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Structural coarsening during annealing of an aluminum plate heavily deformed using ECAE

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Abstract. The microstructure and softening behaviour have been investigated in an aluminum plate heavily deformed by equal channel angular extrusion and subsequently annealed at 170 °C. It is found that at this temperature the microstructure evolves by coarsening with no apparent signs of recrystallization even after 2 h of annealing. Both coarsening and softening are rapid within first 10 minutes of annealing followed by a slower evolution with increasing annealing duration. Evidence of triple junction (TJ) motion during coarsening is obtained by inspecting the microstructure in one region using the electron backscatter diffraction technique both before and after annealing for 10 minutes. The fraction of fast-migrating TJs is found to strongly depend of the type of boundaries composing a junction. The greatest fraction of fast-migrating TJs is in the group, where all boundaries forming a junction are high angle boundaries. The fraction of fast-migrating TJs is significantly lower for junctions containing at least one low angle boundary.

1. Introduction

It has been reported that annealing of materials cold rolled to high plastic strains can result in significant structural coarsening during recovery [1–5], which can occur by migration of both deformation-induced boundaries and triple junctions (TJs). It is considered that the process of coarsening during recovery of heavily deformed materials is facilitated by the presence of a large fraction of high angle boundaries (HABs) produced by deformation. As the fraction of HABs in a deformed microstructure increases with increasing plastic strain [6], it is expected that in a material deformed to a very high strain, the fraction of TJs containing HABs should also be high.

Equal channel angular extrusion (ECAE) is a well-known deformation technique which enables very high strains by multiple passes through a die with two intersecting channels, and which has been widely used for producing heavily deformed samples of rod, bar and plate shapes [6–11]. In our previous work [12], ECAE was applied for severe plastic deformation of 15 mm thick plates of commercial purity aluminum. By comparing the microstructure in the center of two plates processed either with or without sequential 90° rotations about the plate normal between passes, it was found that deformation with 90° rotations resulted in a fairly homogeneous microstructure with a high fraction of HABs, 70% as measured using the electron backscatter diffraction technique (EBSD) [12]. The evolution of this ECAE-processed microstructure during recovery annealing is investigated in the present work, and the results obtained are compared with those reported previously for the heavily-rolled material [1–4].

2. Experimental

An AA1050 aluminum plate with dimensions of 15 × 75 × 75 mm^3 was extruded at room temperature using an ECAE die with a sliding floor, a sharp outer corner and a 90° angle between channels (see [12] for more details). For this die configuration the strain per pass was 1.15, and the plate was deformed by 8 ECAE passes with 90° sequential rotations between passes. The deformed material was then annealed at 170 °C and samples were taken after 10 minutes, 30 minutes, 60 minutes and 2 h of annealing.

Vickers hardness measurements and EBSD analysis of these samples were conducted near the middle plane in the polished longitudinal section which contained the extrusion direction and the plate normal. The hardness was measured applying a load of 200 g and a dwell time of 15 s. A step size of 70 nm was used for statistical EBSD analysis of the average subgrain size and the HAB fraction in each deformed and annealed condition. In addition, one region with an area of 180 μm^2 was investigated with a step size of 25 nm both in the deformed condition and after 10 minutes of annealing at 170 °C without re-polishing. Because of the limited angular resolution of the EBSD technique [13,14], misorientations less than 2° were ignored in the EBSD data. Therefore, low angle boundaries (LABs) were defined as boundaries with a misorientation angle θ = 2–15°. Boundaries with θ > 15° were defined as HABs.

3. Results

The deformed microstructure obtained after ECAE is shown in figure 1a. The average subgrain size *d* in this microstructure is ~0.5 µm and the fraction of HABs is 70%. The subgrains are mostly elongated although some fairly equiaxed subgrains are also present in the microstructure. The average aspect ratio in this as-deformed condition is almost 2, and the Vickers hardness is 55 HV0.2. During annealing at 170 °C comparatively rapid coarsening to d = 0.63 µm takes place within the first 10 minutes followed by a slower increase in the average subgrain size (figure 1 and figure 2a). However, the average subgrain size remains in the submicrocrystalline range, ~0.8 µm, even after 2 h of annealing.

