Investigation of the grain size effect on mechanical properties of Ti-6Al-4V alloy with equiaxed and bimodal microstructures

Yan CHONG1*, Tilak BHATTACHARJEE1,2, Akinobu SHIBATA1,2, Nobuhiro TSUJI1,2
1 Department of Materials Science and Engineering, Kyoto University, Kyoto, Japan
2 Element Strategy Initiative for Structural Materials (ESISM), Kyoto University, Kyoto, Japan
*E-mail: chong.yan.6w@kyoto-u.ac.jp

Abstract. In this study, equiaxed microstructures with various grain sizes ranging from 6.0μm to 0.3μm and bimodal microstructures with grain sizes of primary α ranging from 5.0μm to 0.6μm were successfully fabricated by hot deformation and subsequent annealing in α+β two phase region in Ti-6Al-4V alloy having a martensite initial microstructure. The mechanical properties of both microstructures with different grain sizes were tested at room temperature. It was found that the strength (yield and tensile strength) of both microstructures increased with the decrease of the grain size. In the equiaxed microstructure, the uniform elongation gradually decreased with the decrease of the grain size, which was in consistent with the behavior of other metallic materials with ultrafine grains. However, in the bimodal microstructure, relatively large uniform elongation (~8%) was stably obtained regardless of the grain size. The unique and superior balance between strength and uniform elongation in bimodal microstructures was discussed, considering the contribution of interfaces between primary α grains (αp) and transformed β areas (βtrans).

1. Introduction
Ti-6Al-4V is one of the most popular α (HCP) + β (BCC) titanium alloys, which has been widely used for structural application due to its high strength-to-weight ratio and exceptional corrosion resistance [1]. A variety of microstructures including lamellar/martensite microstructure, bimodal microstructure and equiaxed microstructure can be obtained in Ti-6Al-4V by designing different heat treatments or thermomechanical processing routes [1]. Compared with the lamellar/martensite microstructure, the equiaxed and bimodal microstructures had a better balance between strength and ductility, making them more widely used in the industrial applications.

Grain refinement is the only strengthening mechanism that can increase the strength and toughness of metallic materials at the same time. It has been, on the other hand, found that yield strength and tensile strength significantly increase due to grain refinement, whereas uniform and total elongation decrease when the grain size becomes ultrafine (smaller than a few micro-meters). This is attributed to the loss of work-hardening ability and resultant early plastic instability in ultrafine-grained (UFG) materials compared to its coarse-grained counterparts. One way to restore the work hardening ability of UFG materials is to make the UFG structures dual phase comprised of soft matrix phase and hard second phase. Calcagnotto et al. [2] have shown that by reducing the ferrite...
grain size from 12.4μm to 1.2μm in a ferrite-martensite dual phase steel, both yield strength and tensile strength significantly increased while uniform elongation and total elongation were hardly affected. Similar results have also been reported in other dual phase steels [3-5].

Recently, Matsumoto and other researchers [6-11] proposed a method so-called warm deformation (T≤800°C) of martensite microstructure for obtaining ultrafine-grained (UFG) equiaxed microstructure in Ti-6Al-4V alloy, following the process using martensite initial microstructure in carbon steels [12]. Although the obtained UFG equiaxed microstructure showed a low-temperature or/and high strain rate superplasticity [7, 8], the corresponding room temperature mechanical properties suffered from poor uniform elongation (~1%) [11]. On the other hand, no efforts have been made on getting UFG bimodal microstructure as well as its mechanical properties. Therefore, the main scope of the present study is to fabricate equiaxed and bimodal microstructures with different fine grain sizes in Ti-6Al-4V alloy and then systematically investigate the influence of grain size on the mechanical properties of both microstructures.

