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Comparison of fatigue strengths of biocompatible Ti-15Zr-4Nb-4Ta alloy and other titanium materials

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ABSTRACT

The fatigue strength of an annealed Ti-15Zr-4Nb-4Ta alloy at 1×10^8 cycles was approximately 730 MPa. The fatigue strength of its alloy was much improved following an ageing treatment after a solution treatment. The tension-to-tension fatigue strengths of annealed Ti-6Al-4V, V-free Ti-6Al-7Nb, Ti-6Al-2Nb-1Ta, and Ti-15Mo-5Zr-3Al alloys at 1×10^8 cycles were approximately 685, 600, 700, and 350 MPa, respectively. The ratios of fatigue strength at 1×10^8 cycles to ultimate tensile strength for the α- and (α+β)-type Ti materials were higher than 65%.

1. Introduction

Metallic biomaterials have been widely used to replace failed hard tissues, such as bone screws, bone plates, compression hip screws, intramedullary fixations, short femoral nails, artificial hip joints, artificial knee joints, and spinal instruments. Among the materials used for implants today, titanium (Ti) and Ti alloys have been the focus of attention because their biomechanical and biochemical compatibilities are better than those of stainless steel and Co-based alloys. Recently, studies of conventional Ti alloys have been carried out, given the long-term use of these alloys in the human body and the possible long-term health problems associated with the release of toxic ions and fatigue fractures [1–8]. The results steered developments in Ti alloy innovation towards long-term high biocompatibility, excellent corrosion resistance, and fatigue strength.

Fatigue is a type of damage often observed in implanted metals and is regarded as an important cause of orthopedic metal implant failure [9–16]. Chao et al. investigated the causes of in vivo fracture of a cementless Ti-6Al-4V alloy hip prosthesis. Their results indicate that the fracture is initiated at the junction radius of the neck with the collared device, with approximately 90% fractures being due to a fatigue mechanism. The fracture of the prosthesis occurred because of fatigue due to the stress concentration that develops at the junction of the neck with the collared device. The existence of a notch markedly reduces the fatigue strength of Ti materials [11]. The 17,000 clinical cases that we referred to in the Food and Drug Administration database (Manufacturer and User Facility Device Experience Database, U.S.A.) of 1992–2003 have also shown a marked increase in the number of orthopedic implant failures. These findings highlight the importance of evaluating the fatigue strength and mechanical properties of Ti-based alloys that can potentially replace those used today.

In terms of developing highly durable devices, testing the fatigue strength of materials is crucial for predicting the durability of an implant. As fatigue testing methods, several standards have been established in the Japanese Industrial Standards (JIS), the American Society for Testing and Materials (ASTM), and the International Organization for Standardization (ISO). JIS T 0309 [17] and ASTM F 1801 [18] provide testing methods for investigating the fatigue of metallic implant materials. JIS T 0310 [19] standardizes a fatigue testing method for notch sensitivity and fatigue crack growth properties, and ISO 7206-4 [20] specifies a method of investigating the durability of artificial hip stems. Our fatigue test was conducted on the basis of these standardized testing methods. As Ti alloys have high notch sensitivity, the effect of specimen shape on fatigue strength was investigated.

In this study, we conducted low-cycle and high-cycle fatigue tests on Ti materials. A Ti-15Zr-4Nb-4Ta alloy [21–24] was used to determine the extent to which the fatigue strength is affected by the specimen configuration, wave frequency, wave shape, and torsional stress. To evaluate the effects of the alloy elements and microstructure on fatigue properties, the S–N curves (maximum stress vs. number of cycles) of the Ti-15Zr-4Nb-4Ta alloy were compared with those of 4 grades of commercially pure Ti (C. P. Ti): Ti-

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6Al-4V, V-free Ti-6Al-7Nb, Ti-6Al-2Nb-1Ta, and Ti-15Mo-5Zr-3Al alloys. Tensile tests were also carried out on these materials at room temperature to examine the correlation between their mechanical and fatigue strengths. Microstructural observations were conducted on Ti-6Al-4V, Ti-15Zr-4Nb-4Ta, and Ti-15Mo-5Zr-3Al alloys to examine the effects of macrostructures, particularly precipitation and matrix (α- or β-phase) structures, on their fatigue strengths.

