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Formation of equiaxed $\alpha$ during ageing in a severely deformed metastable $\beta$ Ti alloy

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Careful TEM examination has confirmed the formation of equiaxed $\alpha$ in nanocrystalline $\beta$ during ageing of a severely deformed metastable $\beta$ Ti alloy. It is revealed that the Burgers orientation relationship is obeyed upon $\alpha$ nucleation, but grain growth has led to the eventual incoherent $\alpha/\beta$ interfaces. In addition, intragranular acicular $\alpha$ would form when the $\beta$ grains prior to ageing are much coarser than $\sim$10 nm.

Thanks to their high strength, good ductility and biocompatibility, beta titanium ($\beta$-Ti) alloys have found many applications [1]. The main strengthening mechanism is $\alpha$ precipitation in $\beta$ [2,3]. The precipitation starts with grain boundary alpha (GBA) followed by acicular $\alpha$ branching from either GBA/$\beta$ or $\beta/\beta$ interfaces and nucleating inside $\beta$ grains (intragranular $\alpha$) [4–6]. The acicular $\alpha$ obeys the Burgers orientation relationship (BOR) with $\{110\}_\beta/(0002)_\alpha$ and $\{1T1\}_\beta//\{1120\}_\alpha$ [7], creating coherent $\alpha/\beta$ interfaces with low growth rates [8]. Recently, it has been shown that severe plastic deformation (SPD) can bring about a change in the $\alpha$ morphology from acicular to equiaxed [9–12]. Although such a change is attributed to a large number of dislocations and substructures in $\beta$ following SPD, the proposed mechanisms are mostly speculative with little experimental support. In particular, it is unclear whether the BOR is still obeyed and whether the equiaxed $\alpha$ can be formed inside $\beta$ grains, given that the BOR is not observed and equiaxed $\alpha$ is only found at triple junctions of nano $\beta$ grains, owing to a lack of detailed observations of the nucleation stage of $\alpha$ precipitation.

The present study aims to explore the mechanism for the $\alpha$ morphology change by examining $\alpha$ precipitation in a severely deformed metastable $\beta$ Ti alloy, focusing on the very early stages. It was revealed that $\alpha$ nucleated following the BOR, but the coherent interfaces were replaced by incoherent ones after grain growth, and an entirely equiaxed $\alpha$ structure was only achieved with $\beta$ grain sizes of $<\sim$10 nm while intragranular acicular $\alpha$ formed in coarser grains.

A commercial Ti–5Al–5V–5Mo–3Cr (wt.%) metastable $\beta$ alloy was used. Cylindrical rods of 10 mm in diameter were cut from the casting and solution treated (ST) at 1000 °C for 1 h followed by water quenching, producing $\beta$ grains of $\sim$1 mm. High pressure torsion (HPT) was conducted at room temperature on ST discs of 10 mm in diameter and 1.5 mm in thickness for 5 turns under 6 GPa and at 1 rpm. Ageing was performed at 600 °C, with a high heating rate of $\sim$100 °C/min, for 30 s to 6 h. A ST disc of 25 mm in diameter and 1 mm in thickness was also subjected to shear punching (SP) using a die with a punch of 12 mm in diameter and clearance of 0.025 mm. The SP sample was annealed at 900 °C (above $T_D$ of 860 °C) for 120 s followed by water quenching and 1 h of ageing at 600 °C. Characterisation was performed using TEM (FEI Tecnai F20) and SEM (FEI Quanta 200). TEM samples were taken at 5 mm from the HPT disc centre by SEM/FIB (FEI Nova 200 Nanolab DualBeam).

