In-situ study of the effect of strain path change on dislocation boundary evolution in commercial purity aluminum

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Abstract. An in-situ study of the effect of strain-path change on deformation microstructure and slip activity has been investigated using samples of commercially pure aluminum. Changes in dislocation boundary arrangements and crystal lattice rotations were tracked using CMOS-based electron backscattered diffraction (EBSD) detector combined with in-situ tensile deformation of ε = 10%. Samples were first cold-rolled to 5% reduction and then deformed by uniaxial tensile along the transverse direction. Despite the use of a CMOS-based detector, analysis of deformation microstructure at such low plastic strains using EBSD is still challenging. Nevertheless in some grains details of the dominant deformation microstructure can be discerned. Slip line observations confirm that overall slip system activity is not influenced by the strain path change, but the EBSD observations show that in some cases dislocation boundary alignment is influenced by the presence of pre-existing boundaries, and in some grains the crystal rotations differ to those expected for tensile deformation of fully recrystallized grains of similar orientation.

1. Introduction

Over the past few decades extensive systematic studies have been carried out to characterize the microstructure evolution in medium-to-high stacking fault energy single-phase face-centered cubic (fcc) metals, such as Al, Cu and Ni [1-10]. Based on these studies it has been established that after deformation the stored dislocations are organized into dislocation rotation boundaries [1-3]. Two categories of dislocation boundaries have been identified, namely geometrically necessary boundaries (GNBs) and incidental dislocation boundaries (IDBs) [2, 3]. Investigations of dislocation structures formed during monotonic deformation (e.g. tension, compression or rolling) have demonstrated that for both the GNBs and IDBs the average boundary spacing decreases and the average misorientation angle across the boundaries increases with increasing strain [4-6]. Additionally it has been found that the dislocation boundary morphology strongly depends on the crystallographic grain orientation, with this relationship extending down in some cases to grain sizes of less than 1 μm [7, 9].

These studies show a clear correlation between dislocation boundary morphology and final grain orientation. However, during manufacturing processes of metal components, the plastic deformation process is often complex including changes of strain path. In contrast to the clearly findings regarding dislocation structure evolution under monotonic deformation, many aspects of the evolution of dislocation structures under strain path change deformation are still unclear [11-14]. For example, Sellars et al. [12] determined GNBs inclination angles in tension followed by compression for a Al-3%
Mg alloy and concluded that the compression step did not change the inclination angle of the GNBs formed in tension, but the misorientation angle across these GNBs decreased, and a new set of GNBs with different angle was formed. Pedrosa et al. [13] examined forward and reverse torsion (using an equal strain value of \( \varepsilon = 0.23 \) for each direction) on AA1200 Al, and concluded that the reverse strain led to a reduction in the misorientation angle of GNBs formed during the forward deformation, followed by the generation of new GNBs with a lower average misorientation compared with those formed in monotonic torsion to the same strain. Lewandowska et al. [14] studied the influence of both forward/reverse shear and combined rolling-tension-shear strain path changes on the dislocation structures formed in Al 5128 and Al 6016 alloys, observing that reversal of the shear direction partly destroyed the pre-existing dislocation boundary arrangements, while orthogonal deformation promoted an increased dislocation density between the pre-existing GNBs.

A major reason for limited understanding of the microstructural changes that take place during strain path changes is the difficulty of making in-situ observations to follow directly dislocation boundary rearrangements. Examination of thin foils in a transmission electron microscope (TEM) technique is the most reliable and standard method for characterization of dislocation boundary arrangements in deformed metals in metals [15]. This technique only allows, however, in-situ deformation of very small-size samples, with a limited range of both strain and deformation mode. Recently new techniques have been developed for measurement and analysis of crystal orientations using electron backscatter diffraction (EBSD) with improved angular resolution, including a high-sensitivity CMOS-based detector, offering the potential for study of strain path changes on bulk samples deformed in-situ in a scanning electron microscope. The objective therefore of the present work is to explore the possibility of EBSD for the study of the effect of strain path change on dislocation boundary evolution, using as a test material commercial purity Al.

2. Experimental

The material used in this study is a commercial purity Al (AA1200) sheet of thickness 1.2 mm, annealed to a fully recrystallized condition with an average grain size of 35 \( \mu \)m. The Al sheet was first cold rolled to 5% reduction and then dog-bone shaped tensile specimens (gauge width, length and thickness of 10mm, 2mm and 1mm, respectively) were cut from the rolled sheet, with the tension direction taken parallel to the transverse direction of the rolled sheet.