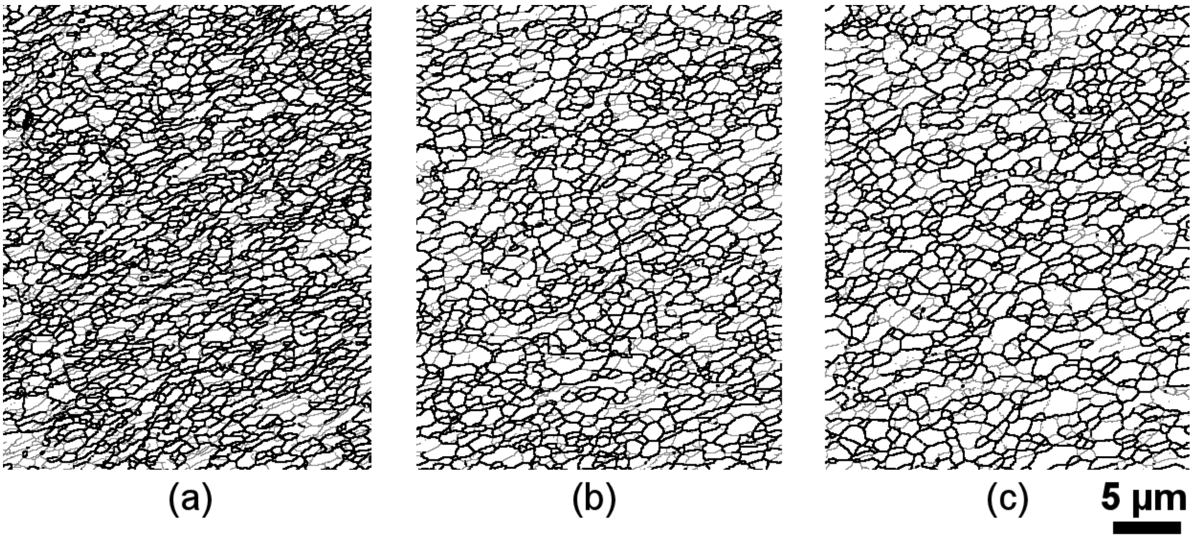

Figure 1. EBSD maps representing the microstructure of aluminum after ECAE (a) and subsequent annealing at 170 °C for 10 minutes (b) and 2 h (c). Gray and black lines show LABs and HABs, respectively. The extrusion direction is parallel to the scale bar.

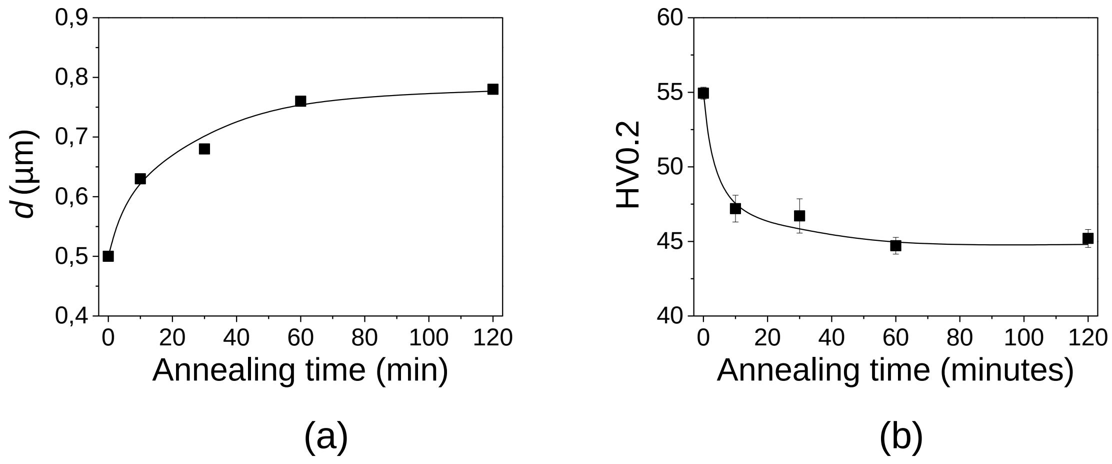

Figure 2. The effect of annealing at 170 °C on the average subgrain size (a) and Vickers hardness (b) in the ECAE-processed aluminum plate.