2. Experimental methods

The chemical composition of the Ti-6Al-4V alloy used in this study was Ti-6.29Al-4.35V-0.155O-0.225Fe (in wt.%). Cylinder samples (8mm in diameter, 12mm in height) cut from the as-received billet were solution-treated at 1050°C for 30min and followed by water quenching to get a fully martensite microstructure shown in Fig. 1(a). There are two advantages in using martensite microstructure rather than lamellar microstructure as the initial microstructure of subsequent thermomechanical treatment. The first advantage is the much finer α-lamellae thickness in martensite microstructure than that in lamellar microstructure, which would enhance grain refinement of α phase in hot deformation. The average α-lamellae thickness in the martensite microstructure was measured to be ~1μm. The second advantage is the much larger area having uniform microstructures in hot-deformed specimens, which made mechanical testing of obtained equiaxed/bimodal microstructures possible and reliable. Hot compression experiments were conducted on a thermomechanical processing simulator (Thermecmaster-Z) at various deformation temperatures and strain rates. The processing route is illustrated in Fig. 1(b). The samples with the martensite microstructure were heated to the deformation temperatures at a heating rate of 10°C/s and held for 20min for homogenizing temperature of the specimen. After that, the samples were uniaxially hot compressed to a true strain of 0.8 at a certain temperature and strain rate. After the hot deformation, in order to get equiaxed microstructures, the samples were subsequently cooled at a slow cooling rate (10°C/min) to 600°C and followed by water quenching. On the other hand, in order to get bimodal microstructures, the samples were re-heated to 910°C and held for different periods of time (2~1200s) followed by water quenching.

After the hot compression and heat treatment, the cylindrical specimens were cut in half along a diameter. The sections parallel to the compression axis and including the center of the specimens were then ground and polished in standard metallographical methods. Electrical polishing in a solution of 10% perchloric acid and 90% methanol at the temperature of -30°C was conducted to reveal dual-phase microstructures. Microstructural observations of the hot-compressed and annealed specimens were carried out at the center of the specimens on the sections described above. Backscattered electron (BSE) observations were conducted using a JEOL 7800F scanning electron microscope (SEM). The accelerating voltage for the BSE experiments was 15kV. Electron backscattered diffraction (EBSD) investigations were conducted using TSL system attached to a field emission gun SEM (JEOL 7100F). The accelerating voltage was 15kV. The collected data were analyzed using OIM software. Microtensile specimens were cut from the center of the specimens, with the gauge length of 2mm, width of 1mm and thickness of 0.5mm. The broad face of the sheet-type tensile specimen was parallel to the
compression axis, and the tensile direction of the specimen was perpendicular to the compression axis. SEM characterization for all the tensile samples were conducted before tensile experiments to confirm the microstructures. Tensile strains of the micro-specimens were precisely measured by digital image correlation (DIC) method. For each thermomechanical condition, at least two specimens were tested.

**Figure 1.** (a) EBSD inverse pole figure (IPF) map of the martensite initial microstructure used in this study; (b) Illustration of thermomechanical processing for obtaining equiaxed and bimodal microstructures with different grain sizes.

### 3. Results and discussion

Through the thermomechanical processes shown in Fig. 1(b), eight kinds of equiaxed microstructures with different average grain sizes ranging from 6.0μm to 0.3μm and eight kinds of bimodal microstructures with average grain sizes of equiaxed primary $\alpha$ ranging from 5.0μm to 0.6μm were successfully fabricated. Examples of some typical microstructures are shown in Fig. 2. In Fig. 2 (a)-(c), image quality + grain boundary maps are shown for the equiaxed microstructures with average grain sizes of 6.0μm, 2.1μm and 0.5μm, respectively. In these figures, blue and red lines represent high angle and low angle grain boundaries, respectively. Fairly uniform microstructures were observed in Fig.2 (a)-(c), and the fraction of retained $\beta$ phase was about 5%. In Fig. 2 (d)-(f), BSE images are shown for the bimodal microstructures with average grain sizes of 5.0μm, 2.4μm and 0.8μm, respectively. The bimodal microstructures were comprised of primary $\alpha$ grains with equiaxed morphologies (highlighted by red dotted lines in Fig. 2(d)) and transformed $\beta$ areas, i.e., the $\beta$ areas till final cooling (highlighted by yellow dotted lines in Fig. 2(d)). The transformed $\beta$ area was made of the fine secondary $\alpha$-lamellae, which formed during the water quenching process after annealing at 910°C in Fig. 1(b), and a small amount of retained $\beta$ between the secondary $\alpha$-lamellae. It should be noted that the volume fractions of primary $\alpha$ grains were around 47% for all the bimodal microstructures with different grain sizes.

