Evaluation of the Fatigue Properties of Ti-6Al-4V Alloy with Harmonic Structure in 4-Points Bending

by

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Titanium alloy (Ti-6Al-4V) having a bimodal “harmonic structure”, which consists of the coarse-grained structure surrounded by the network structure with fine grains, was fabricated by mechanical milling (MM) and spark plasma sintering (SPS) to achieve high strength and good plasticity. The microstructure of the MM-processed powder and the sintered compacts were characterized using a micro-Vickers hardness tester, scanning electron microscope (SEM) and an electron backscatter diffraction technique (EBSD). Harmonic structure was created in the sintered Ti-6Al-4V compacts prepared from the MM-processed powders having fine grains at its surface. The Ti-6Al-4V alloy with harmonic structure exhibited high tensile strength and good plasticity. The effects of the harmonic structure on the 4-points bending fatigue properties of Ti-6Al-4V alloy was investigated under the stress ratio $R = 0.1$ in ambient air without any controls of the temperature. The compacts with harmonic structure exhibited higher fatigue strength compared to the conventional coarse-grained material prepared from as-received initial powders. This was because the Ti-6Al-4V alloy with harmonic structure had higher tensile strength and hardness. Moreover, fatigue fracture mechanism of the Ti-6Al-4V alloy with harmonic structure was discussed from viewpoints of fractography and crystallography. As results of observing and analyzing the fracture surfaces, the Ti-6Al-4V alloy with harmonic structure failed from the coarse grain in the harmonic structure in the surface fracture mode.

Key words:
Fatigue, Titanium alloy, Ultra-fine grain, Fractography, EBSD, Powder metallurgy, Harmonic structure

1 Introduction
Titanium alloy (Ti-6Al-4V) has been widely used for various applications such as turbine blades, aerospace components and bio-implants due to its high specific strength and excellent corrosion resistance. Since mechanical properties of metallic materials are sensitive to their microstructural factors, the grain-refinement process is effective to improve their yield strength and fatigue strength. However, a homogeneous fine-grained structure leads to decrease the ductility of materials due to their plastic instability instead of increasing the strength with decreasing grain size.

Previous investigations have reported the microstructural design which improves both of the strength and ductility of materials. Wang et al. reported that pure copper with a bimodal grain size distribution created by a thermo-mechanical treatment exhibited stable tensile deformation leading to a high tensile ductility. Kondo et al. reported that pure titanium with high strength and high elongation was fabricated by the elemental mixtures of TiH$_2$ and TiO$_2$ powders in solid state. The author’s group has developed an exquisite microstructural design, called “harmonic structure”, which consists of the coarse-grained structure surrounded by the network structure with fine grains. In the previous studies, metallic materials with harmonic structure, such as pure copper, stainless steel, Co-Cr-Mo alloy, commercially pure titanium and Ti-6Al-4V alloy exhibited high strength and high ductility compared to their homogeneous counterparts. The fatigue properties of the material with harmonic structural should be examined to achieve the practical use of it.

The present work deals with the evaluation of the 4-points bending fatigue properties of the titanium alloy (Ti-6Al-4V) with harmonic structure, which exhibits superior mechanical properties. In addition, fatigue fracture mechanism of this alloy was discussed from viewpoints of fractography and crystallography.

2 Experimental Procedures
2.1 Specimen Preparation
The material used in this study was the titanium alloy (Ti-6Al-4V) powder with the chemical composition shown in Table 1. This Ti-6Al-4V powder (186 μm diameter) was produced using the plasma rotating electrode process (PREP).
The powders were mechanically milled (MM) in a Fritsch P-5 planetary ball mill with tungsten carbide vial and SUJ2 steel balls in the argon gas atmosphere at room temperature. Mechanical milling was performed at a rotational speed of 200 rpm for 25 h under the condition of the ball-to-powder mass ratio 1.8 : 1.

