Original Article

Influence of phase volume fraction on the grain refining of a Ti-6Al-4V alloy by high-pressure torsion

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\section*{A R T I C L E   I N F O}

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\section*{A B S T R A C T}

A cold-rolled Ti-6Al-4V sheet was subjected to three different heat treatments prior to processing by high pressure torsion (HPT). Quantitative measurements revealed that the volume fractions were 70% equiaxed α phase and 30% lamellar (α + β) (Ti64-1), 47% equiaxed α phase and 53% lamellar (α + β) (Ti64-2) and 25% equiaxed α phase and 75% lamellar (α + β) (Ti64-3) and the grain sizes of the α phase were 7.0 ± 2.0 μm, 9.0 ± 1.5 μm and 9.5 ± 1.5 μm, respectively. The processing by HPT was performed at room temperature with a pressure of 6.0 GPa and a rotation speed of 1 rpm. Processing of the three heat treatment batches was conducted through a total number of revolutions, N, of 1/4, 1, 5, 10 and 20. The results show that the microhardness increases with increasing numbers of turns in HPT processing and for all three conditions stable microhardness values are reached after about 20 turns. The grain sizes after 20 turns of HPT were 115 ± 30 nm in Ti64-1, 85 ± 25 nm in Ti64-2 and 75 ± 15 nm in Ti64-3 so that the grain size decreases as the volume fraction of α phase decreases and the lamellar (α + β) increases.

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\section*{1. Introduction}

Light-weight Ti-6Al-4V (Ti64) alloys possess high strength and corrosion resistance so that they have been used in aerospace engineering and power generation. They are also utilized in medical applications due to their excellent biocompatibility. As grain refining is a very effective way to improve the mechanical properties of materials, severe plastic deformation (SPD) techniques have been used to refine the microstructures of Ti-6Al-4V alloys [1–11].

There are some limited reports on grain refining of Ti-6Al-4V by high pressure torsion (HPT) since it is an effective method to refine the grains to the nano-scale [9,10]. In HPT processing, the factors influencing the grain size as well as the homogeneity of the microstructure are the numbers of turns...
in torsion, the applied pressure, the frictional effects and temperature. The influence of the phase volume fraction on grain refining of Ti-6Al-4V alloys by HPT was also investigated [10] and it was reported that higher hardness values and smaller grain sizes were attained in the alloy with approximately equal volume fractions of the constituent phases compared with an alloy containing 85% equiaxed α phase and 15% lamellar (α + β). However, the grain refining effect by HPT remains unclear when the lamellar (α + β) is the major phase constituent in the Ti-6Al-4V alloy. In the present investigation, Ti-6Al-4V materials with three different phase volume fractions were chosen and the influence of the phase volume fraction on the grain refinement of the alloy was examined through processing by HPT.

2. Experimental material and procedures

The experiments were conducted using a commercial Ti-6Al-4V alloy which was received as a cold-rolled plate with a thickness of 2.3 mm. Prior to processing by HPT, the materials were divided into three batches to conduct different heat treatments. These three batches were subjected to solution and annealing treatments at temperatures of 1183 K/45 min + 823 K/3 h, 1223 K/45 min + 823 K/3 h and 1243 K/45 min + 823 K/3 h, respectively. The processing by HPT was performed at room temperature under quasi-constrained conditions [12,13] in which there is a small outflow of material around the periphery during the pressing operation. A pressure of 6.0 GPa was applied to the disk and the lower anvil rotated with a speed of 1 rpm. Processing of the three batches was conducted through total numbers of revolutions, N, of 1/4, 1, 5, 10 and 20.

After HPT processing, measurements were taken to determine the Vickers microhardness, Hv, on the disks along the radial directions by using an FM-700 microhardness tester under a load of 0.5 kg and with a dwell time of 15 s. The procedure for taking microhardness measurements along radial directions is shown in Fig. 1. The separations between measurements were 0.5 mm and the final value was determined as the average of the microhardness measurements along 4 different directions. The location of a sample for transmission electron microscopy (TEM) is also shown in Fig. 1. The TEM samples were ground to about 50 μm and punched as round

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Fig. 1 – Schematic illustration of the HPT sample showing the four lines defined for the microhardness measurements and the position of the TEM sample.