It is apparent that in each annealed condition the microstructure is morphologically similar to the deformed microstructure. Compared to the as-deformed material, the average aspect ratio of the subgrains decreases only by 10% (to ~1.8) after 2 h of annealing. The variation in the fraction of HABs during annealing is also very small, being in the range, 71–74%. The structural coarsening is accompanied by softening. Similar to the evolution in the average subgrain size, the largest change in hardness is observed after the first 10 minutes at 170 °C followed by only minor additional softening during further annealing (figure 2b).

To monitor changes in the deformed microstructure during the period of rapid coarsening in more detail, figure 3 shows fragments of the EBSD maps obtained in one region with a step size of 25 nm both before annealing and after 10 minutes of annealing. It is evident from this figure that boundary migration and TJ motion took place in many locations of this region. To quantitatively analyze which junctions are more prone to migration than the others, 653 TJs identified in the inspected region were first grouped into four types: HHH, HHL, HLL and LLL, where the number of "H" and "L" corresponds to the number of HABs and LABs in each junction. Thus, HHH junctions contain three HABs, HHL junctions contain two HABs and one LAB, etc. The TJs were then sorted out into two categories: (i) fast-migrating, i.e. junctions migrating over at least 100 nm; and (ii) all other TJs, which were considered slow or stationary.

The results of this categorization approach are presented in figure 4. It is seen that in the deformed microstructure the largest group of TJs was of the HHH type. HHL junctions were slightly less frequent (see figure 4a). 112 TJs were LLL and only 25 of all TJs were of the HLL type. The frequencies of rapidly migrating TJs (green in figure 4a) in the inspected region can be ranged in the same order: HHH, HHL, LLL and HLL. However, when the frequency of rapidly migrating junctions is normalized to the number of TJs within each group, it is clear that the fraction of migrating HLL-type junctions is considerably higher than that of the LLL-types (see figure 4b). Thus, the percentage of fast-migrating TJs is highest within the HHH group, i.e. when all boundaries are HABs, and this percentage consistently decreases with increasing number of LABs present in TJs, being lowest for the LLL group.

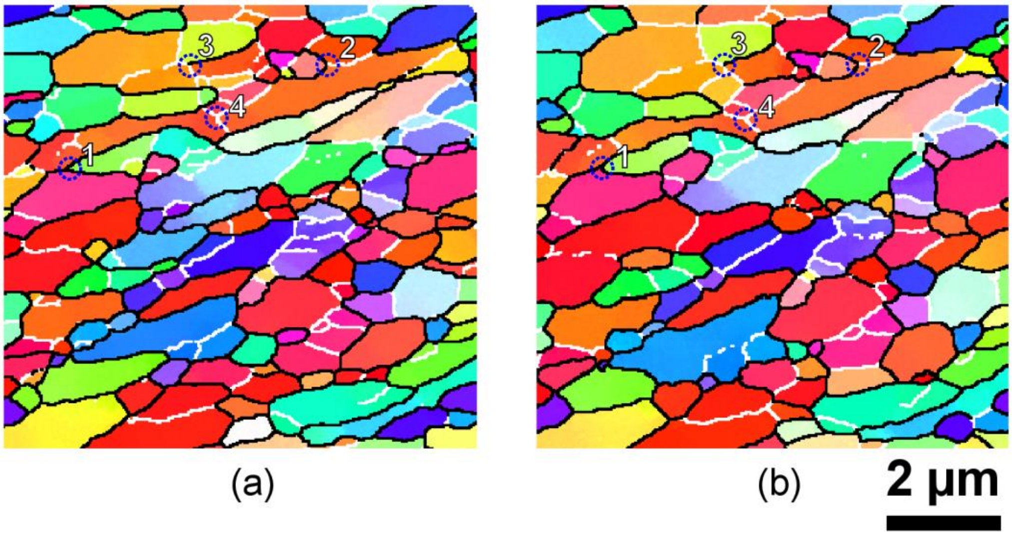

Figure 3. Changes in one region mapped using the EBSD technique in the deformed condition (a) and after annealing at 170 °C for 10 minutes (b). White and black lines show LABs and HABs, respectively. Examples of four different TJ types considered in this work are marked by blue circles and numbers: 1 – HHH, 2 – HHL, 3 – HLL and 4 – LLL. The extrusion direction is parallel to the scale bar.