After mechanical milling, the powder was consolidated by a spark plasma sintering (SPS) at 850 °C for 30 min under vacuum and 50 MPa applied stress using a graphite die with 15 and 25 mm internal diameters for tensile test and 4-points bending fatigue test, respectively. Figure 1 shows the schematic illustration showing the creation of the material with harmonic structure. The titanium alloy with harmonic structure can be created by mechanical milling and spark plasma sintering because fine grains are formed by mechanical milling near the surface of powders, as discussed in section 3.1. In addition, the compact prepared from the as-received initial (IP) powders was also prepared as a coarse-grained material.

Figure 2 shows the specimen design for (a) tensile test and (b) 4-points bending fatigue test. Every specimen was polished into 1 mm thickness with emery papers (#320 to #4000), and then mirror-finished using SiO2 suspension.

### 2.1 Specimen Preparation

#### 2.2 Characterization of the Microstructure

The microstructure of the as-received initial powder, MM-processed powder and the sintered compacts were characterized using scanning electron microscope (SEM) and an electron backscatter diffraction technique (EBSD). Hardness of the as-received initial and MM-processed powders was measured at cross section using micro-Vickers hardness tester at a load of 0.254 N after polishing the powders embedded in epoxy resin. An average value of 10 measurements was considered as the representative average hardness of the powders.

### 2.3 Testing

Tensile tests and 4-points bending fatigue tests were performed in ambient air without any controls of the temperature. Tensile tests were conducted using a Shimadzu AGS-10kND tensile testing machine at a strain rate of 5.6 x 10-4 s-1. 4-points bending fatigue tests were performed at a frequency of 10 Hz under the stress ratio R = 0.1. In this study, the fatigue strength was defined as the highest maximum stress without fatigue failure at N = 107 cycles. After testing, the fracture surfaces of the failed specimen were observed using SEM and analyzed using EBSD to discuss the mechanism of initiating a fatigue crack.

### 3 Results and Discussion

#### 3.1 Microstructure of Milled Powders and Sintered Compacts

Figure 3 shows the morphology of the as-received initial Ti-6Al-4V powders and MM-processed powders. The initial powders have almost spherical shape with a smooth roughness on its surface (Fig. 3(a)). In contrast, the MM-processed powders showed an irregular shape (Fig. 3(b)) due to the plastic deformation induced by mechanical milling.

Figure 4 shows the cross-sectional SEM micrographs of the (a) as-received initial powder and (b) MM-processed
powder etched with the Kroll’s solution. Microstructure of the MM-processed powder can be classified by the outer and inner regions. Hereafter, these outer and inner regions are referred to as the “shell” and “core”, respectively. The difference of the contrast in the powder was clearly observed because the shell has deformed heavier than the core and its microstructure changed near the treated surface. Volume fraction of the shell structure was 49.2% in the MM-processed powder. As results of Vickers hardness tests, the hardness value of the shell (463.6 ± 17.2 HV) was higher than those of the core region (428.2 ± 22.4 HV) and of the as-received initial powder (396.0 ± 26.1 HV). The present authors have previously reported that almost equiaxed nano-sized grains/crystallites grains were observed near the surface of the MM-processed Ti-6Al-4V powder[10] and the milling-induced severe plastic deformation led to grain-refinement via fragmentation and sub-division of the initial coarse grains.

Figure 5 shows the EBSD image quality (IQ) maps of the compacts (φ25 mm) prepared from the (a) as-received initial powders and (b) MM-processed powders, respectively. The IP-compact has coarse acicular microstructure (Fig. 5(a)). On the other hand, the compact prepared from the MM-processed powders indicated two different microstructures; fine-grained and coarse-grained structures which were corresponded to the shell and core regions in the MM-processed powders, as shown in Fig. 4(b). The shell region formed a network structure; indicated by arrow marks in Fig. 5(b), and the core region was surrounded by the shell network. In this study, the shell region was defined as the microstructure with crystal grains less than 5 μm.