2. Materials and methods

2.1. Alloy specimens and heat treatment

Vacuum-arc melting was conducted on the Ti-15Zr-4Nb-4Ta alloy (JIS T 7401-4) [25], α-type C. P. Ti of grades 2, 3, and 4 (ISO 5832-2) [26], (α+β)-type Ti-6Al-4 V (ISO 5832-3) [27], Ti-6Al-7Nb (ISO 5832-11) [28], Ti-6Al-2Nb-1Ta (JIS T 7401-3) [29], and Ti-15Mo-5Zr-3Al (ISO 5832-14) [30] alloys for medical implants. All the fabricated ingots were soaked and β-forged into rods under the following conditions: 1050 °C-4 h for Ti-15Zr-4Nb-4Ta; 1100 °C–3 h for C. P. Ti; 1150 °C–3 h for Ti-6Al-2Nb-1Ta, Ti-6Al-7Nb, and Ti-6Al-4 V; and 1100 °C–4 h for Ti-15Mo-5Zr-3Al. The rods were then α-β-forged (starting temperatures: 780 °C for Ti-15Zr-4Nb-4Ta; 850 °C for C. P. Ti, 930 °C for Ti-6Al-2Nb-1Ta, Ti-6Al-7Nb, and Ti-6Al-4 V; and 950 °C for Ti-15Mo-5Zr-3Al). Some of the Ti-15Zr-4Nb-4Ta rods were α-β-forged into a plate. After α-β forging, all the Ti rods and the plate were annealed at 700 °C for 2 h, except Ti-15Mo-5Zr-3Al. Ti-15Mo-5Zr-3Al was solution-annealed at 780 °C for 0.5 h and then quenched in water. Some of the α-β-forged Ti-15Zr-4Nb-4Ta rods were solution-treated at 785 °C for 1 h and then quenched in water. After the solution treatment, these Ti-15Zr-4Nb-4Ta rods were aged at 400 °C for 8 h and then cooled in air (aged Ti-15Zr-4Nb-4Ta). Table 1 shows the chemical compositions of the materials used in this study.

2.2. Specimens for fatigue test

Four fatigue test specimens were used in accordance with JIS T 0309 and ASTM F 1801. Fig. 1 shows four types of specimen cut from an annealed Ti-15Zr-4Nb-4Ta alloy. The specimens with rectangular and circular cross sections (plates and rods) are of two types. In one type, the test specimen has tangentially blending fillets between the reduced section and the ends as shown in Fig. 1(a) and (b) (uniform plate and rod specimens, respectively), and in the other type, the test specimen has a continuous radius between the ends as shown in Fig. 1 (c) and (d) (hourglass-shaped plate and rod specimens, respectively). The specimens were machined with their longitudinal directions parallel to the forged direction. To remove the inner strain formed on the surfaces during the manufacturing, the surfaces were fully ground using 600 and 1200 grit waterproof emery paper in the direction parallel to the test specimen.

2.3. Fatigue test

The fatigue test was conducted in accordance with JIS T 0309. An electro-hydraulic-servo testing machine was used in air and in

