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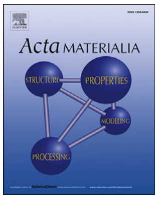

Full length article

A study of deformation twinning in a titanium alloy by X-ray diffraction contrast tomography

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ABSTRACT

A specimen of Ti–4Al deformed in-situ to about 0.7% compressive strain using neutron diffraction, and showing early stages of twinning, has been investigated in 3D using synchrotron X-ray diffraction contrast tomography (DCT). A cylindrical volume of 900 μm × 400 μm diameter was observed using DCT, containing about 400 grains of which almost 60 grains were identified to have twinned predominantly by $\{10\bar{1}2\}\ \langle\bar{1}011\rangle$ 011 tensile twinning. To consider possible twin nucleation criteria, non-twinned grains of similar orientation to the twinned grains were compared against the family of twinned grains. Such comparison highlights that the twinned grain family has a grain size distribution shifted to a higher mean value than the corresponding family of grains that has not twinned. An initial neighbourhood analysis did not reveal any significant differences of the two grain families. However, complex twin chains and clusters were identified forming a slightly imperfect network demonstrating the importance of the 3D analysis. Analysis of the parent grain orientations within those chains/clusters using the Luster-Morris parameter revealed a significantly higher propensity of prismatic $\langle a\rangle$ slip transfer compared to the neighbourhood of the non-twinned grain family while no difference was observed for the likelihood of twin shear transfer. The findings suggest that grain chains/clusters with high ability of prismatic $\langle a\rangle$ slip activity and slip transfer does promote formation and clustering of twins, which is likely associated with the build up of tensile intergranular strain along the $\langle c\rangle$ axis perpendicular to the loading direction recently suggested by crystal plasticity modelling.

1. Introduction

Metals with a hexagonal close-packed (HCP) crystal structure, such as Ti, Mg and Zr, are known to display easy $\langle a\rangle$ slip, either on the prismatic or basal plane. In the case of titanium, the most common slip mode is $\{10\bar{1}0\}\ \langle 1\bar{2}10\rangle$ prismatic slip while pyramidal $\langle c+a\rangle$ slip has been observed but only in small fractions [1–3]. This is due to pyramidal $\langle c+a\rangle$ slip having a critical resolved shear stress (CRSS) at room temperature about 3–5 times higher than for prismatic $\langle a\rangle$ slip [4,5]. However, plasticity that provides shear with a $\langle c\rangle$ component is necessary to achieve significant plastic deformation in polycrystalline titanium [6]. This is because the $\{10\bar{1}0\}\ \langle 1\bar{2}10\rangle$ prismatic slip mode alone is not sufficient to accommodate an arbitrary plastic strain, which requires five independent slip systems [7], according to the von Mises criterion [8].

More recent work has demonstrated that plasticity in polycrystalline materials can be accommodated by fewer independent slip systems per grain [1] but, in the absence of easy slip including a $\langle c\rangle$ component, twinning is commonly observed in Ti and other metals with an HCP crystal structure. This is particularly the case during compression loading [9–16]. For example, twinning can provide the majority of plastic deformation in Mg alloys, if the starting texture promotes grains ideally orientated for twinning but not for $\{0001\}\ \langle 1\bar{2}10\rangle$ basal slip [17–24].

In hexagonal titanium, four different twinning modes have been reported [11]. At room temperature, the predominant twinning mode is the $\{10\bar{1}2\}\ \langle\bar{1}011\rangle$ tensile twin [25–28,6,29,30,22,31], which corresponds to a rotation of 85° around the $\langle 11\bar{2}0\rangle$ axis. In

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some cases, this twin mode has been observed to result in almost complete grain reorientation after only modest levels of strain [32–38].

Statistical analysis of twin nucleation in hexagonal metals has suggested that twin activation does not necessary follow the traditional Schmid law, meaning that in some cases the twin variant that forms is not the one with the highest Schmid factor [35,23,39]. However, more recent work has demonstrated that once the local stress state is taken into account by the use of a crystal plasticity model, the twin variant selection follows the stress criterion [40]. The applied stress is expected to play an important role in twin nucleation, as discussed in Yoo [1] and Meyers et al. [12]. In general, they state that the applied stress is proportional to the number of twins per unit of observed area, as found similarly in Chichili [33]. This applied stress is an average resolved stress and is a rather crude approximation. To understand the increasing density of twins with increasing levels of applied stress there can be two scenarios: (i) the stress is considered as uniform field applied on an array of potential twin nucleation sites, in which the increasing levels of applied stress allow more sites to be activated; (ii) the critical stress for nucleation is met only at the most potent of the stress concentrations. When increasing the applied stress, more of the available stress concentrators provide stresses that attain the critical level. In most of the cases, the reality is most likely to correspond to a combination of these two scenarios.

Localised slip resulting in dislocation pile up at grain boundaries has also been found to play a role in twin nucleation, which has been recently investigated in commercially pure titanium grade 1 [41–44], together with the free surface relaxation and non-Schmid stress effects on twin nucleation [45]. The slip activity in a grain well aligned for prismatic $\langle a\rangle$ slip leading to twin nucleation in a neighbouring grain not well aligned for $\langle a\rangle$ type slip was shown to be significant when a tensile stress state was created [42]. Twin to twin shear transfer across grain boundaries has also been observed, particularly in boundaries with misorientations lower than 30° [41]. These correlations were quantified using the slip transfer parameter *m’*, which was first introduced by Luster and Morris [46] based on observations by Clark et al. [47].