3.2 Mechanical Properties of Ti-6Al-4V Alloy with Harmonic Structure

Figure 6 shows the results of tensile tests for the Ti-6Al-4V compacts prepared from the initial and MM-processed powders, and the representative nominal stress-nominal strain curves are shown in this figure. It was obvious that the sintered compacts with harmonic structure exhibited higher tensile strength $\sigma_{\text{TS}}$ compared to the IP-compact. Moreover, there were no noticeable differences in total strain to fracture and uniform elongation between the IP-compact and the compact with harmonic structure. This was because harmonic structure promoted uniform distribution of strain during plastic deformation, resulting in suppressing the localized plastic deformation in the early stages of deformation[15]. These results indicate that the Ti-6Al-4V alloy with harmonic structure exhibits superior mechanical properties; high strength and good plasticity, compared to the IP-compact with coarse-grained acicular microstructure.

3.3 4-points Bending Fatigue Properties of Ti-6Al-4V Alloy with Harmonic Structure

Figure 7 shows the results of 4-points bending fatigue tests for the Ti-6Al-4V compacts prepared from the as-received initial and MM-processed powders. Since both series have a large scatter of fatigue life and the number of fatigue test data was not so enough to analyze the statistical aspect of the fatigue properties, $S-N$ curves were not determined in this study. Especially, one of the IP-compact specimen failed from the Si-rich microstructure at the maximum stress $\sigma_{\text{max}}$ of 717 MPa with short life ($N_i = 1.3 \times$
milling-induced severe plastic deformation led to powder etched with the Kroll’s solution. Microstructure of the grain-refinement via fragmentation and sub-division of the surface of the MM-processed Ti-6Al-4V powder\(^\text{15)}\) and the referred to as the “shell” and “core”, respectively. The inner regions. Hereafter, these outer and inner regions are MM-processed powders indicated two different the hardness value of the shell (463.6 ± 17.2 HV) was higher than those of the core region (428.2 ± 22.4 HV) and of the microstructure changed near the treated surface. Volume 100μm 100μm 100μm microstructure with crystal grains less than 5.3.3 4-points Bending Fatigue Properties of Ti-6Al-4V 3.2 Mechanical Properties of Ti-6Al-4V Alloy with as-received initial and MM-processed powders. Since both fatigue properties due to the existence of the harmonic structure having fine grains and high hardness compared to the conventional coarse-grained material.

Figure 5 shows the EBSD image quality (IQ) maps of the compacts (a)

Figure 5 shows the EBSD image quality (IQ) maps of the compacts (b)

“Core” as observed near the surface. Volume microstructure with crystal grains less than 5.3.3 4-points Bending Fatigue Properties of Ti-6Al-4V 3.2 Mechanical Properties of Ti-6Al-4V Alloy with as-received initial and MM-processed powders. Since both fatigue properties due to the existence of the harmonic structure having fine grains and high hardness compared to the conventional coarse-grained material.

Harmonic Structure

In total strain to fracture and uniform elongation between the microstructure. Moreover, there were no noticeable differences in stress-nominal strain curves are shown in this figure. It was compared to the IP-compact with coarse-grained acicular microstructure. Harmonic Structure

implies that fatigue properties of Ti-6Al-4V alloy depends on the tensile strength and that harmonic structure improves the 4-points bending fatigue properties of Ti-6Al-4V alloy due to its high tensile strength in the macroscopic analysis. However, since there were differences in the normalized fatigue strength \(\sigma_{\text{mean}}/\sigma_T\) between every specimen, statistical S-N properties should be investigated in future works.

3.4 Mechanism of Initiating Fatigue Crack in the Ti-6Al-4V Alloy with Harmonic Structure

Fracture surfaces of all the failed specimens were observed by means of SEM to clarify the mechanism of initiating a fatigue crack in the microscopic analysis. In this study, every specimen failed in the surface fracture mode. Figure 9 shows a typical feature of the fracture surfaces of the IP-compact observed at angle of 20 degrees. A large facet was clearly observed at the crack initiation site. The size and shape of this facet were corresponded to the packet of the coarse acicular microstructure of the IP-compact (Fig. 5 (a)).