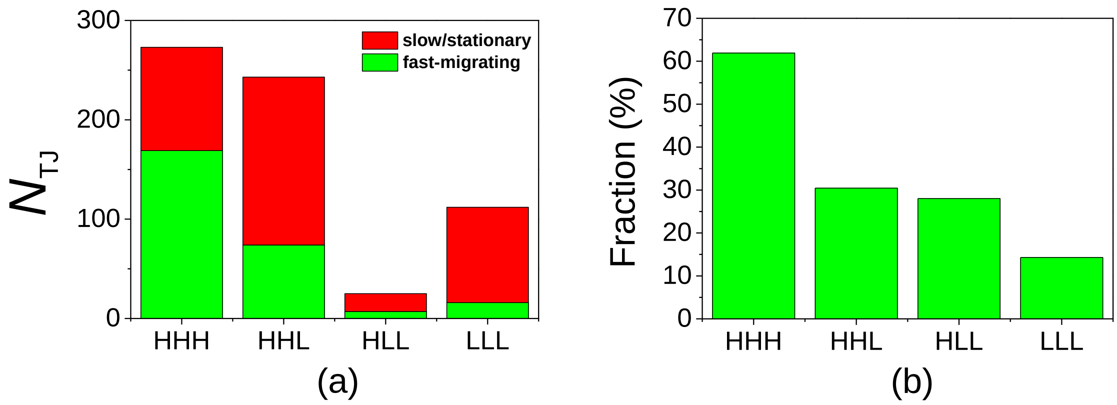

Figure 4. Populations of different TJs investigated in one region both in the deformed condition and after 10 min at 170 °C: (a) frequencies of TJs classified as either fast-migrating or slow/stationary; (b) fractions of fast-migrating junctions in each TJ group.

4. Discussion

Structural coarsening taking place in the heavily ECAE-processed AA1050 plate during annealing at 170 °C increases the average subgrain size from 0.5 µm in the as-deformed condition to ~0.8 µm after 2 h of annealing, and softens the material (see figure 2). The distinct feature of this coarsening process is that the increase in the average subgrain size does not appreciably affect the morphology of the microstructure. In this regard, the coarsening process in the ECAE-processed AA1050 plate is phenomenologically similar to that previously reported for heavily rolled aluminum [1–4], where a lamellar morphology obtained by rolling was preserved during coarsening, which proceeded via lateral motion of TJs. For ECAE-processed aluminum investigated in the present work it is also found that TJ motion plays a significant role during coarsening of the deformed microstructure at least within the first 10 minutes at 170 °C.

Categorization based on types of boundaries composing TJs, conducted in our study, clearly indicates that the susceptibility to rapid TJ motion during this initial period of coarsening depends on the number of LABs and HABs in a TJ. HHH-type TJs are found to be much more prone to rapid TJ motion than any other TJ type (figure 4b). Taking into account that the HHH-type TJs are also most frequent in the deformed microstructure of the given ECAE-processed aluminum plate (figure 4a), it can be concluded that HHH junctions play a dominant role in the process of TJ motion of the microstructure investigated in the present work. It should however be mentioned that since the TJs of the present sample have been characterized only using surface inspection, it is possible that the observed migration may also be influenced by the motion of boundary segments and TJs below the surface.

Whereas TJs of the HHL-type are also rather frequent in the deformed microstructure and thus can appreciably contribute to coarsening via TJ motion (see figure 4a), the total number and the frequency of fast-migrating boundaries of the HLL- and LLL-type TJs are too small to have a significant contribution to the coarsening process. The fact that TJs containing LABs migrate less than HHH-type TJs is not surprising as the mobility of LABs is known to be generally lower than that of HABs (except for coherent twin 60° ⟨111⟩ boundaries, which are however very rare in Al). Considering this, it appears more surprising that some LLL-type TJs were able to migrate quickly. Analysis of the 16 fast-migrating LLL junctions indicated that 11 of them contained at least one boundary with a misorientation of above 10°. Therefore, their properties can be considered similar to those of HHL and HLL-type TJs. Nevertheless, the remaining 5 LLL junctions formed by boundaries with misorientations less than 10° also migrated over fairly large distances.

