Effects of severe plastic deformation on grain refinement and martensitic transformation in a metastable $\beta$ Ti alloy

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Abstract. A Ti-5553 metastable $\beta$ alloy was subjected to high pressure torsion (HPT) to obtain a nano $\beta$ grain structure. The $\beta$ grains were dramatically refined from an initial size of $\sim1$ mm to $<50$ nm during HPT. The stress induced martensitic $\alpha''$ plates divided $\beta$ grains into smaller domains and enabled subgrains to form even in very fine grains. The reverse $\alpha''$ to $\beta$ transformation occurred when the thickness of the $\alpha''$ laths reached $<\sim10$ nm, leading to pure $\beta$ nano-grains. Further deformation to an equivalent strain of 242 resulted in grain growth and the formation of well defined grain boundaries.

1. Introduction

The introduction of severe plastic deformation (SPD) methods to metallurgical engineering has enabled researchers to produce bulk nanocrystalline metallic alloys with superior mechanical properties such as high strength and good fatigue resistance [1, 2]. The production of nano-grained titanium (Ti) alloys has recently drawn the attention of scientists due to their low density and compatibility with the human body, making them suitable candidates for aerospace and biomedical applications. However, the majority of studies has focused on the influences of SPD on the $\alpha$-phase dominated materials, such as pure titanium and $\alpha$/$\beta$ alloys [3-6] although the lack of ductility in such alloys has made SPD processing difficult, preventing the formation of uniform nano-grained Ti alloys.

On the other hand, the $\beta$ Ti alloys have great potential to be successfully SPD processed thanks to their better ductility as compared to the $\alpha$ and $\alpha$/$\beta$ Ti alloys. Recent investigations have revealed that the stability of the $\beta$ phase could play a vital role in the formation of $\beta$ nano-grains. A stress induced martensitic phase, known as $\alpha''$, forms in metastable $\beta$-alloys during plastic deformation, contributing significantly to $\beta$ grain refinement [7-10]. It has also been claimed that $\alpha''$ is changed back to $\beta$ during SPD [7-10]. The reverse phase transformation of $\alpha'' \rightarrow \beta$ is attributed to the generation of heat either at the $\alpha''$/$\beta$ interfaces [7-9] or in adiabatic shear bands [10] as a result of mechanical work, increasing local temperatures over the austenitic starting temperature ($A_s$). There is, however, little direct experimental evidence to support the speculated mechanisms and explanations. In the present investigation, a metastable $\beta$ Ti alloy (Ti-5553) was subjected to high pressure torsion (HPT) to shed...
light on the role of the stress induced martensitic phase transformation in the microstructural evolution. Extensive transmission electron microscopy (TEM) revealed that the α” martensitic plates broke down large β grains into fine blocks, accelerating the grain refinement process, and that grain boundary migration at a very high level of strain could be responsible for the growth of nano-sized grains.

2. Experimental materials and procedures

A commercial cast Ti-5Al-5V-5Mo-3Cr (wt.%) metastable β alloy was used. Discs of 1.5 mm in thickness and 10 mm in diameter were cut from cylindrical rods of 10 mm in diameter after solution treatment at 1000 °C for 1 hour followed by water quenching, obtaining β grain sizes of ~1 mm. HPT was conducted at room temperature under a pressure of 6 GPa and at a rotation speed of 1 rpm for 0.5, 1, 5 and 20 turns. The equivalent strain is calculated using [1]

\[ \varepsilon_{eq} = \frac{2\pi N r}{t \sqrt{3}} \]  

where \( r \) is the distance from the disc centre, \( N \) the number of turns, and \( t \) the thickness of the disc.

Materials characterisation was carried out by transmission electron microscopy (TEM, FEI Tecnai F20). A scanning electron microscope with focused ion beam (SEM/FIB, FEI Nova 200 Nanolab DualBeam) was used to prepare TEM samples at \( r \) of 0.5, 2.5 and 5 mm, respectively.

3. Results

![TEM microstructures and corresponding SADPs after 0.5 turn of HPT at \( r = 0.5 \) (a-c), 2.5 (d-f) and 5 mm (g-i).](image)

