatigue tests. The smooth plate specimens were subjected to uniaxial fatigue loading at room temperature in air at a frequency of 10 Hz with a stress ratio $R=0.1$, as recommended by Niinomi for testing the fatigue behavior of implant materials [14]. A sinusoidal waveform was selected [15] with one cycle representing the stance phase. The specimens were cycled with constant stress amplitude until failure, or for 10^7 cycles if they did not fail. The initial load was set up at a maximum level estimated at about 65% YS. The stress was reduced until failure did not occur.

Due to the presence of casting-induced porosity and other defects, the fatigue lives of as-cast Ti alloys are usually distributed in a wide range [16]. For this reason, the "fatigue strength" in this study is defined as the maximum stress at which the specimen does not fail at 10^7 cycles for the first time. At every load four samples were tested. The strain-controlled fatigue behavior of the materials was evaluated by the calculation of the ratio between fatigue stress and elastic modulus, as recommended by Long and Rack [3].

To locate fatigue crack initiation site and help determine fatigue fracture mechanism, the fracture surfaces of the materials were examined using a field emission SEM (XL40, Philips, Holland). The fracture surfaces were ultrasonically cleaned in ethanol and dried prior to examination.

3. Results and discussion

3.1. Phases and morphology

The XRD patterns and morphology of all investigated materials are shown in Figs. 1 and 2, respectively.