Prior to in-situ tensile deformation, the top surface of the tensile specimen was ground to 4000-grit SiC and then electropolished using 10% HClO\(_4\) in ethanol. The in-situ tensile deformation was carried out using a push-to-pull tensile stage at a strain rate of approximately \( 5 \times 10^{-3} \) s\(^{-1}\). Before and after the tensile deformation (to a strain of 10%), the microstructure in a selected region on the surface was characterized using EBSD. For the EBSD measurements an Oxford Instruments high sensitivity CMOS-detector was used, with data collected in high-resolution mode. For both the as-rolled and rolled plus tension samples an EBSD step size of 1 \( \mu \)m was used.

3. Results and discussion

Figure 1 shows the EBSD results of the same region in the sample rolled to 5% (figure 1(a-c)) and after additional tension deformation of 10% (figure 1(d-f)). The inverse pole figure (IPF) coloring in figure 1(a,d) shows the crystal direction parallel to the extension direction during rolling (i.e., the rolling direction, RD), whereas figure 1(b,e) shows the crystal direction parallel to the tension axis (TA) during in-situ deformation. By comparing the IPF maps it can be seen that, aside from a few grains with orientation close to the cube-orientation (some examples are highlighted with a white ellipse), for most grains the crystal direction parallel to the extension axis is different for the tension step compared to the initial rolling. For example some grains with <001> nearly parallel to RD but with the TA close to either <110> or <111> are marked with a black ellipse. Accordingly the orientation relationship of most grains with respect to the elongation direction changes during the experiment. Figure 1(c,f) also show kernel average misorientation (KAM) maps (constructed using a 3 \( \times \) 3 kernel) in which some details of the dislocation boundary microstructure can be seen, even after the low strains used in the present
experiment. Comparison of the IPF maps before and after 10% tensile deformation reveals an increase in the orientation variation inside most grains. A similar comparison of the KAM maps reveals some clear changes in the deformation microstructure, including an increase in the misorientation angle of some boundaries, and the appearance of many new dislocation boundaries.

**Figure 1.** EBSD observations of a selected region in (a-c) the pre-rolled specimen and (d-f) in the same region after 10% tensile deformation (along the transverse direction, TD): (a, d) and (b, e) are inverse pole figure maps corresponding to the rolling and tensile directions, respectively; (c, f) are kernel average misorientation maps constructed using a $3 \times 3$ kernel, with an upper cut-off of 5°.

**Figure 2.** (a) Low magnification forescatter detector (FSD) images of slip bands in the examined region after 10% tension deformation; (b-e) high magnification FSD images together with slip system traces and for selected grains colored based on the Schmid factor (SF) ranking.
It has been reported that slip system activation is a dominant factor in determining the dislocation boundary morphology [16]. In the present experiment the slip traces resulting from the in-situ tensile deformation have been examined from images collected using a forescatter electron detector (FSD). Some examples are shown in figure 2, where it can be seen that slip traces are clearly visible, and well-defined in most of the grains in the examined area. The low magnification FSD image (figure 2a) reveals that one or two sets of slip bands are observed in each grain, with most slip bands straight and only a small fraction showing a curved or wavy appearance. It is interesting to note also that many slip bands are observed to cross the grain boundaries, in agreement with previous studies [17, 18]. Based on the measured orientation of each grain a slip trace analysis has also been carried out where it is found that in each grain the observed slip traces correspond to slip systems with highest (or second highest in the case of grains where two sets of slip traces are visible) Schmid factor [19]. Some examples are shown in figure 2(b-e). Note that the labelling of the {111} planes is arbitrary (depending on the specific crystal symmetry variant reported in the EBSD data) used, but the coloring indicates whether the trace corresponds the highest (red), second highest (blue), or other (yellow) slip system.

It has recently been shown that maps plotting the rotation axis in the specimen coordinate frame of each pixel to the grain average orientation can be useful for highlighting both grain subdivision, and in some cases dislocation boundary arrangements, even in samples deformed only to small plastic strains [20]. Example grain average deviation (GAD) maps are shown for two selected grains in figure 3(a,b), where for comparison KAM maps of the same grains are also shown. The GAD (a1) and KAM (a3) maps of grain labelled G28 reveal the presence of a few dislocation boundaries with relatively high misorientation (marked with red boxes in the figure) after 5% rolling. The GAD (a2) and KAM (a4) maps for the sample after additional tension deformation show these boundaries remain with slightly increased KAM values, indicating additional dislocation storage at these boundaries after the strain path change. In addition the presence of two sets of GNBs formed during tension is seen in both the GAD and KAM maps. This grain has an average tensile axis 6° from [221], for which it is expected that tensile deformation should result in GNBs forming close to the (441) plane [16]. The closest set to this trace (marked with a red dashed line in figure 3(a2)) for this grain is approx. 16° from this trace.