The mechanical properties of both kinds of microstructures with different grain sizes were tested at room temperature. The engineering stress-strain curves obtained are shown in Fig. 3. In the equiaxed microstructures (Fig. 3(a)), the yield/tensile strength increased with the decrease of the grain size, while the uniform elongation gradually decreased. When the grain size was smaller than 1μm, early necking of tensile specimens happened shortly after yielding, which indicated the early plastic instability due to the limited work-hardening ability of the UFG equiaxed microstructure. This result agreed with the previous studies on single-phased UFG materials [13-15]. There was also a tendency
of slight decrease in total elongation with the grain refinement for the equiaxed microstructures. In the bimodal microstructures (Fig. 3(b)), the yield/tensile strength also increased with the decrease of the grain size. However, unlike the gradual decrease of uniform elongation in the equiaxed microstructures, the uniform elongation in the bimodal microstructure was maintained after the grain refinement. Even in the UFG bimodal microstructure with a grain size of 0.6\(\mu\)m, 7% uniform elongation could be achieved, which was much larger than that of the UFG equiaxed microstructure with a grain size of 0.5\(\mu\)m. There was no clear change and tendency of the total elongation with the grain refinement in the bimodal microstructures. It can be concluded, therefore, that the grain refinement of the bimodal microstructure of Ti-6Al-4V alloy provides a possibility to increase the strength without sacrificing the ductility.

![Figure 2](image1.png)

**Figure 2.** Examples of equiaxed and bimodal microstructures with different grain sizes obtained after thermomechanical processing shown in Fig.1(b): (a) equiaxed, average grain size \(D=6.0\mu\)m, (b) equiaxed, \(D=2.1\mu\)m, (c) equiaxed, \(D=0.5\mu\)m, (d) bimodal, average grain size of equiaxed primary \(\alpha\) \(D=5.0\mu\)m, (e) bimodal, \(D=2.4\mu\)m, (f) bimodal, \(D=0.8\mu\)m.

![Figure 3](image2.png)

**Figure 3.** Engineering stress-strain curves of equiaxed (a) and bimodal microstructures (b) with different grain sizes of equiaxed \(\alpha\).
The bimodal microstructure can be technically considered as a composite of equiaxed α grains and martensite (transformed β areas), as shown in Fig. 4(a). Therefore, in order to evaluate the separate contribution of these two constituents to the excellent work-hardening ability of bimodal microstructure, the engineering stress-strain curves of fully equiaxed, martensite and bimodal microstructures are compared in Fig. 4(b). In this figure, the full martensite microstructure was obtained by solution treatment in β single phase region and followed by water quenching. The grain sizes of the equiaxed microstructure and bimodal microstructure were similar to each other (0.98μm and 0.80μm). It is interesting that neither the martensite nor the equiaxed microstructures showed good work-hardening ability and large uniform elongation, although their combination (bimodal microstructures) showed nice work-hardening and uniform elongation. In the equiaxed microstructure with a grain size of about 1μm, necking occurred shortly after yielding. In the martensite microstructure, the peak stress was reached shortly after yielding and the flow stress remained nearly the same till fracture. However, when the two constituents were combined together, the bimodal microstructure exhibited slightly lower yield strength than other two microstructures but much larger work-hardening ability with ~8% of uniform elongation. This might suggest that the strain partitioning behavior between primary α grains and transformed β areas (martensite) as well as the interfaces between these two constituents played important roles in obtaining the superior work-hardening ability in the bimodal microstructure.

![Figure 4](image)

Figure 4. (a) BSE image of a bimodal microstructure, which can be considered as a composite of equiaxed α grains and martensite (transformed β areas); (b) Comparison of engineering stress-strain curves between fully equiaxed, martensite and bimodal microstructures.

To further investigate the deformation behavior of bimodal microstructures, interrupted tensile deformation was carried out on specimens with pre-polished surfaces. A bimodal microstructure with a mean grain size (of equiaxed primary α) of 2.4μm (Fig.5(a)) was used. Engineering stress-strain curve of the bimodal microstructure shown in Fig. 5 was divided into two regions separated by the peak stress. The tensile deformation was stopped at different tensile strains, i.e., in region (1) (before the peak stress), at the peak stress, and in region (2) (after the peak stress). BSE images of the specimens at different stages are shown in Fig.5 (b), (c), and (d), respectively. In region (1) (Fig. 5(b)), which corresponded to the uniform deformation region, a number of straight slip lines were observed within the primary α grains, as were pointed out by white arrows in the figure. No clear slip lines were observed within the transformed β areas, probably due to the much finer lamellar structure inside. At the peak stress (Fig. 5(c)), more slip lines with larger contrast were observed within the primary α grains, as indicated by a white arrow. Micro-shear bands formed within the transformed β areas, as
pointed out by red arrows. After necking (Fig. 5(d) and (e)), both slip lines within the primary $\alpha$ grains and micro-shear bands within the transformed $\beta$ areas became more severe, especially within necked part of the tensile specimen (Fig. 5(e)). Some micro-cracks were found either within the micro-shear bands or at the interface between primary $\alpha$ grains and transformed $\beta$ areas.