Fig. 1a and b show selected area electron diffraction pattern (SAEDP) and dark field (DF) TEM for $\beta$ after HPT, revealing a ring pattern and nanocrystalline $\beta$ with high angle grain boundaries (HAGBs), as well as the stress induced martensitic $\alpha^\prime$. TEM results for the samples after HPT and ageing for 30 and 120 s are depicted in Fig. 1c–f and g–j, respectively. Ageing for 30 s led to the disappearance of $\alpha^\prime$ and precipitation of $\alpha$ (Fig. 1c), since the ageing temperature was well above the austenitic starting temperature ($A_s$) of the alloy ($\sim$166 °C [13]).
HRTEM and inverse fast Fourier transform (IFFT) on the 30-second sample (Fig. 1e) identified α and β with d-spacings of 2.55 Å for (001)α and 2.33 Å for (110)β and showed an α nucleus (~2 nm) at a β grain boundary, creating two α/β interfaces – one coherent and the other incoherent. The coherent interface is confirmed by fast Fourier transform (FFT) of Fig. 1e with a misorientation (θ) of 4.6° across the α/β interface (Fig. 1f).

SAEDP in Fig. 1g also shows complete ring patterns for α and β after 120 s of ageing, similar to Fig. 1c. However, comparing DF TEM image for β after 120 s of ageing (Fig. 1h) to the one after 30 s (Fig. 1d) revealed that the β grain size increased from ~5–10 nm to ~20 nm with the longer ageing. Further, ageing for 120 s produced high angle (HA) α/α and α/β interfaces (Fig. 1i and j) with misorientation as large as ~40°.

Fig. 2a shows SEM of a SP sample annealed at 900 °C for 120 s (water quenched) and then aged at 600 °C for 1 h. A mixture of equiaxed and acicular α was observable in the shear zone, in contrast to the Widmanstätten α outside. Samples deformed by HPT were also aged at 600 °C for longer durations of 1 and 6 h. The STEM image and elemental maps after ageing for 1 h are shown in Fig. 2b–f, and the backscattered electron (BSE) image after 6 h in Fig. 2g. The concentration of Al in α and segregation of V, Mo and Cr in β are clear. There is no intragranular α in both Fig. 2b and g.

Several important observations can be made. First, equiaxed α forms between nanoscale β grains upon ageing, as observed earlier [10,11]. However, the α nucleation sites here are mostly HAGBs rather than the triple junctions found in [9,12]. Second and more importantly, the initial α nucleus always obeys the BOR with one of the β grains (the other interface is incoherent) although the BOR is apparently absent in the eventual equiaxed α which has undergone substantial grain growth. Third, acicular α inside β grains does form during ageing even in the severely deformed region if the initial β grains are much coarser. Fig. 3a-d propose the microstructural evolution during ageing. The grain boundaries created by SPD contain a large number of dislocations and elastically distorted layers, resulting in diffused grain boundaries [14–16] (Fig. 1b). A short ageing at 600 °C appears to have started the β recrystallisation and the grain boundary α precipitation although there is still a high level of lattice distortion (Fig. 1c, d and 3a). Further ageing would eventually lead to complete β recrystallisation with sharp, well defined boundaries and increased α precipitation (Fig. 1h and 3b). Although the estimated recrystallisation starting temperature is ~700 °C for a cold rolled VT-22 with a composition similar to Ti-5553 [17], higher than the ageing temperature used here, HPT applies much greater strains than rolling, increasing the number of crystal defects [15] and driving force for recrystallisation [18]. Together with the enhanced diffusion [19–22], the recrystallisation temperature is expected to be much lower [23]. Although α precipitation also occurs after short ageing (Fig. 1c), it is safe to assume that it has happened after β recrystallisation. This is because α nucleation requires an increase in Al as well as decreases in V, Mo and Cr, which rely on diffusion. Indeed, α precipitation can happen before β recrystallisation when the heating rate is slow to give time for elemental partitioning [17]. However, the heating rate of ~100 °C/min used appears to be too high for significant diffusion.

Therefore, α precipitation would follow β recrystallisation. Fig. 3c illustrates the nucleation of GBA between nano-sized β grains. Such
consume grain A, the coherent interface is replaced by an HA α/β interface (Fig. 3e). The growth of α grains in a similar way can lead to α HAGBs. Both scenarios are illustrated in Fig. 3d, with β grain 1 consuming β grain 2 to form an incoherent α/β interface, replacing the original coherent one, and the two α grains growing to form an HAGB. The HA α/β interface can migrate at much higher rates to form an equiaxed α particle (Fig. 3d).

