doi:10.1017/S1431927619008183
Microsc. Microanal. 25 (Suppl 2), 2019

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

A. K. Ackerman[1*], C. Ophus[2], M. Danaie[3], P. Karamched[4], A. J. Knowles[1, 5], A. Wilkinson[4], D. Rugg[6] and D. Dye[1]

1. Department of Materials, Imperial College, London, UK
2. NCEM, Lawrence Berkeley National Laboratory, University of California, Berkeley, USA
3. Electron Physical Sciences Imaging Centre (ePSIC), Diamond Light Source, UK
4. Department of Materials, University of Oxford, Oxford, UK
5. School of Metallurgy & Materials, University of Birmingham, Birmingham, UK
6. Rolls-Royce plc, Derby, UK
* Corresponding author: a.ackerman14@imperial.ac.uk

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), $(0002)_{\alpha} // \{110\}_{\beta}$. 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 *u* direction as (0002) and the *v* 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 ε_{uu}, but there is increasing strain in the shear term, ε_{uv}, as with lattice rotation. Therefore it can be assumed that as the fine secondary α forming encourages lattice extension along the growth direction $[0002]_{\alpha}$, and contraction perpendicular to this.

Fine scale secondary α formation has therefore been observed *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.

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[4] The authors acknowledge funding from EPSRC (EP/M506345/1), Diamond Light Source Ltd. for access and support in the use of the electron Physical Science Imaging Centre (EM18190). Work at the Molecular Foundry was supported by the Office of Science, Office of Basic Energy Sciences, of the U.S. Department of Energy under Contract No. DE-AC02-05CH11231.

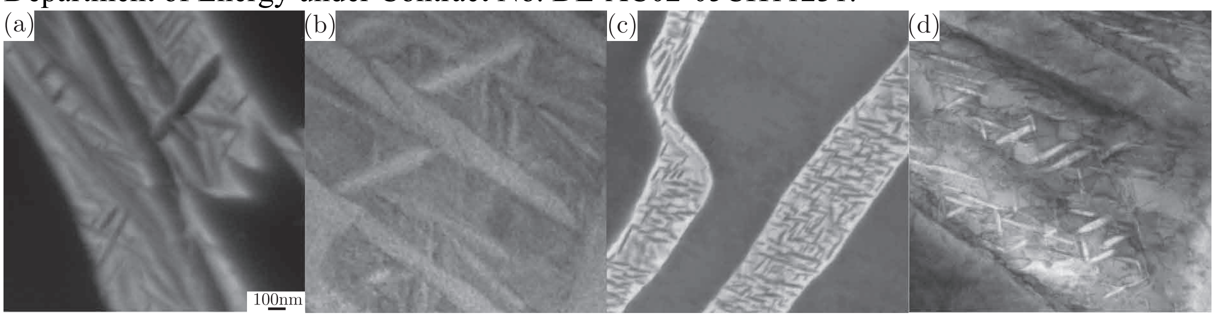

Figure 1. As-received microstructure in (a) BSE, (b) STEM-ADF cold rolled and aged in (c) BSE, (d) STEM-ADF

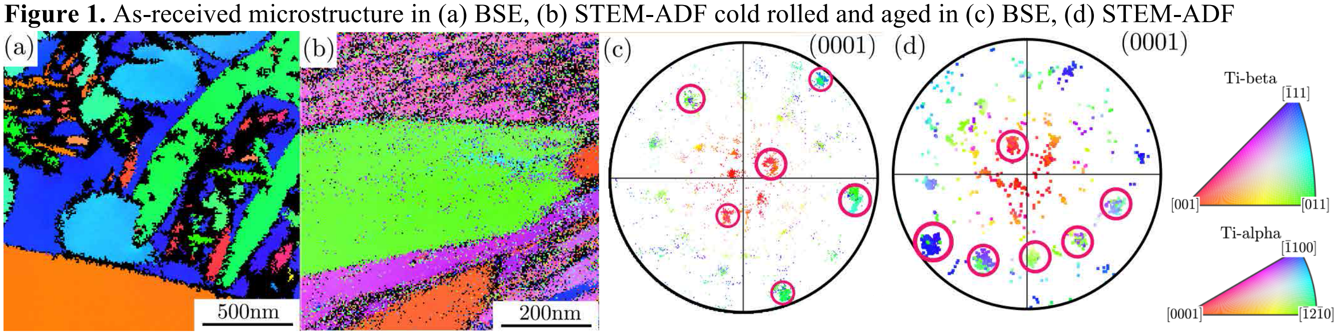

Figure 2. TKD of (a) as-received, (b) cold rolled and aged, plus the corresponding pole figures

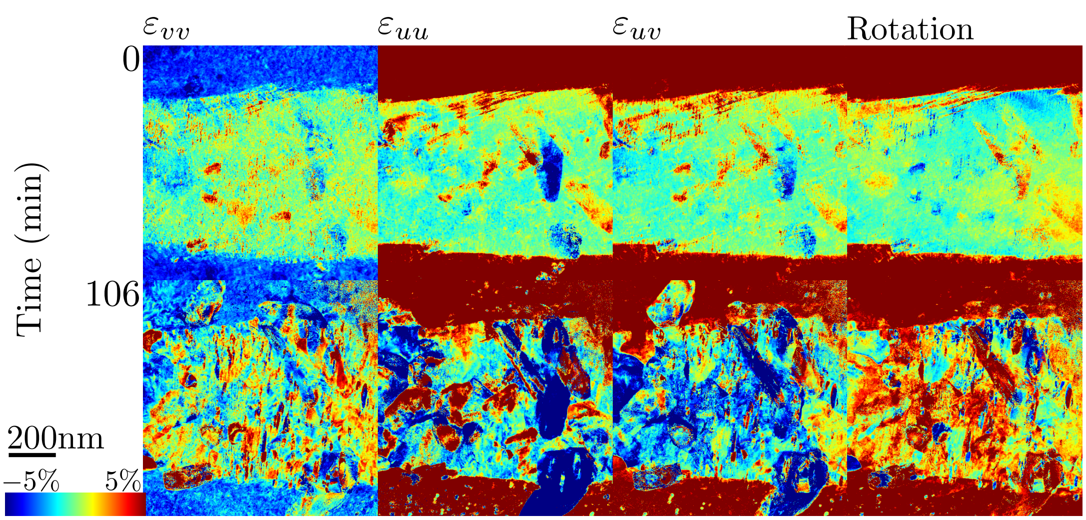

Figure 3. Lattice strain measurements at 0 min and 106 min taken using 4D STEM, whilst simultaneously in situ heating.

https://doi.org/10.1017/S1431927619008183 Published online by Cambridge University Press