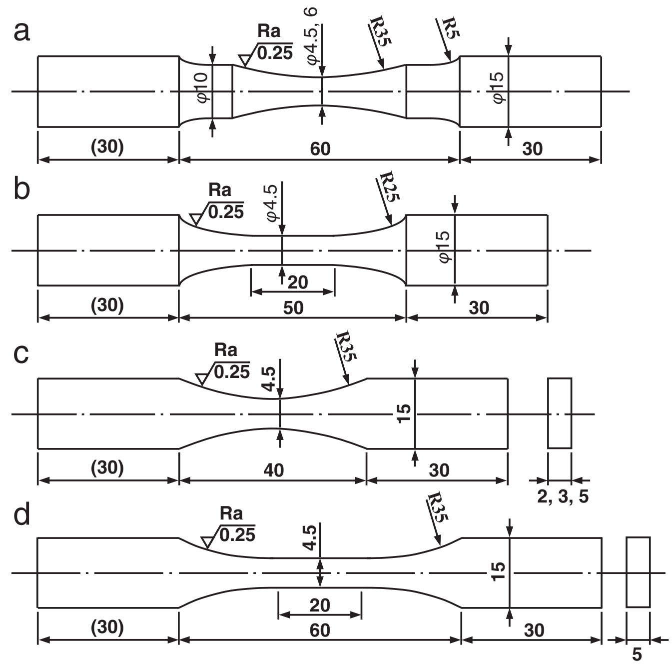

Fig. 1. Dimensions of specimens for fatigue test. (a) Rod specimen with a continuous radius between ends (hourglass-shaped rod); (b) rod specimen with tangentially blending fillets between the test section and the ends (uniform rod); (c) plate specimen with a continuous radius between ends (hourglass-shaped plate); (d) plate specimen with tangentially blending fillets between the test section and the ends (uniform plate).

Ringer's solution, which is considered to be an appropriate solution for corrosion testing in ISO 16428 [31]. For the test in the solution, the specimen was fitted into a polyethylene testing cell containing Ringer's solution and then set on a fatigue testing machine. The solution temperature inside the cell was maintained at 37 °C using heated water circulating around the cell. The tests were carried out mainly in the tension-to-tension mode with a sine wave. The stress ratio (R = (minimum stress)/(maximum stress)) was 0.1 and the wave frequencies were 1, 2, 10, 20, and 30 Hz. S–N curves (maximum stress (maximum applied load/area of cross section) vs. number of cycles) were measured for various specimens.

To determine whether the wave shape affects the S–N curve, tension-to-tension fatigue tests were conducted with annealed and aged Ti-15Zr-4Nb-4Ta alloys. The waves used for comparison were a sine wave and a hip-joint-load profile (Fig. 2(b)), which was estimated by analyzing the movement of a human hip joint and the forces acting on it [32].

Two types of torsional fatigue test were conducted using a sine wave in air. One torsional fatigue test was a torsion-to-torsion mode, in which a torsional moment was loaded at an angle of 90° to the tensile axis. The other torsional fatigue test was a tension-to-torsion mode, in which both torsional loading and axial loading were applied at the same time. When a torsional moment (T) is loaded to a rod specimen, torsional shear stress (τ, MPa) occurs towards the

Table 1
Chemical compositions (mass %) of materials used.

Alloy	Zr	Nb	Ta	Pd	Al	V	Fe	O	N	H	C	Mo	Ti
Ti-15Zr-4Nb-4Ta	15.52	4.0	4.0	0.18			0.026	0.20	0.042	0.0011	<0.005		Bal.
C.P. Ti Grade 2							0.043	0.108	0.004	0.0049	0.002		Bal.
C.P. Ti Grade 3							0.12	0.18	0.004	0.0034	0.009		Bal.
C.P. Ti Grade 4							0.197	0.275	0.003	0.0069	0.011		Bal.
Ti-6Al-4V					6.40	4.40	0.10	0.07	0.02	0.0027	0.025		Bal.
Ti-6Al-7Nb		6.55	0.01		5.97		0.22	0.18	0.01	0.0035	0.01		Bal.
Ti-6Al-2Nb-1Ta		2.0	1.13		6.22	<0.01	0.22	0.07	0.003	0.0099	0.007	0.81	Bal.
Ti-15Mo-5Zr-3Al	5.13				2.80		0.03	0.12	0.005	0.0049		14.20	Bal.