![Figure 3](image)

**Figure 3.** Grain average deviation (GAD) and KAM maps of two typical grains before and after tensile deformation: GAD map of G28 (a1) before tension and (a2) after tension, KAM map of G28 (a3) before tension and (a4) after tension, GAD map of G33 (b1) before tension and (b2) after tension, KAM map of G33 (b3) before tension and (b4) after tension.

Fig. 3(b) shows the microstructure before and after tensile deformation for a grain (G33) deviated by 14° from {001}<100>. The GAD (b1) and KAM (b3) maps for this grain before tension (after 5% rolling) show a weak cell-structure with one clearly discernable dislocation boundary traversing the grain (marked with a red box). After additional tension deformation the major boundary is partly preserved with higher local misorientation (KAM value). Additionally, a new set of GNBs (highlighted...
with a red dashed line in b2) is observed, lying along almost parallel to the (T 11) slip trace. For this grain, with the tensile axis just 4° from <001>, it is expected, however, [7] that tension deformation should lead to the formation of a dislocation cell structure, rather than the formation of GNBs.

On account of the in-situ deformation, another aspect of the deformation behavior that can be probed is the lattice rotation of each grain during tension deformation. These rotations can be compared to the rotations both predicted by theory and measured in bulk grains using X-ray synchrotron diffraction [19] for tension deformation of initially recrystallized grains (i.e. without any strain path change). Some examples are shown in figure 4, which also shows the expected rotation paths for tension as a function of the tensile loading axis (figure 4a). It can be seen that for the grains plotted in figure 4(b,d) the rotation paths (as determined from the grain average orientation before and after tension) differ significantly from the expected paths, though some of this difference may be attributed to the difference between the behavior of surface and bulk grains. For grain G28 (figure 4d) a more complex behavior is seen, where part grain rotates along the predicted path (black arrow) while another part of grain rotates along an approximately opposite path (red arrow).

The results presented above show that optimized EBSD observations using a high sensitivity detector are able to at least reveal some features of the changes in deformation microstructure during strain path change, even at the low plastic strains used in this study. One limitation, however, of the present work is that with the current in-situ tension rig, observations have to be made on the rolling-plane of the as-rolled sample, whereas it is known that the deformation microstructure after rolling can be resolved much more easily in the longitudinal plane (formed by the rolling and normal direction). To illustrate the potential for using EBSD to characterize dislocation boundaries in the RD-ND plane at low strain, a map from the same sample (rolled to 5% reduction) in this plane is shown in figure 5. It is seen that at least in some grains a number of dislocation walls are clearly revealed and the microstructure is easier to characterize than in the RD-TD plane. It remains a challenge, however, to design an experiment where in-situ deformation can be carried out allowing viewing of this sample plane.

**Figure 4.** Evolution of crystal direction parallel to the tensile axis during tensile deformation: (a) previously obtained results for bulk recrystallized grains using X-ray synchrotron radiation (image adapted from [19]); (b-d) experimental results for three selected grains. Black pixels represent the initial tensile axis and grey pixels represent the tensile axis after 10% tensile deformation. Red arrows indicate rotations that differ significantly from previous experimental data on fully recrystallized samples.

**4. Conclusions**

A study has been carried out to assess the potential of using EBSD for in-situ studies of the effects of strain path change on the evolution of the deformation microstructure. The following conclusions from this work can be made:

- Visualization using EBSD of dislocation boundaries in metals deformed to low plastic strain remains a challenge, and a full characterization of the deformation microstructure is still only possible using transmission electron microscopy. Nevertheless in some grains details of the dominant deformation microstructure can be discerned, and EBSD investigation both allows in-situ experiments to be performed and provides reliable data on grain rotations.
During an orthogonal strain path change from rolling to tension, where the elongation direction is rotated by 90°, some dislocation boundaries formed during the initial 5% rolling remain after 10% tension, with increased local misorientation. Slip line observations after tension deformation suggest that the overall slip system activity is not influenced by the strain path change, but EBSD observations show that in some cases dislocation boundary alignment is influenced by the presence of pre-existing boundaries.

In-situ deformation allows crystal rotations of surface grains to be tracked and reveals some significant differences to predictions based on tensile deformation of fully recrystallized grains.

Figure 5. EBSD observations of the longitudinal (RD-ND) plane of a sample rolled to a reduction of 5%: (a) kernel average misorientation (KAM) using a 3 x 3 kernel; (b) grain average deviation map plotting for each grain the axis component in the sample coordinate frame of the misorientation between each pixel and the grain average orientation.

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