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In situ single-grain peak profile measurements on Ti–7Al during tensile deformation

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ABSTRACT

High-energy three-dimensional X-ray diffraction with medium and high reciprocal space resolution was applied to study *in situ* tensile deformation of Ti–7Al specimens. Samples with planar and random dislocation microstructures were prepared and characterized by electron microscopy. Stress tensors of individual grains were obtained at several loads up to 2% deformation. The stress tensors were found to rotate, and resolved shear stresses were calculated. High-resolution reciprocal space maps of selected grains were recorded. Azimuthal and radial distributions were visualized and discussed in terms of idealized dislocation structures. Heterogeneous grain rotations were observed for the planar microstructure and found to be consistent with activation of the highest stressed basal slip system. Intra-granular strain gradients were detected in excess of the intrinsic radial dislocation peak broadening. The potential of combining the applied techniques with modeling to obtain multiple length-scale information during deformation of bulk specimens is discussed.

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1. Introduction

Synchrotron X-rays have enabled the creation of new generations of diffraction-based materials characterization experiments for polycrystalline materials. The importance of processing phenomena – such as microplasticity, recrystallization, and microcrack initiation – have been understood for decades. Individual grains set a natural length-scale within polycrystalline materials, but it is also understood that the dislocation structure formation within grains and grain–grain interactions may play an important role. Laue micro-diffraction techniques have been developed that provide sub-micron three-dimensional spatial resolution but the point-by-point data acquisition is comparatively slow [1]. The technique has mostly been applied on the grain and subgrain length-scale [2]. The three-dimensional X-ray diffraction (3DXRD) technique is based on "tomographic" data acquisition using high-energy X-rays and area detectors and therefore enables fast data acquisition at lower spatial resolution [3,4]. Furthermore, the interior of several mm-thick samples can be probed. Recently, high reciprocal space resolution 3DXRD has been developed and applied to characterize subgrain formation in copper during tensile deformation [5]. This work aims at evaluating the potential of combining conventional and high-resolution 3DXRD with electron microscopy in order to characterize the plastic deformation of a hexagonal close packed titanium alloy at multiple length-scales.

Titanium alloys are extensively utilized in a wide range of high-performance components requiring materials of high strengths and low densities. The typical slip systems in the HCP phase are the basal, $(0\,0\,0\,1)\ \langle 1\,1\,\bar{2}\,0\rangle$, and prismatic, $\{0\,1\,\bar{1}\,0\}\ \langle\bar{2}\,1\,1\,0\rangle$. These are $\langle\vec{a}\rangle$-type slip systems – they can accommodate no slip along the *c*-axis. Commercial materials, particularly those having a majority volume fraction of the alpha (HCP) phase, are typically alloyed with significant concentrations of aluminum (4–8 wt%). These additions, intended to increase strength and modulus, both suppress deformation twinning and promote persistent, highly localized $\langle\vec{a}\rangle$, $\vec{b} = \langle 1\,1\,\bar{2}\,0\rangle$ dislocation slip on basal, $(0\,0\,0\,1)$, prism, $\{1\,\bar{1}\,0\,0\}$, and first order pyramidal, $\{1\,\bar{1}\,0\,1\}$, planes [6–9]. Transmission electron microscopy (TEM) studies of deformation microstructures in titanium alloys typically show densely packed and highly planar dislocation arrays separated by regions of undeformed material [6,10]. The tendency for this type of slip behavior has been shown to

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Table 1
Composition of the Ti–7Al material.

Al (wt%)	Fe (wt%)	O (wtppm)	N (wtppm)	C (wtppm)	H (wtppm)	Ti (wtppm)
7.10	0.015	1100	100	100	30	Balance

be dependent on the degree of short-range ordering (SRO) present in the microstructure [6]. Small regions of SRO (on the order of tens of Angstroms [11,12]), that may be thought of as the precursors to fully ordered Ti_3Al, act in a manner similar to shearable precipitates in that they localize slip both by progressively softening a slip plane with the passage of dislocations and by inhibiting large-scale out-of-plane dislocation cross-slip.

This paper demonstrates the potential of combining results using novel X-ray interrogation capabilities with more traditional methods like TEM. We chose Ti–7Al as an illustrative material for several reasons. First, its large strength to stiffness ratio, which makes it ideal for the lattice strain experiments. Secondly, Ti–7Al can be processed differently to produce materials with very different properties depending on the existence of SRO domains. Our investigation interrogates specimens in both states to examine underlying differences that may be apparent in the micromechanical behavior of each. In the following sections we lay out the experimental methods and present results for selected reference grains within each type of sample. We focus on two types of *in situ* measurements during tensile deformation. In the first, we measure lattice strains and determine crystal-scale stress states. Using a different detector we then interrogate a single reflection with high reciprocal space resolution to understand grain subdivision via the formation of dislocation structures. In addition to the X-ray diffraction experiments, we employ electron microscopy to examine identical specimens in order to understand the microstructure.