A small subset from figure 3a is shown in figure 5a. This subset contains three migrating LLL-type junctions with $\theta \approx 6°$ across boundary 1–4; $\approx 7°$ across boundaries 1–2 and 2–3; $\approx 9°$ across boundaries 2–4, 3–4, 5–6; and approximately 2° and 11° across boundaries 1–5 and 1–6, respectively. After 10°minutes at 170 °C subgrain 2 becomes rather large, thus extending the length of LABs 1–2 and 2–4, whereas subgrains 3 and 8 disappear (see figure 5b). The disappearance of these subgrains results in the removal and the formation of several HABs and LABs in the microstructure. Since subgrains 2 and 8 were almost equaixed in the as-deformed condition, their evolution during annealing may be described as a result of conventional curvature-driven subgrain growth.

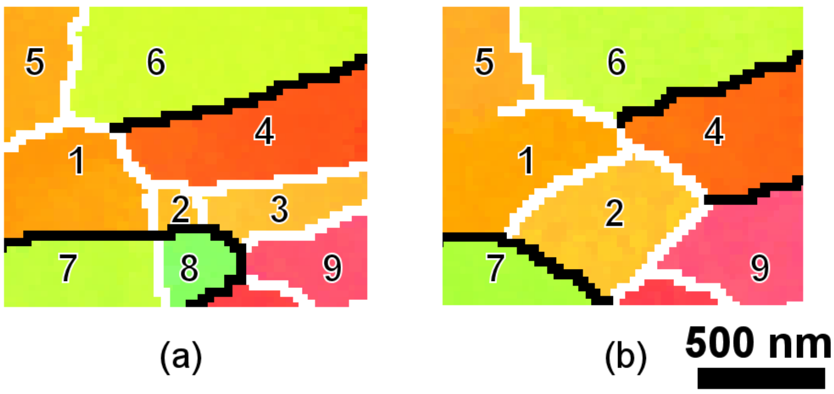

Figure 5. An example showing migrating LLL-type junctions (a) before and (b) after annealing at 170 °C for 10 minutes. White and black lines show LABs and HABs, respectively. Numbers mark subgrains present in this area.

It is apparent that coarsening in the ECAE-processed microstructure affects all boundary types, thus maintaining the proportion of HABs and LABs in the coarsened microstructure similar to that in the as-deformed sample. The latter contrasts our previous observations made in heavily rolled materials, where coarsening was found to result in considerable reductions in the fraction of HABs [3–5]. This difference in the evolution of the HAB fraction in either rolled or ECAE-processed samples may result from the difference in crystallographic textures produced by the two deformation processes: i.e. between a very strong rolling texture in the heavily rolled material [4,5] and a rather weak texture in the ECAE-processed plate [15].

Another difference between heavily rolled aluminum analyzed in [1–4] and the ECAE-processed sample studied in the present work is the extent of annealing regime where TJ motion dominates. For Al cold-rolled to a strain of 6, coarsening is such that the average boundary spacing measured along the normal direction increases 3 times before a significant amount of curvature-driven coarsening is observed [3]. In contrast, for the ECAE material of the present study some evidence of curvature-driven subgrain coarsening is seen when the average subgrain size increases only approximately 1.3 times (after 10 minutes of annealing at 170 °C, see figure 5). This suggests that for the ECAE sample there is a greater competition between regular coarsening and TJ motion. This difference, as compared to the rolled samples, may arise both from the pronounced lamellar morphology of the rolled microstructure characterized by a higher aspect ratio [1] and from differences in the spatial arrangement of HABs and LABs in each sample.

5. Summary

EBSD investigation of an ECAE-processed aluminum plate reveals the importance of TJ motion in microstructural coarsening during annealing at 170 °C. Coarsening by TJ motion in this material is phenomenologically similar to that seen during annealing of aluminum rolled to similarly high plastic strains, although the extent of this coarsening is smaller for the ECAE sample. Direct evidence for coarsening via TJ motion is provided by examination of the same region before and after annealing for 10 minutes at 170 °C. In this experiment it is found that junctions formed by three HABs play a dominant role in the process of TJ motion in the given material, and that the presence of even one LAB has a significant effect on the likelihood of rapid TJ motion.

Acknowledgements

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