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Local deformation mechanisms of two-phase Ti alloy

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This paper describes a study of local deformation mechanisms in two-phase Ti alloy, Ti–6Al–2Sn–4Zr–2Mo, by performing *in-situ* micropillar compression tests. A colony microstructure was examined and select grains identified for examination were chosen with EBSD measurements. These grains were chosen to isolate individual slip systems within each test. Micropillars of tri-crystal (α–β–α) structure were fabricated from four determined regions, and compression tests were performed using a displacement-controlled nanoindenter set inside a SEM, with a constant displacement rate. The results show that the α/β morphology significantly affects the local deformation behaviour. For these colony structures, Schmid's law in general enables anticipation of local slip activity, but the presence and morphology of the β phase can significantly alter the apparent yielding point and work hardening response. The role of interfaces within these tri-crystal pillars is discussed.

1. Introduction

High strength-to-weight ratio, corrosion resistance and excellent mechanical properties have made titanium alloys attractive to many industrial applications, particularly in gas turbine and aerostructures. However, their highly anisotropic and localised deformation behaviour, when significant fractions of the α (Hexagonal Close Packed, HCP) phase are present, lead to difficulties in understanding fatigue crack nucleation and hence in predicting lifetime and failure of components in service [1]. In the last few decades, extensive researches have been devoted to understanding deformation mechanisms in α phase titanium (e.g., CP–Ti or Ti–Al single crystals) [2–7]. These investigations have clearly shown slip/twinning behaviours and the corresponding dislocation evaluation as a function of Al content, temperature and crystallographic orientation. On the other hand, relatively little is known about the precise micromechanical deformation mechanisms in two-phase Ti alloys. Complexities on studying α/β Ti alloys arise due to the microstructure-dependent-mechanical properties, room temperature creep behaviour, microtexture and interaction between the α and β (Body Centred Cubic, BCC) phases [8–12].

Early work by Chan et al. [8] showed the deformation behaviour of a colony-structured α/β Ti alloy, Ti–8Al–1Mo–1V. Compression experiments of individual colony samples revealed that significant yield stress variations were found with respect to the angle between the slip direction and the normal to the α/β interface, and failure of Schmid's law was observed in α colony orientations where angles between the slip plane normal and the loading direction vary from 15° to 63°, except the case when slip occurred parallel to the β phase. Slip system activity strongly influences stress–strain behaviour and interestingly the authors insisted based on experimental observation that the β phase is stronger than the α phase, although this has not yet been confirmed.

Savage et al. [11] and Suri et al. [12] investigated the effects of α/β interfaces in Ti–6Al–2Sn–4Zr–2Mo–0.1Si and Ti–5Al–2.5Sn–0.5Fe, respectively, with respect to slip transmission mechanisms between the α and β phases governed by Burgers orientation relationship (BOR). Experimental observation revealed that significant anisotropy in deformation behaviour occurs in three distinctive (a_1, a_2 and a_3) basal and prismatic slip systems in the α phase which was caused by the relative misalignment with a slip system in the β phase. The morphological effect of the β phase in deformation was studied by Sandala [10] using lamellar structured Ti–6Al–2Sn–4Zr–6Mo with different widths of β. It revealed that the β width significantly affects plastic deformation behaviour, whilst no pronounced effect was found in the elastic regime. Some effects of β volume fraction can be found in recent work done by Qiu et al. [9]. With a higher β volume fraction obtained by increasing Mo content in Ti–6Al–2Sn–4Zr–*x*Mo (*x*=2–6), a smaller slip band spacing was produced and significantly reduced accumulated strains were observed during dwell fatigue tests.

Previous studies have shown some aspects of deformation mechanisms in macroscopic polycrystalline two-phase Ti alloy samples, but it is thought that small-scale experiments on a

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localised, confined area are required to improve understanding of the fundamental mechanisms on the level of the individual microstructural constituents. In recent years, Uchic et al. [13] have developed an innovative methodology for studying micromechanical behaviour of materials by adopting experiments on small-scale architectures fabricated by focused ion beam, with a nanoindenter as a means of applying force. Extensive research contributions have been devoted to investigate the length scale and size effects in plasticity of metallic materials [13–17], yet this has mainly focused on FCC materials such as Cu and Ni. Some work on Ti single crystals has been carried out to study the size effect on slip [18] and deformation twinning [19]. Recently, Ding, Gong and colleagues [20] have worked on two-phase Ti–6Al–4V using micro-cantilevers, investigating slip activities through the α/β interface. They found a cross-slip away from the $\{10\bar{1}1\}$ pyramidal plane only for compression case (not for tension) and this could elucidate asymmetric CRSS behaviour, which was similarly observed by Jones and Hutchinson [21].

In this work, we investigate local deformation mechanisms in two-phase Ti alloys using micropillar compression focusing on how two phase structures change local slip behaviour. We investigate the significance of α/β structural morphology on deformation and its effect on Schmid's law, which is used for anticipating local slip activity. This work provides the basis for further investigation on fundamental micromechanics of two-phase Ti alloys and potentially other materials.