**Figure 1.** TEM microstructures and corresponding SADPs after 0.5 turn of HPT at \( r = 0.5 \) (a-c), 2.5 (d-f) and 5 mm (g-i).
Figures 1a-c show the TEM results at $r = 0.5$ mm after HPT for half a turn ($\varepsilon_{eq} = 0.6$). Some needle-shaped plates are visible in figures 1a and 1b with an average thickness and length of ~100 nm and ~1 µm, respectively. The selected area diffraction pattern (SADP) shown in figure 1c identifies the plates as the stress induced $\alpha''$ phase. Figures 1a and 1b also indicate that the $\alpha''$ plates were fragmented into smaller pieces as a result of shearing during HPT. Figures 1d and 1e show TEM microstructures at a greater distance from the centre (i.e. $r = 2.5$ mm and $\varepsilon_{eq} = 3$). As seen in figure 1d, the secondary $\alpha''$ ($\alpha''_II$) formed between the primary $\alpha''$ ($\alpha''_I$) plates. Figure 1e also shows parallel $\alpha''$ and $\beta$ layers, indicating a twin structure consistent with what was reported earlier [11-13]. The length and thickness of a large number of the $\alpha''_I$ plates were measured to be ~200 and ~90 nm, respectively. The broken ring in the SADP (figure 1f) suggests the formation of a very fine grain structure after straining to $\varepsilon_{eq} = 3$. At the edge of the disc (i.e. $r = 5$ mm and $\varepsilon_{eq} = 6$), as shown in figures 1g and 1h, no $\alpha''$ was observed, although the $\alpha''$ ring was detected in the corresponding SADP (figure 1i). The more complete rings in figure 1i indicates the formation of nanocrystalline $\beta$ and $\alpha''$.

Figures 2a and 2b show the TEM microstructures at $r = 2.5$ mm after 1 turn of HPT ($\varepsilon_{eq} = 6$). Although the equivalent strain is the same as that in the specimen shown in figures 1g-i, martensitic plates of 10-20 nm in thickness are visible. The SADP shown in figure 2c indicates a sharper and more complete $\alpha''$ ring compared to that in figure 1i, indicating the presence of a higher amount of the martensitic phase. Further straining to 5 turns at $r = 5$ mm ($\varepsilon_{eq} = 30$), however, contributed to the apparent disappearance of the $\alpha''$ plates in the bright field (figure 3a) although a faint, broken $\alpha''$ ring in SADP (figure 3b) suggests the martensitic phase still exists.

**Figure 2.** (a) Bright field, (b) dark field and (c) SADP at $r = 2.5$ mm after HPT for 1 turn.

Figures 4a and 4b show the dark field TEM image and SADP, respectively, at $r = 2.5$ mm after HPT for 20 turns ($\varepsilon_{eq} = 242$). The average $\beta$ grain size was measured to be 30-50 nm (figure 4a) and no $\alpha''$ was detectable in the SADP (figure 4b). The $\beta$ grain boundaries in figure 4a are much better-defined compared to those in figures 2 and 3.
4. Discussion

4.1. Grain refinement mechanism

Molybdenum equivalency ($M_{O_{eq}}$) is one of the most commonly used criteria for determining the stability of the $\beta$ phase in Ti alloys. It is reported that the alloys with $M_{O_{eq}} < 10$ wt.% are not stable enough to prevent the formation of the stress induced $\alpha''$ martensitic phase during deformation [14]. $M_{O_{eq}}$ is calculated to be 8.1 wt.% for Ti-5553 and $\alpha''$ is expected to form during HPT as observed in figures 1-3. According to previous studies on such metastable $\beta$-Ti alloys [7-9], the formation of the martensitic plates would divide large $\beta$ grains into smaller blocks, facilitating grain refinement. The results of the present work provide consistent evidence. As seen in figures 1a and 1b, the $\alpha''$ plates broke down the $\beta$ grains into smaller blocks and as the deformation continued the secondary $\alpha''$ ($\alpha''_{II}$) formed (figure 1d), dividing $\beta$ into even finer domains. Further, the sizes of the $\alpha''$ plates became smaller with increasing strain (figures 1d-i, 2 and 3) due to their fragmentation (figures 1a and 1b), leading to plates of 10-20 nm at $\varepsilon_{eq} = 6$ (figure 2). However, the division of $\beta$ grains by itself cannot produce a nanocrystalline $\beta$ structure since the divided $\beta$ blocks all have the same orientation. The
generation of a high density of dislocations inside these small domains during HPT would lead to fine subgrains and eventually grains with high angle boundaries from the rotation of the subgrains. The dividing by \(\alpha''\) would repeat in these smaller \(\beta\) grains, accelerating the grain refinement process.