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Fig. 2 – Microstructures of the alloy: (a) as-received and after heat treatments for (b) 1183 K/45 min + 823 K/3 h (Ti64-1), (c) 1223 K/45 min + 823 K/3 h (Ti64-2), (d) 1243 K/45 min + 823 K/3 h (Ti64-3).
3. Experimental results

3.1. Effect of heat treatment on the microstructures of the Ti64 alloy

The microstructures of the as-received material and the materials after solution and annealing treatments are shown in Fig. 2 where Fig. 2(a) shows the as-received condition and Fig. 2(b)–(d) shows the microstructures after the different treatments. It is apparent there are an elongated and distorted α phase and a small amount of β phase among the α grains after heat treatment at 1183 K/45 min + 823 K/3 h in Fig. 2(b).

After the heat treatments in the α + β region, the elongated α phase disappears and transforms to an equiaxed configuration as shown in Fig. 2(c) and (d). As the solution temperature increases, the fraction of the equiaxed α phase decreases. When treated at 1183 K, the fraction of the α phase is about 70%, but it decreases dramatically when the heat treatment temperatures are 1223 K and 1243 K (~47% and ~25%, respectively). The microstructure consists of equiaxed α phase and lamellar (α + β) structure after 1243 K/45 min + 823 K/3 h heat treatment in Fig. 2(d). This is because this solution temperature approaches the β transformation temperature and part of the α phase transforms to β phase when heated and secondary α phase forms within the grains and at the grain boundaries during cooling. The primary α grain sizes are ~7.0–9.5 µm which increase as the solution temperature increases. Quantitative measurements revealed that the grain sizes of the α phase are 7.0 ± 2.0 µm, 9.0 ± 1.5 µm and 9.5 ± 1.5 µm for Ti64-1, Ti64-2 and Ti64-3, respectively. The size of the colony and the thickness of the lamellar (α + β) structure also increase as the solution temperature increases.

3.2. Microhardness measurements

Fig. 3(a) shows plots of the average microhardness values, HV, against the distance from the center of each disk for the as-received material and after processing by HPT for different numbers of turns in Ti64-1. Prior to HPT, the microhardness of the Ti64-1 material is homogeneous and the initial microhardness is ~335. After HPT processing for 1/4 turn, the microhardness increases and the average reaches ~360, but the microhardness distribution along the radial direction is not homogeneous and ranges from ~375 at the periphery to ~345 in the center. After 1 turn, the microhardness increases...
to \( \sim 395 \) at the edge but it remains relatively low in the center at \( \sim 355 \). After 5 turns, the values at the edges of the disk are very close to the saturation value whereas the hardness values in the disk center are lower. After 10 turns, the average microhardness increases to \( \sim 405 \) with a slight increase at the periphery but a very significant increase in the center area with Hv \( \approx 380 \). When the numbers of turns are increased to 20, the average microhardness reaches a saturation at \( \sim 410 \) and the hardness distribution is essentially homogeneous except over a central diameter of about 3 mm.

From Fig. 3(b) and (c), it is readily apparent that the changes in the microhardness of Ti64-2 and Ti64-3 after HPT exhibit essentially the same trends as Ti64-1. The microhardness values increase from Hv \( \approx 345 \) to \( \sim 430 \) for Ti64-2 and from \( \sim 350 \) to \( \sim 435 \) for Ti64-3 after 20 turns of HPT, respectively. Inspection shows also that the areas having low microhardness values in the centers of the samples decrease from \( \sim 3.0 \) mm to \( \sim 2.0 \) mm and then to \( \sim 1.0 \) mm as the volume fraction of the \( \alpha \) phase decreases.

