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In-situ investigations of structural changes during cyclic loading of aluminium by high resolution reciprocal space mapping

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ABSTRACT

A major failure reason for structural materials is fatigue-related damage due to repeatedly changing mechanical loads. During cyclic loading dislocations self-organize into characteristic ordered structures, which play a decisive role for the materials lifetime. These heterogeneous dislocation structures are identified by high resolution reciprocal space mapping. The synchrotron technique using high energy X-rays was applied successfully in-situ during cyclic deformation of macroscopic aluminium samples at the Advanced Photon Source to reveal the structural reorganization within single grains embedded in the bulk material during cyclic deformation. As evident from the changes in the radial profiles of four grains, the adaption of the deformation structure to cyclic deformation is completed already after 800 cycles. Individual subgrains have been followed through a 7350 tension–tension cycles while monitoring macroscopic stress and strain during cyclic loading. The elastic back strains of subgrains are Gaussian distributed with larger subgrains showing larger back strains. The detailed characterization of the microstructure during cyclic loading by in-situ monitoring of the internal structure within individual grains facilitates the understanding of materials behaviour during cyclic deformation.

1. Introduction

The majority of metallic components fail as a consequence of periodically varying stresses causing structural changes in the material, which result in cracks and fracture after a sufficient number of cycles. During mechanical loading of metals, plastic deformation occurs on the microscale by motion of dislocations causing a fraction of dislocations to be stored in the material. Characteristic low-energy dislocation structures develop during cyclic deformation in face-centered cubic metals and consist of dislocation-rich walls and dislocation-free subgrains [12]. These structures have been extensively studied in copper, while the corresponding microstructural changes in aluminium are less frequently reported. Grosskreutz et al. [6] and later Madhoun et al. [3] analysed the reorganization of dislocations in aluminium into 1 μm to 5 μm large cells during cycling deformation by means of transmission electron microscopy. Dislocation walls evolving during cyclic deformation of polycrystals subdivide the original grains into numerous subgrains, i.e. dislocation-free regions of slightly different orientations, as shown for aluminium in Fig. 1, where the different orientations are revealed by different intensity. The details of the progressing self-organization of dislocations into dislocation walls separating subgrains, however, remain unknown, because it is not possible to study the substructure of grains in the bulk of a relevant sized polycrystal during ongoing deformation by means of electron microscopy. Such information can be provided by sophisticateded synchrotron technique while other techniques such as electron microscopy or conventional X-ray diffraction are either restricted to surface near regions, destructive or obtain solely an average over a number of grains with various orientations.

High Resolution Reciprocal Space Mapping (HRRSM) [8,9] enables to follow the microstructure of individual grains embedded within a polycrystalline bulk sample in-situ during deformation by obtaining three-dimensional reciprocal space maps with high resolution ($\Delta q/q = 10^{-4}$ for the diffraction vector q). Utilizing a custom-made load frame, the evolution of the subgrains and the associated internal stresses in individual grains of commercially pure, polycrystalline aluminium can be monitored in-situ during cyclic deformation. In this manner, the evolution of deformation structure can be related in an unprecedented way to the mechanical loading experienced by the sample, for instance, during unloading and reversed loading [1] or after an increasing number of load cycles [2]. In the present study, four grains of similar orientation (having a crystallographic ⟨1 0 0⟩ direction

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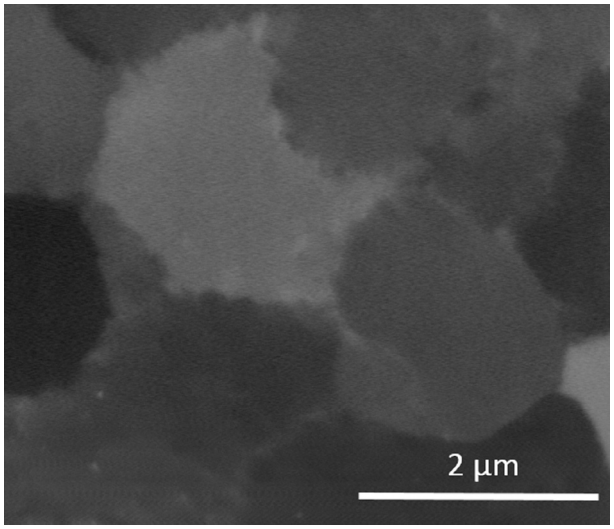

Fig. 1. Deformation structure within an individual grain in polycrystalline aluminium (AA1050) after cyclic deformation in tension–tension for 40,500 cycles with a nominal strain amplitude $\hat{\varepsilon}_{nom}$ of $6.7 \cdot 10^{-4}$. The electron channelling contrast image reveals subgrains of about 2 µm size by their orientation difference originating from excess dislocation in the dislocation walls.

along the loading axis) are followed through an increasing number of tension–tension load cycles. In each of the grains, at least 80 subgrains are identified from their corresponding high-intensity peaks and their evolution during cyclic deformation is analysed.