Numerous deformation studies on metals with a HCP crystal structure have used in-situ loading in combination with neutron diffraction to study intergranular strain evolution of various grain families [48–53]. When carrying out compression loading such approach also enables easy detection of $\{10\bar{1}2\}\ \langle\bar{1}011\rangle$ twinning as the $\langle c\rangle$ axis tends to rotate into the loading direction, which is monitored by measuring the {0002} integrated peak intensity. This method is particularly useful to compare twin activities and enables one to capture the moment of early twin formation.

Considering the importance of neighbourhood in relation of twinning, several synchrotron X-ray techniques might be of particular interest as they enable 3D analysis of polycrystalline materials non-destructively. Examples here are the differential aperture X-ray microscopy (DAXM) approach that was developed at the advanced photon source (APS) by a group from the Oak Ridge National Laboratory USA [54–57] and far-field 3D X-ray diffraction (3DXRD), which was first developed at Risø DTU in Denmark [58–62].

X-ray diffraction contrast tomography (DCT) [63,64] is a variant of the 3DXRD microscopy technique enabling simultaneous reconstruction of the 3D microstructure (shape and orientation) in suitable polycrystalline materials, along with the absorption map of the specimen. The X-ray DCT methodology provides access to the 3D shape, orientation and elastic strain state of the individual grains from polycrystalline materials fulfilling some conditions in terms of grain size and intergranular orientation spread.

In the present work the onset of twinning is studied in a binary Ti–4Al alloy, using two different diffraction techniques, neutron diffraction and X-ray DCT. Samples were first compressed and the activation of $\{10\bar{1}2\}\ \langle\bar{1}011\rangle$ tensile twins was followed in-situ by means of neutron diffraction.

Subsequently, small samples were extracted from the deformed samples (about 0.04% of the initial sample volume) at selected applied strains, and characterised using the X-ray DCT methodology to reveal the 3D grain structure, allowing a grain-by-grain study of the shape and location of twins. A statistical analysis was carried out in which the parent grains of twins where grouped together and compared with similarly orientated grains that had not twinned. Comparisons were carried out regarding grain size, the general Schmid factor *m* (based on an uniaxial stress) [65] and slip transfer parameter *m’* [42,66–68] across the grain boundaries. Finally, a search for possible clustering of twins was carried out and cluster neighbourhoods were again analysed.

2. Experimental procedure

2.1. Material preparation

For the purpose of this research, 200 g binary Ti–4Al (i.e. 4wt.%) alloy buttons were double melted in a tungsten arc furnace under inert gas atmosphere. Each button was beta forged at 1100 °C at the TIMET research facility in Witton, UK. The measured chemical composition of the alloy is given in Table 1. Subsequently, the buttons were cross-rolled in bar shape (14 × 14 × 260 mm) on a “2 high Robertson mill” (WHA Robertson & Co Ltd) at 870 °C followed by a recrystallization (RX) heat treatment at 993 °C (30 °C below the beta transus temperature) in a tube furnace under Argon shielding for 5 h followed by air-cooling. The lattice parameters and the c/a ratio were determined at the neutron spallation source ISIS, Chilton, UK. They are $\langle a\rangle = 2.935$ Å and $\langle c\rangle = 4.678$ Å giving a c/a ratio of around 1.5938 [69]. The average grain size of the studied samples is 73 μm, which was measured by using the linear intercept method [69]. The {0002} pole figure and the initial microstructure of the Ti–4Al raw material used in this analysis is shown in Fig. 1.

Three samples with a diameter of 5 mm and length of 12.35 mm each were cut by electro-discharge machining (EDM) with the cylinder axis parallel to the original rolling direction (RD). The texture of the material is such that the $\langle c\rangle$ axes of the grains tend to be oriented perpendicular to the cylinder axis, which promotes tensile twinning during compression loading as the $\langle c\rangle$ axis is strained in tension.

2.2. In-situ loading using neutron diffraction

In the first part of the experiment, each sample was placed in a stress-rig based on the Strain Analyser for Large and Small scale engineering Applications (SALSA) beam line at the Institut Laue-Langevin (ILL) in Grenoble, France [70,71]. It uses a thermal neutron beam with wavelength $\lambda = 1.62$ Å corresponding to a two-theta angle of 40.5° for the {0002} reflection for Ti–Al alloy, which was measured in the loading direction. The two-dimensional position sensitive micro-strip detector has an active area of $80 \times 80\ \mathrm{mm}^2$ with 256 × 256 channels and was positioned in a way that the scattering vector q of the {0002} reflection was parallel to the loading direction. The angle covered by each channel is 0.02° at a sample-detector distance of 1 m. The 2D data were integrated to produce a one-dimensional diffraction peak profile covering 5°, in which the {0002} and $\{10\bar{1}1\}$ peaks are visible, and fitted using a Gaussian function. Because of the poor neutron scattering properties of Ti the counting time per load step was 30 min in order to obtain a fittable {0002} reflection. As the main concern was the