### 3.3. Microstructural observations

The microstructures after 20 turns of HPT were examined by optical microscopy (OM) and TEM. From the low magnification OM images in Fig. 4(a)–(c), it can be seen that the dark areas in the centers of the samples which are inhomogeneous exhibit well-defined turbulence patterns, which are very similar to the results reported earlier for experiments on high-purity aluminum [14]. As the fraction of the \( \alpha \) phase decreases, the inhomogeneous areas decrease so that the measured areas are \( \sim 3.0 \) mm, \( \sim 2.3 \) mm and \( \sim 1.5 \) mm for these three batches. It is interesting to note that these measured areas of turbulence correspond closely to the regions of lower microhardness as recorded after 20 turns in Fig. 3.

With a higher magnification as in Fig. 4(d)–(f), it is apparent that the microstructural changes in the centers of the disks are different for the three batches of material. In Ti64-1, the grains undergo slight deformation after HPT and part of the \( \alpha \) grains become elongated and curved as shown in Fig. 4(d). The deformation in Ti64-2 in Fig. 4(e) is severe and the lamellar (\( \alpha + \beta \)) structure becomes obscure. However, the deformation in Ti64-3 is even more severe to the extent that it is no longer possible to identify the original grain boundaries.

The TEM micrographs in Fig. 5 show bright and dark field images for the three materials after 20 turns of HPT. Measurements give an average grain size in Ti64-1 of \( \sim 115 \pm 30 \) nm in Fig. 5(a) but the microstructure is not homogeneous with a small number of short rod-like grains. The grains are more refined and more homogeneous in Ti64-2 in Fig. 5(c) with an average grain size of \( \sim 85 \pm 25 \) nm. However, the grain refinement was most effective in Ti64-3 in Fig. 5(e) with a homogeneous distribution of grains having an average size of \( \sim 75 \pm 15 \) nm.
4. Discussion

In the present investigation, three different volume fractions were attained in a Ti-6Al-4V alloy using appropriate heat treatments. The three sets of samples exhibit similar trends after processing by HPT, with the hardness values increasing such that the values increase more obviously around the peripheries of the disks and ultimately reach reasonably stable values after processing through 20 turns.

In the earlier report, it was concluded that grain refining occurs more easily when the initial unprocessed material contains approximately equal volume fractions of the two constituent phases [15,16], but the present results show that grain refining is most effective in Ti64-3, which contains 25% α phase and 75% lamellar (α + β) phase. This result suggests that the marked grain refining effect in Ti64-3 is associated with the presence of the lamellar structure which contains many boundaries. It is also worth noting that the areas of low microhardness in the centers of the samples decrease as the volume fractions of the lamellar structure increase and this effect is beneficial for the use of this alloy in commercial applications.

5. Summary and conclusions

1. A Ti-6Al-4V alloy was subjected to three heat treatments so that different volume fractions of the two constituents, α phase and lamellar (α + β) phase, were attained. The volume fractions were 70% α phase and 30% lamellar (α + β) in Ti64-1, 47% α phase and 53% lamellar (α + β) in Ti64-2 and 25% α phase and 75% lamellar (α + β) in Ti64-3 and the grain sizes of the α phase were ∼7.0, ∼9.0 and ∼9.5 μm in these three alloys, respectively.

2. These three materials were processed by HPT at room temperature under an applied pressure of 6.0 GPa. The results show that the microhardness increases as the numbers of HPT turns increases. Initially the distribution of microhardness along the radial direction is not homogeneous with lower values in the center and higher values at the edge. The microhardness reaches a maximum and the distribution along the radial direction tends to be reasonably homogeneous when the HPT is continued to 20 turns.

3. As the lamellar (α + β) structure increases, the grain refining effect by HPT becomes more effective. After 20 turns of HPT
processing, the grain sizes were ~115 nm in Ti64-1, ~85 nm in Ti64-2 and ~75 nm in Ti64-3, respectively.

4. As the fraction of the α phase decreases, the diameters of the areas of low microhardness gradually decrease from ~3.0 to ~2.0 mm and then to ~1.0 mm. This is consistent with the inhomogeneous regions of the microstructure observed after HPT through 20 turns.

Conflicts of interest

The authors declare no conflicts of interest.

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