2. Experimental investigation

2.1. Material

Tensile test specimens were manufactured from an AA1050 sheet homogeneously cold-rolled to 90% thickness reduction to a final thickness of 1 mm. Dog bone-shaped specimens with a gauge section of 15 mm in length and 5 mm in width were designed to fit to a custom-made screw-driven load frame. Sample cutting was done by spark cutting and tensile specimens were then annealed at 600 °C for 2 h to ensure complete and homogeneous recrystallization. The microstructure after annealing was investigated metallographically. Using both, light optical microscopy and scanning electron microscopy, grain sizes were estimated to be between 30 µm and 100 µm, and the microstructure was found to be homogeneous throughout the entire cross section of the gauge showing a pronounced cube texture. Tensile testing of such recrystallized specimens with a nominal strain rate of $10^{-3}\,s^{-1}$ revealed their yield strength being 16 MPa.

2.2. Pre-deformation

Prior to the in-situ investigation by HRRSM, cyclic pre-deformation was carried out in order to introduce a microstructure conform to cyclic deformation in the specimen using an MTS Acumen 3 kN Electrodynamic Test System equipped with Station Manager MTS FlexTest 40 and pneumatic grips. The investigated sample was initially deformed by 1% in tension with a grip speed of 0.015 mm/s and then cycled at a rate of 0.5 Hz under displacement control with a displacement amplitude of 10 µm corresponding to a nominal engineering strain amplitude $\hat{\varepsilon}_{nom}$ of $6.7 \cdot 10^{-4}$. 18,000 tension–tension cycles were performed by repeatedly decreasing the displacement by 20 µm from the maximal displacement of 150 µm achieved after tensile deformation to 1% and increasing it again to 150 µm. During the cyclic deformation, a stress ratio $R = \sigma_{min}/\sigma_{max}$ between the stresses at minimum and maximum load of 0.65 was observed.

2.3. Experimental set-up at synchrotron facility

For the synchrotron investigations, the sample was equipped with a pre-wired strain gauge Omega KFG-3 350 Ω at the center of the gauge section and aligned with the tension axis to monitor the axial strain in-situ. The sample is mounted in a custom-made screw-driven load frame equipped with a 5 kN load cell as presented in Fig. 2a. Using flat grips, this load frame allows for mechanical loading in tension and compression while monitoring the local microstructure within the tensile specimen in-situ using synchrotron radiation. The load frame was placed with the load axis horizontally on a *xy*-translation stage allowing to move the sample and with this the selected grain of interest – which becomes displaced with respect to the load frame during mechanical loading – to the center of the beam after each loading step. The *xy*-translation stage is mounted on top of a rotation stage allowing rotation of the entire load frame around the vertical *z*-axis to obtain reciprocal space maps by rocking in small intervals around this axis. An additional *z*-translation stage allows adjustment for possible changes in the height of the selected grain of interest due to mechanical loading.

High Resolution Reciprocal Space Mapping was carried out at beam line 1-ID-E at the Advanced Photon Source at Argonne National Laboratory with a monochromatic beam of 52 keV while loading the pre-fatigued sample with position control. A sketch of the experimental set-up is shown in Fig. 2b.

First, suitable grains are identified with the help of a large area detector, an amorphous silicon flat panel from General Electrics, (detector 1) placed 86 cm behind the sample on a horizontal translation to cover the first 6 diffraction rings of aluminium. Individual grains are

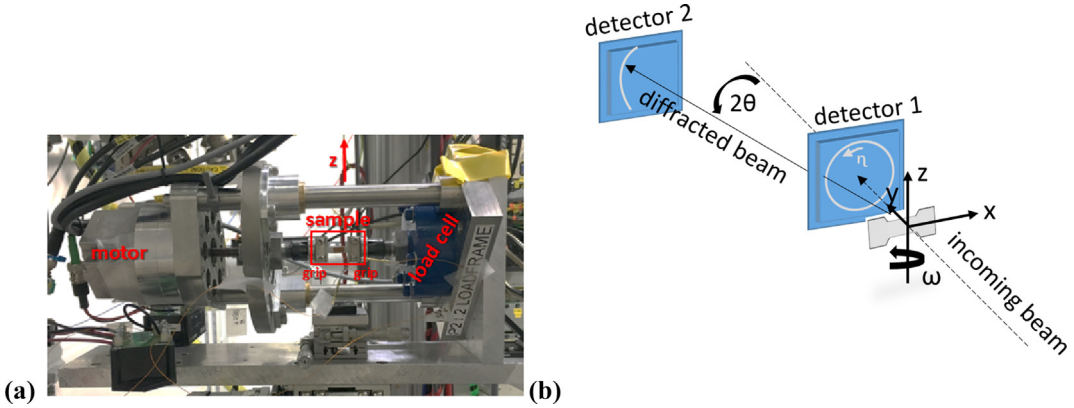

Fig. 2. (a) Load frame used for High Resolution Reciprocal Space Mapping in-situ during mechanical loading at APS, 1-ID-E. The sample equipped with a strain gauge (highlighted with a red rectangle) is positioned in the center of rotation on top of several translation and rotation stages for alignment und acquisition. (b) Sketch of the diffraction geometry and the position of the detectors used at APS, 1-ID-E.

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