Figures 1g-i, 3 and 4 show that the amount of \(\alpha''\) decreases with increasing strain and it completely disappeared at sizes of \(< 10\) nm at \(\varepsilon_{eq} = 242\). The disappearance of the \(\alpha''\) phase is attributed to a reverse \(\alpha'' \rightarrow \beta\) transformation. A local increase in temperature above \(A_s\) has been suggested as the cause of the reverse transformation [7-10]. However, calculations based on a model by Pereira et al. [15] revealed that the temperature of the HPT discs may only reach a maximum value of 66 °C, much lower than \(A_s = 166\) °C [16]. Therefore, temperature rise could not explain the reverse transformation observed in the current study. Instead, the martensitic plate’s size is viewed as a crucial factor in the reverse transformation. A model has been developed, taking the Gibbs-Thomson effect into consideration [17]. The analysis results in that the \(\alpha''\) plates with sizes of \(< 10\) nm become unstable because of a drastic increase in the Gibbs free energy. As a result of the reverse phase transformation, all \(\alpha''\) plates eventually change back to \(\beta\) and the final microstructure consists of entirely \(\beta\) grains of 30-50 nm in size (figure 4).

In comparison, the finest grain size attained in a stable Ti-20Mo alloy is in the range of 100-150 nm [18]. This is largely attributed to the presence of the stress induced \(\alpha''\) plates in Ti-5553, which not only divide the \(\beta\) grains but also act as obstacles to dislocation movement, reducing the rate of dislocation absorption into grain boundaries. Therefore, subgrains and high angle grain boundaries could continue to form even inside the \(\beta\) grains smaller than 100 nm.

4.2. Differences between microstructures at \(r = 2.5\) mm after 1 turn and \(r = 5\) mm after half a turn

The equivalent strain is apparently the same (\(\varepsilon_{eq} = 6\)) both at \(r = 2.5\) mm after 1 turn and at \(r = 5\) mm after half a turn. Nevertheless, larger \(\alpha''\) plates are observed in the former (figure 2) whereas \(\alpha''\) is only identified by SADP in the latter (figure 1i). However, the equality of strains for these two different experimental conditions is true only if the thickness of the disc is uniform across its diameter. Considering the fact that HPT discs are normally thinner at the edge, the \(\varepsilon_{eq}\) would be greater than 6 (Eq. 1). Therefore, the fragmentation and refinement of the martensitic plates at the edge of the sample \((r = 5\) mm) would be more severe than those at \(r = 2.5\) mm after one turn of HPT, leading to the formation of invisible fine \(\alpha''\) plates and probably the reverse phase transformation. In addition, the hydrostatic pressure is small at the edge and torsion is mostly responsible for the deformation [19]. In other words, the portion of torsion at the edge is greater than that closer to the centre. This may lead to more fragmentation of the \(\alpha''\) plates and reduction of their sizes at the edge so that they are not observable in bright and dark field images (figures 1g and 1h).

4.3. HPT induced grain growth

Figure 4a shows \(\beta\) grains of 30-50 nm with well-defined grain boundaries formed after HPT for 20 turns, whereas the \(\beta\) grain boundaries are not clear after 1 and 5 turns of HPT (figures 2 and 3). HPT induced grain growth may be responsible for this phenomenon. It is believed that deformation induced grain growth occurs in nanocrystalline materials as a result of grain boundary-mediated deformation mechanisms such as grain rotation and grain boundary sliding or migration. It is possible that grains have rotated and coalesced during HPT [20]. Ni et al. [21] studied this phenomenon in a Ni-Fe alloy. They showed that three-dimensional low angle subgrain boundaries, containing a high density of dislocations, formed while grains were rotating during HPT. Further straining led to the elimination of the subgrain boundaries from which dislocations glide away on different \{111\} planes. A similar mechanism could occur in the HPT of the Ti-5553 alloy. In other words, the \(\beta\) grains obtained after 1 to 5 turns of HPT could be smaller than those formed after 20 turns owing to deformation induced grain growth.
5. Conclusions

(1) Grains of < 50 nm were obtained in a commercial Ti-5553 alloy by HPT. The grain refinement is attributed to the formation of the stress induced martensitic phase, $\alpha''$, which divide large $\beta$ grains into small blocks and enable subgrains to form even in nano-sized $\beta$ grains.

(2) Fragmentation of the $\alpha''$ plates led to their decreasing thickness with increasing strain. Theoretical calculations reveal that the reverse $\alpha''$ to $\beta$ transformation is triggered when the size of the $\alpha''$ plates is reduced to $< ~10$ nm owing to significantly rising free energy from the Gibbs-Thomson effect.

(3) Even though the equivalent strain at $r = 2.5$ mm after 1 turn of HPT is calculated to be the same as that at $r = 5$ mm after half a turn, the size of the $\alpha''$ plates in the latter was much smaller. This is because the calculation assumes the same disc thicknesses at $r = 2.5$ and 5 mm, but in reality, the disc was usually thinner at the edge ($r = 5$ mm), giving rise to a larger strain and consequently smaller martensitic plates.

(4) Deformation induced grain growth may have occurred after 20 turns of HPT, making the $\beta$ grain boundaries well-defined.

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