2. Experimental procedure

2.1. Material and samples

The titanium alloy examined in this study was provided in the form of an extrusion approximately 3 cm in diameter and 75 cm in length; processing details are reported elsewhere [13]. The material composition is listed in Table 1.

Once formed, the extrusion was cut into smaller coupons, which were vacuum encapsulated and annealed high in the α-phase field, at 1235 K, for 24 h producing an equiaxed grain structure with an average line intercept grain size of ~75 μm.

The orientation distribution (OD) was constructed from previously published scanning electron microscope (SEM) based electron back-scattered diffraction (EBSD) texture measurements [13]. The annealed materials displayed a weak texture with a texture index of about 3 (mean square value of the OD normalized to "multiples of a uniform distribution").

The SRO state was modified by cooling rate from the annealing temperature. All specimens were allowed to naturally cool from the annealing temperature producing the air-cooled (AC) condition with strong SRO. Selected specimens were subsequently solutionized at ~1200 K for 10 min in an environment of flowing argon gas and directly quenched into ice water. This treatment, designated IWQ, had no influence on material structure other than to suppress the formation of SRO. Transmission electron microscopy studies [13] have shown no evidence of SRO in the IWQ specimens; a relatively strong SRO state, observed as diffuse superlattice reflections in diffraction patterns, was found in the AC specimens.

Flat, hourglass-shaped specimens of 1 mm thickness and a gage section of 2.5 mm × 12 mm were cut by electrical discharge machining from both the AC and IWQ materials such that the deformation axis was parallel to the extrusion direction of the bar.

2.2. Electron microscopy

The dislocation structures produced by the plastic deformation IWQ and AC materials were observed both by conventional bright-field transmission electron microscopy (BFTEM) and SEM EBSD. Thin foils, prepared by traditional electro-chemical polishing methods reported elsewhere [13], were extracted from the plane normal to the loading axis. Foils were imaged in a LaB_6 Philips CM-30 TEM operated at 300 keV with first-order pyramidal, $\vec{g} = \{1\,\bar{1}\,0\,1\}$, reflections under two-beam diffraction conditions; dislocation line orientation and Burgers vector were determined by tilt experiments. As presented in Figs. 1 and 2, the two materials display markedly different distributions of $\langle \vec{a} \rangle$ dislocations. While the dislocations in the IWQ material are homogenously distributed throughout the microstructure, dislocations in the AC material are tightly arranged in planar arrays. Close inspection of the AC microstructure reveals variation in the background intensity from one side of a slip band to the other. These variations are the result of slight deviations in Bragg condition.

Post-mortem SEM EBSD measurements were performed on regions near the center of the gage sections of each of the tensile bars; specimens were carefully prepared (see [13] for details) such that only internally situated grains were observed in a plane parallel to the loading axis and normal to the broad face of the tensile flat. Automated scans were performed in a Philips FEG XL-30

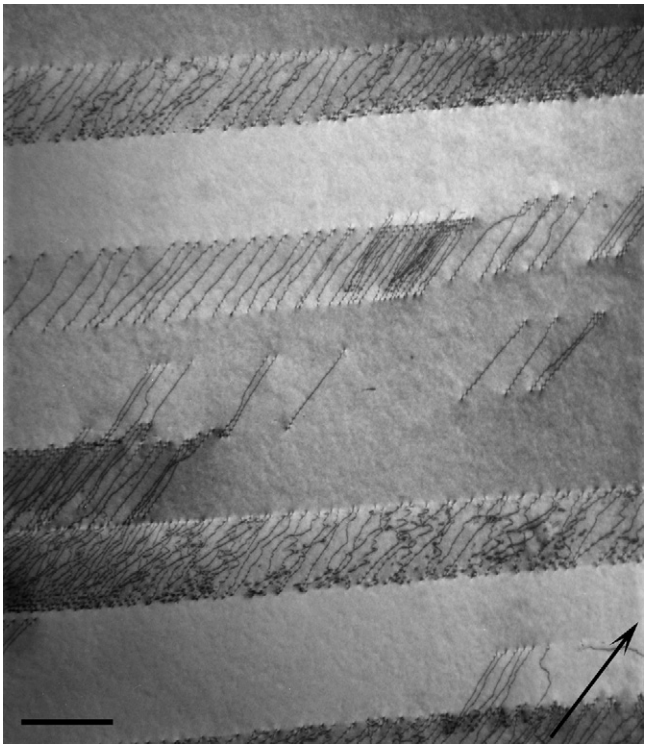

Fig. 1. Dislocation arrangements observed in the AC material. The presence of SRO leads to highly planar arrays of $\langle \vec{a} \rangle$ screw dislocations. For reference, the arrow indicates the Burgers vector, and a 1 μm scale bar is shown.