Although only equiaxed α between β grains are present following ageing for 1 h at 600 °C in the HPT sample (Fig. 2b), acicular α exists inside β grains after the same ageing of the β annealed SP sample, in addition to equiaxed α (Fig. 2a). This is attributable to the initial β grain sizes before ageing. A large number of β HAGBs formed after β recrystallisation in the HPT sample, resulting in nanocrystalline β (Fig. 1d) with plenty of nucleation sites for α to reach the equilibrium amount without needing intragranular precipitation. This can be justified using the model in Fig. 4. Assuming square β grains of size d with continuous GBA layers of 2 nm in thickness (t) after ageing at 600 °C for 30 s (Fig. 1e), the area fraction of α \((f_{\text{m,α}})\) in a total area \((A_\text{t})\) of \(1000 \times 1000 \text{nm}^2\) containing \(N = (1000 / d)^2\) number of β grains is calculated using

\[
f_{\text{m,α}} = \frac{A_\alpha}{A_\text{t}} = \frac{A_\alpha - N(d-2t)^2}{A_\text{t}} = \frac{1000^2 - \left(\frac{1000}{d}\right)^2 \times (d-4t)^2}{1000^2}
\]

where \(A_\alpha\) is the total area of GBA surrounding β grains. Assuming that the area fraction equals the volume fraction \([27]\) and there is very small difference between the densities of α and β, \(f_{\text{m,α}}\) can be taken as the mass fraction of α \((f_{\text{m,α}})\). The equilibrium \(f_{\text{m,α}}\) is calculated to be ~0.7 using the Pandat software. Using Eq. 1, \(f_{\text{m,α}}\) would be ~0.7 if d is ~9 nm, i.e. GBA alone can accommodate the equilibrium amount of α. Therefore, the HPT sample with β grains of ~10 nm did not develop intragranular α after ageing. On the other hand, after 1 h of ageing the average grain size in the shear zone of the β annealed SP sample reached ~500 nm (Fig. 2a), five times greater than that attained following the same ageing in the HPT sample (Fig. 2b), suggesting that the initial grain sizes before ageing in the β annealed SP sample would be several tens of nanometres, much bigger than the critical grain size of ~9 nm. In addition, the depletion of Al and enrichment of V, Mo and Cr in β are more difficult with larger grains. Consequently, the intragranular acicular α forms in addition to the intergranular equiaxed α (Fig. 2a).

Comparing Fig. 2b and g also shows that the sizes of the α and β grains remain in the same range of ~100−200 nm after prolonged ageing up to 6 h. This is attributed to the Zener pinning \([28]\). The α grains are surrounded by highly stabilised β grains, preventing them from growth, and vice versa.

In summary, we confirm the formation of equiaxed α during ageing of a severely deformed metastable β alloy. There are two new observations of great significance. First, the BOR is obeyed at the nucleation stage of α and the equiaxed α with incoherent interfaces has developed later as a result of grain growth. Second, such equiaxed α is always found at β grain boundaries and any intragranular α would be acicular in shape in the usual way. Further, we have shown that intragranular α would form with β grain sizes of ~10 nm. The duplex structure of equiaxed α and β grains of ~100−200 nm is stable at 600 °C thanks to Zener pinning.

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Fig. 3. Schematic of microstructural evolution during ageing at 600 °C following HPT, showing (a) the start of β recrystallisation and intergranular α precipitation in a still highly distorted β matrix after a short period, (b) complete β recrystallisation with well-defined, dislocation free HAGBs and increased amount of α precipitation after a longer period, (c) precipitation of GBA with one coherent interface obeying the BOR, and (d) grain growth to form an incoherent α/β interface and an α HAGB. (e) Formation of an HA interface from grain growth, showing (left) a HAGB separating β grains A and B and a coherent interface between an α grain with (0001)α, and β grain A with (110)β, and (right) the replacement of the coherent interface by a high angle α/β interface due to the growth of grain B at the expense of grain A.

Fig. 4. Schematic of square β grains of d nm in size formed in an area of 1000 x 1000 nm² (left) and precipitation of GBA of t in thickness (right).

References