In Situ Analysis of nm-Scale Alpha Formation in Titanium Alloys

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Ti 6246 (6Al-2Sn-4Zr-6Mo wt%) is a titanium alloy that is used in the intermediate pressure compressor of jet engines due to its high specific strength and good corrosion resistance. It is used in a condition with basketweave μm-scale primary hcp α-Ti laths in a matrix of bcc β-Ti reinforced by smaller secondary α. Lengthscale strengthening by the secondary α is believed to be the main source of strength in the alloy, and provides a barrier against slip band formation, which can be deleterious for the fatigue performance [1].

In order to observe the formation of the secondary α, Ti-6246 was heated to 850°C for 6 hrs to dissolve the secondary α present in the as-received material, and then deformed via cold rolling to provide a population of dislocations. Electron backscatter diffraction (EBSD) was then used (using a Zeiss Sigma 300 fitted with an Oxford Instruments EBSD detector) to identify an α/β grain pair that lies 90° to the Burgers orientation relationship (BOR), (0002)α // {110}β. A focused ion beam (FIB) was used to lift out a foil on this zone axis and placed on DENS solutions wildfire heating chip, using an FEI Helios NanoLab fitted with an Omniprobe™. 4D scanning transmission electron microscopy (STEM) combined with fast diffraction pattern detection and in situ heating was then used to analyse strain evolution of the growing secondary α phase. This was completed using a JEOL Grand ARM with a Medipix3 detector operated at 200kV accelerating voltage and aligned in nano-beam scanning probe mode with small probe convergence semi-angle (~1 mrad), in conjunction with a DENS solutions Wildfire in situ single tilt heating holder, heating the sample to 1050°C for 106 min. Transmission Kikuchi diffraction (TKD) was completed on a foil lifted out from a specimen aged at 600°C for 30 min, that also had fine secondary α, to analyse the orientation of the resulting variants.

Figure 1 shows the as-received microstructure of Ti-6246 compared to the cold rolled and aged microstructure (subsequently analysed using TKD, figure 2) in both backscattered electron imaging and STEM-ADF. All 12 possible variants of secondary α are represented in the as-received and cold rolled and aged samples, showing that there is no significant change in orientation population of the secondary α. Figure 3 shows the evolution of strain during α is growth in the TEM. 4D STEM data has been analysed taking the α direction as (0002) and the β direction as (101)[2]. Firstly, the fine scale secondary α is not nucleating and growing from the α/β interface. It can be assumed that nucleation is occurring from defects within the β matrix. It can also be seen that there is growth along {110} slip bands within the β matrix, as in [3]. There is decreasing strain in εuu, but there is increasing strain in the shear term, εuv, as with lattice rotation. Therefore it can be assumed that as the fine secondary α forming encourages lattice extension along the growth direction [0002]α, and contraction perpendicular to this.
Fine scale secondary α formation has therefore been observed \textit{in situ} in Ti-6246 using in situ TEM heating, showing nucleation within β matrix, which is assumed to be from defects. Additionally, as the α phase grows, the strain evolution in the lattice shows lattice extension along the growth direction.

References:
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Figure 1. As-received microstructure in (a) BSE, (b) STEM-ADF cold rolled and aged in (c) BSE, (d) STEM-ADF

Figure 2. TKD of (a) as-received, (b) cold rolled and aged, plus the corresponding pole figures

Figure 3. Lattice strain measurements at 0 min and 106 min taken using 4D STEM, whilst simultaneously in situ heating.