2. Materials and experiments

2.1. Material preparation

A titanium alloy 6242 (hereinafter referred to as Ti6242), with the chemical composition (in wt%): 6.12Al–2.00Sn–3.95Zr–2.00Mo–0.012C–0.031Fe–0.021Si–0.0002H–0.072O–0.0069N and Ti balance was supplied by IMR (Institute of Metal Research, China) as a forged bar of 20 mm diameter. The bar had been produced by a triple VAR melt, followed by β forging at the temperature of T_β (β transus)+150 °C with 30% deformation (resulting in the ingot diameter from 205–210 mm to 170 mm) and α/β forging at the temperature of 30% α_p (primary α phase) with 70% deformation giving a finishing diameter of 90 mm.

The sample was sectioned perpendicular to the bar axis and prepared metallographically with SiC papers (up to 4000 grit) and then polished with ~50 nm OP-S (Oxide Polishing Suspensions) diluted with H_2O by a ratio 1:5 of OP-S:H_2O. The sample was lightly etched with Kroll's reagent for ~15 s (2% HF, 10% HNO_3 and 88% H_2O). The final etch and polish procedure was repeated 2–3 times until the grain structure was clearly visible with polarised light microscopy.

Initial studies of the as-received Ti bars revealed complex lamellar structures, where isolation of individual microstructure features would be difficult and the effect of prior strain would make interpretation of the results difficult (see Fig 1). Therefore the sample was heat treated to produce a colony microstructure with large α-lamella separated by thin β-ligaments, in clear prior-β grain structures to make micropillars of a tri-crystal (α–β–α) structure (see Fig. 2(i)). For this heat treatment, the as-received sample was held at the temperature of β transus+50 °C (i.e., 1040 °C) for 8 h and cooled down with a rate of 1 °C/min. This provided a microstructure with α phase lamella, with average widths of ~3.7 μm, and β phase ligaments of width ~0.8 μm. Note that the widths of the as-received condition were ~2 μm for α lamella and ~0.5 μm for β ligament.

2.2. EBSD maps

Electron backscatter diffraction (EBSD) maps were generated in a Carl Zeiss Auriga CrossBeam FIB-SEM with Bruker EBSD system consisting of an eFlashHR camera and Esprit v1.9 software, in order to characterise the microstructure (an example is shown in Fig. 1). High current mode was used with a probe current of 10.5 nA and an aperture size of 120 μm. The accelerating voltage of 20 kV was selected for EBSD. A larger area map (~1.6 × 1.2 mm^2 and step size with 1.2 μm) was first mapped, yet due to the large step size the β orientation was not regularly revealed within this map. Therefore higher magnification maps (~151 × 113 μm^2) with finer step sizes of 0.05–0.5 μm were captured for areas with desired crystal orientations for the micropillar tests. Crystal orientations were selected to maximise the resolved shear stress on individual slip systems and trigger single slip.

2.3. Micropillar fabrication

Micropillars of square cross-section were machined using a FEI Helios Nanolab 600. An automated routine was used for fabrication in AutoScript™. A Ga^+ ion beam of 30 kV was used with a series of currents decreasing from 9.3 nA (rough milling) to 2.8 nA (medium milling) and finally to 0.92 nA (final milling). The fabrication time for each pillar was about 17 min.

Micropillars were fabricated in grains of specific crystallographic orientations to control slip activity. Furthermore, pillars

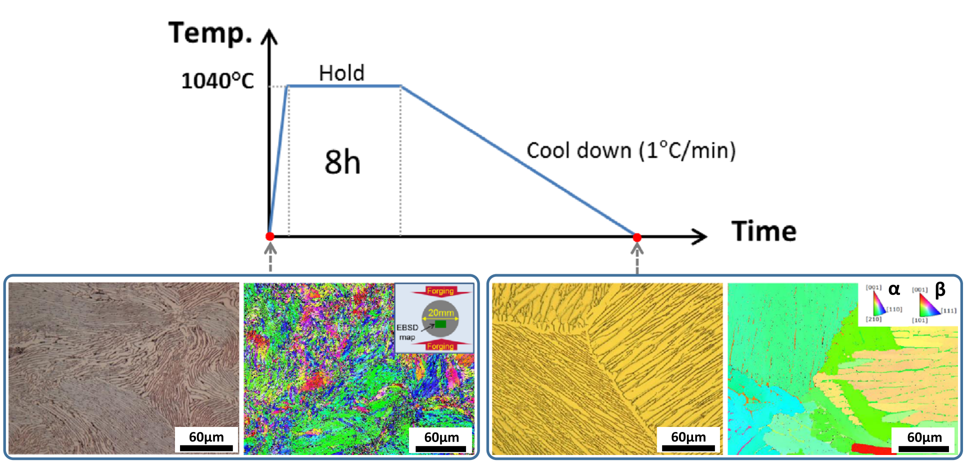

Fig. 1. Schematic diagram of heat treatment (HT) processing route, optical micrographs and EBSD inverse pole figure (IPF) maps observed with respect to normal to the forging direction, taken before and after HT: Sufficiently low cooling rate changed microstructure in as-received (before HT) Ti6242 to fully lamellar structure. All images were observed in different regions.