The above results suggested that the deformation behaviors of primary $\alpha$ grains and transformed $\beta$ areas differed significantly to each other. It is believed in this study that such difference would lead to a strain partitioning between primary $\alpha$ grains and transformed $\beta$ areas, similar to the case of ferrite/martensite dual-phase steels [3-5]. The strain partitioning would also lead to a strain gradient near the interface, which would possibly activate unusual slip systems (like pyramidal) in addition to the basal and prismatic slip systems which are commonly observed in HCP-$\alpha$ of titanium alloys during deformation. With the decrease in the grain size in the bimodal microstructure, more interfaces between primary $\alpha$ grains and transformed $\beta$ areas are introduced into the microstructure and therefore more fractions of pyramidal slip systems could be activated. This can provide additional work-hardening ability compensating the loss of work-hardening ability in the fine-grained structure which would lead to a stable uniform elongation in the fine-grained bimodal microstructures. Nevertheless, the detailed mechanism needs further investigation to show clearer evidences.

Figure 5. BSE images of the bimodal microstructure (D=2.4μm) after interrupted tensile tests: (a) Initial microstructure (undeformed); (b) interrupted before the peak stress, region (1) in the stress-strain curve inserted; only slip lines marked by white arrows were observed within the primary $\alpha$ grains, (c) interrupted at the peak stress; slip lines marked by white arrows were observed within the primary $\alpha$ grains and micro-shear bands marked by red arrows were observed within the transformed $\beta$ areas; (d) interrupted after the peak stress, region (2), out of the necked region; (e) after the peak stress, region (2), out of the necked region. Some micro-cracks were observed at the interfaces between primary $\alpha$ grains and transformed $\beta$ areas.

4. Conclusions
In this study, equiaxed and bimodal microstructures with various different grain sizes were successfully fabricated by thermomechanical processing starting from martensite initial microstructure.
in Ti-6Al-4V alloy. By tensile tests at room temperature, it was found that the bimodal microstructure had a superior balance between strength and ductility, in which the strength increased with the decrease of the grain size while the uniform elongation remained unchanged. The reason for the stable uniform elongation in the bimodal microstructure was briefly discussed. It was considered that the increased volume fraction of interfaces between primary $\alpha$ grains and transformed $\beta$ areas with decreasing the grain size played an important role in enhancing work-hardening ability to maintain the uniform elongation in the UFG bimodal microstructures.

Acknowledgement

The authors were financially supported by the Cross-Ministerial Strategic Innovation Promotion Program on Structural Materials for Innovation (SIP-SM4I) in Japan, and the support is gratefully appreciated.

References

[1] Lütjering G 1998 Mater. Sci. Eng. A243 32-45
[2] Calcagnotto M, Adachi Y, Ponge D and Dierk Raabe 2011 Acta Mater. 59 658-670
[3] Calcagnotto M, Ponge D and Dierk Raabe 2010 Mater. Sci. Eng. A 527 7832-7840
[4] Son Y I, Lee Y K, Park K T, Lee C S and Shin D H 2005 Acta Mater. 53 3125-3134
[5] Calcagnotto M, Ponge D and Dierk Raabe 2012 ISIJ Int. 52 874-883
[6] Matsumoto H, Liu B, Lee S K, Li Y P, Ono Y and Chiba A 2013 Metall. Mater. Trans. 44A 3245-3260
[7] Matsumoto H, Yoshida K, Lee S H, Ono Y and Chiba A 2013 Mater. Let. 98 209-212
[8] Matsumoto H, Velay V and Chiba A 2014 Mater. Des. 66 611-617
[9] Mironov S, Murzinova M, Zherebtsov S, Salishchev G A and Semiatin S L 2009 Acta Mater. 57 2470-2481
[10] Zherebtsov S, Murzinova M, Salishchev G and Semiatin S L 2011 Acta Mater. 59 4138-4150
[11] Zherebtsov S, Kudryavtsev E, Kostjuchenko S, Malysheva S and Salishchev G 2012 Mater. Sci. Eng. A 536 190-196
[12] Tsuji N, Ueki R, Minamino Y and Saito Y 2002 Scripta Mater. 46 305-310
[13] Tsuji N, Kamikawa N, Ueki R, Takata N, Koyama H and Terada D 2008 ISIJ Int. 48 1114-1121.
[14] Tsuji N 2009 J. Phys. 165 012010
[15] Tsuji N, Ito Y, Saito Y and Minamino Y 2002 Scripta Mater. 47 893-899