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Compressive and fatigue behavior of beta-type titanium porous structures fabricated by electron beam melting

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ABSTRACT

β-type titanium porous structure is a new class of solution for implant because it offers excellent combinations of high strength and low Young's modulus. This work investigated the influence of porosity variation in electron beam melting (EBM)-produced β-type Ti2448 alloy samples on the mechanical properties including super-elastic property, Young's modulus, compressive strength and fatigue properties. The relationship between the misorientation angle of adjacent grains and fatigue crack deflection behaviors was also observed. The super-elastic property is improved as the porosity of samples increases because of increasing tensile/compressive ratio. For the first time, the position of fatigue crack initiation is defined in stress-strain curves based on the variation of the fatigue cyclic loops. The unique manufacturing process of EBM results in the generation of different sizes of grains, and the apparent fatigue crack deflection occurs at the grain boundaries in the columnar grain zone due to substantial misorientation between adjacent grains. Compared with Ti-6Al-4V samples, the Ti2448 porous samples exhibit a higher normalized fatigue strength owing to super-elastic property, greater plastic zone ahead of the fatigue crack tip and the crack deflection behavior.

1. Introduction

The medical industry has a huge demand for superior quality implant for patients suffering from severe bone diseases [1]. Recently, the technologies of additive manufacturing (AM), which have capability to build complex porous patient-specific structures using powder materials [2,3], make it possible to further optimize the implant properties. The titanium porous structures produced by additive manufacturing are well-established components in tissue-engineering field because of its high strength, lightweight, excellent corrosion resistance, low Young's modulus and good biocompatibility [3–9].

In general, mechanical properties, such as the Young's modulus, compressive strength and fatigue properties, are influenced by the porosity of a structure [10]. The hip and knee joints must tolerate cyclic loading when people walking, thus the improvement of mechanical attributes, especially fatigue properties, is required. Further understanding the mechanical properties of structures with different porosity is necessary to optimize porosity when designing implants. Currently, Ti-6Al-4V is a widely common applied titanium alloy and its porous structure behaviors, including microstructure and the mechanical properties, have been studied extensively [11–14]. The study on Ti-6Al-4V porous sample with different relative densities from 0.73 to 1.68 g/cm^3 [14] has demonstrated that compressive strength and Young's modulus decrease with increasing porosity and that the interaction of the ratcheting effect and fatigue damage mechanism could adversely affect fatigue properties. Furthermore, the high Young's modulus and brittle deformation behavior, caused by the α' phase formation inside struts, may cause a “stress shielding” effect or reduce the life of the implant in humans [14,15]. Importantly, recent studies revealed that the Ti-6Al-4V alloy would be harmful to patient's health as Al and V elements are released [15–17]. Therefore, non-toxic β-type titanium alloys with low modulus have been attracting increased attention [2,18,19].

The β-type titanium samples made using AM technologies

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exhibit great mechanical properties and may be appropriate alternatives for implant materials. Specifically, Ti-24Nb-4Zr-8Sn (abbreviated as Ti2448) alloy is a type of β-type titanium alloy with a Young's modulus of ~42 GPa (half that of Ti-6Al-4V alloy) and tensile strength of ~820 MPa [20]. Superior mechanical properties for an alternative biomaterial for next generation implants [2] and the AM-produced Ti2448 porous specimens have been studied previously by the authors [3,21,22]. For example, selective laser melting (SLM) produced Ti2448 porous sample, with a low density of ~1 g/cm^3, exhibits excellent ductility (~14%) and strength (~51 MPa) [21]. An EBM-fabricated Ti2448 G7 porous specimen with 70% porosity records a low Young's modulus of ~0.86 GPa [22].

The fatigue properties of the Ti2448 porous samples have been studied, but focus has been on the surface of the strut and associated defects [3]. There is a lack of assessing how the microstructure of Ti2448 affects fatigue properties, which could be influenced by toughness, super-elastic properties, the crack-tip plastic zone size and crack deflection behaviors. Specifically, fatigue crack propagation behaviors may be affected by grain boundaries and the misorientation angle between adjacent grains. It was reported that dislocations or the slip-band might be blocked by grain boundaries [23,24]. Thus the crack propagation may be deflected if the neighboring grains resist dislocations or the slip bands. Such crack deflection behavior could play an active role in fatigue properties.

The mechanical property-porosity relationships, as well as the fatigue crack deflection behaviors, in Ti2448 porous samples are relatively poorly understood. Thus, this work explores the influence of porosity variation on the mechanical properties of the β-type Ti2448 alloy porous samples, in terms of Young's modulus, super-elastic property, strength and fatigue properties. The relationship between the misorientation angle between adjacent grains and the fatigue crack deflection behaviors are also discussed.