Figure 10 shows a typical feature of the fracture surfaces of the material with harmonic structure failed at the maximum stress \(\sigma_{\text{max}} \approx 835\) MPa with \(N = 1.5 \times 10^7\) cycles. The results observed at angle of 0, 20 and 90 degrees for both fracture surfaces etched with Kroll’s solution are shown in this figure. In Fig. 10, the characteristic flat area like facets was clearly observed near the surface. Especially, the transgranular facet fractures were observed at the crack initiation site, indicated by the arrow marks in this figure. The results observed at angle of 90 degrees at lower magnification for both fracture surfaces are combined and shown in Fig. 11. It was clarified that a fatigue crack was initiated at the core region, represented by black colored area, and then propagated to both of the core and shell regions in the harmonic structure.

Moreover, EBSD analysis was conducted for the specimens after fatigue tests. Figures 12 and 13 show the inverse pole figure (IPF) maps obtained by EBSD for both fracture surfaces near the crack initiation site of the IP-compact and the compact with harmonic structure, respectively. In Fig. 12, the IPF maps for the fracture surfaces indicated in Fig. 9 are shown. In both series, a fatigue crack propagated across the grain boundaries; however, the crack profile of the specimen with harmonic structure was smoother.
Fig. 10 SEM micrographs of both fracture surfaces of the specimen with harmonic structure etched with Kroll’ solution observed at the angles of 0, 20 and 90 degrees ($\sigma_{\text{max}} = 835$ MPa, $N_i = 1.5 \times 10^5$).

Fig. 11 SEM micrographs of the both fracture surfaces around the crack initiation site in the core region, represented by the black color, observed at angle of 90 degrees ($\sigma_{\text{max}} = 835$ MPa, $N_i = 1.5 \times 10^5$).

Fig. 12 Inverse pole figure (IPF) maps of fracture surfaces of the IP-compact as shown in Fig. 9 ($\sigma_{\text{max}} = 664$ MPa, $N_i = 3.6 \times 10^5$).

Fig. 13 Inverse pole figure (IPF) maps of fracture surfaces of the specimen with harmonic structure ($\sigma_{\text{max}} = 831$ MPa, $N_i = 2.1 \times 10^9$).

Fig. 14 Grain boundary map of the fracture surface 1 corresponded to the IPF map as shown in Fig. 13.

compared to the IP-compact. This result implies that the microstructure influences the fatigue crack propagation behaviors of Ti-6Al-4V alloy17).

Another finding in Fig. 13 was that a fatigue crack was initiated at the core region in the material with harmonic structure as well as in Fig. 11. Figure 14 shows grain
boundary map for the fracture surface 1 corresponded to the IPF map as shown in Fig. 13. In Fig. 14, the red line represents the grain boundary greater than 15 degrees between each grain, and the gray line represents the one lower than 15 degrees, respectively. It was obvious that there were less grain boundaries near the crack initiation site. This result indicates that the coarse microstructure in the core region; especially has almost the same crystal orientation, takes as a crack starter in the material with harmonic structure. However, the fractured coarse grain in the harmonic structure was finer, as shown in Fig.9, and had higher hardness than microstructure of the IP-compact. Moreover, volume fraction of the coarse-grained structure which showed low fatigue strength was low in the specimen with harmonic structure. These are why the fatigue properties of the harmonic material are improved compared to the IP-compact.

4 Conclusions

The 4-points bending fatigue properties of Ti-6Al-4V alloy with a bimodal “harmonic structure” were investigated. In addition, fatigue fracture mechanism of this alloy was discussed from viewpoints of fractography and crystallography. The following conclusions were reached:

1) Ti-6Al-4V alloy with harmonic structure exhibits superior mechanical properties; high strength and good plasticity, compared to the specimen with coarse acicular microstructure.

2) Ti-6Al-4V alloy with harmonic structure exhibits higher fatigue strength compared to the specimen with coarse acicular microstructure due to its high tensile strength in the macroscopic analysis.

3) A fatigue crack is initiated from the coarse grain in the core region of the harmonic structure. However, the microstructure at crack initiation site in the harmonic structure is finer than that of the compact prepared from as-received initial powders.

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References