2. Experimental procedures

The Ti2448 powder used for EBM was gas atomized from a Ti2448 ingot. The powder had a nominal composition of Ti-23.9Nb-3.9Zr-8.2Sn-0.19O (in wt%) and had a spherical shape with a particle size range of 45–106 μm. Starting from the Ti2448 powder, porous rhombic dodecahedron structures containing 7 × 7 × 14 unit cells were fabricated by an Arcam A1 EBM system (Fig. 1(a)) with powder layer thickness of 70 μm. The powder in the selected area was melted by electron beam with a spot size of 200 μm generated from a tungsten filament in an EBM system. In this work, 6 groups of porous samples were prepared with nominal porosity of 67.9%, 72.5%, 75.0%, 77.4%, 79.5 and 91.2%, which were defined as A, B, C, D, E and F groups, respectively. The annealing treatment for all porous samples was conducted at 750 °C for 1 h followed by air cooling. Prior to annealing treatment, all samples were sealed in vacuum tubes to avoid oxidation. The microstructure of grains and the crack strut surfaces were observed by a JSM-6301F field emission scanning electron microscope (SEM). The SEM samples were etched by reagent (~5% HF, 10% HNO_3, and 85% H_2O) for ~120 s. The slip bands near grain boundaries were examined by transmission electron microscopy (TEM; JEOL-2100; 200 kV). Phase identification of the annealed samples was performed using X-ray diffraction (XRD; D/Max-2500PC). SEM-based electron back scatter diffraction (EBSD) analysis was performed in a field emission SEM (JSM-6301F) equipped with an Aztec EBSD system. The Young's modulus of samples with different porosity was measured using a damping analyzer (RFDAHTVP1750-C) and was averaged from three tests for each group. Finite element modeling (FEM) was conducted by using Comsol 4.2a software. Compression tests were conducted with an Instron 5869 machine at a strain rate of 0.5 mm/min. Compression fatigue tests were performed using an Instron E10000 machine with a stress ratio R of −0.1 and a frequency of 10 Hz. The fatigue tests were controlled by varied applied stresses, i.e. 1, 1.5, 2, 2.5, 3, 4, 6, 8, 10, 12, 14 and 16 MPa. The morphology of 3D strut sample, with crack initiation after fatigue test, was obtained using a Zeiss Versa 500 Micro-CT.

3. Results

3.1. Morphology and phase constitution

The 3D single unit cell model and SEM macroscopic image of the rhombic dodecahedron with porosity of 67.9% are shown in Fig. 1(b) and (c). The thickness of the as-produced strut for Ti2448 (~632 μm) is greater than that of designed strut (~460 μm). Similar phenomenon was also reported previously [12]. This is because the electron beam spot (200 μm in diameter) can create a melt pool with a diameter of ~280 μm [22]. Such a relatively larger real melting area increases the as-produced strut thickness. The EBM-produced struts display a rough strut surface, which is related to the substantial layer thickness [22]. The microstructure (Fig. 1(d)) and XRD pattern (Fig. 1(e)) show that the annealed Ti2448 samples are composed of single β phase. Both β columnar grains and equiaxed grains are evident in the microstructure (Fig. 1(d)).

3.2. Mechanical properties

3.2.1. Super-elasticity

The loading-unloading curves for porous samples with different porosity are shown in Fig. 2(a). All the sample groups were applied to cyclic uniaxial compressive loading with a total strain of 2–3% at a strain step of 0.5%. The results show that all the samples across the entire porosity range exhibit excellent super-elasticity property (only the samples with the porosity with 67.9%, 77.4% and 91.2% are displayed). Interestingly, the super-elastic property increases with increasing sample porosity. The sample with 91.2% porosity is almost fully recovered at 3% elastic strain, but the sample with 67.9% porosity exhibits a weaker super-elastic property by only recovering to 0.5%. Such difference in super-elastic property was explained by FEM analysis. The super-elastic property can be affected by the ratio of tensile stress/compressive stress. The stress distributions of the models for 67.9% and 91.2% porosity samples, at the 3% compressive strain and the compressive stresses of 35.8 MPa (67.9%) and 7.2 MPa (91.2%), are plotted in Fig. 2(b). The tensile and compressive stresses are distributed on the top and bottom of each node, respectively. The ratio of maximum tensile stress/compressive stress for the samples with 67.9% and 91.2% porosity are 0.21 and 0.27, respectively. It's reported [25] that the tensile stress is the dominant factor for the super-elastic property and the tensile/compressive stress ratio increases due to an increase of the porosity. This indicates that a higher tensile/compressive stress ratio enhances the super-elasticity property. Such a variation in tensile/compressive stress ratio can be considered as a function of increasing strain for samples with different porosity, at the same stress level. As such, a better super-elasticity property would result in a greater strain.

3.2.2. Static compression testing and Young's modulus

The uniaxial compression curves for the Ti2448 samples with different porosity are shown in Fig. 3(a). All the samples exhibit large plasticity without apparent layer-wise fracture. Clearly, both the compressive strength and yield strength decrease with increasing porosity. Compared with the Ti-6Al-4V sample with 75% porosity built from the same unit shape, which possesses a compressive strength of ~60 MPa [14], the Ti2448 sample shows a much lower compressive strength of ~38 MPa. In general, the