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Annealing behavior of aluminium deformed by equal channel angular pressing

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Abstract

Samples of commercial purity aluminum (99.5%) deformed by equal channel angular extrusion (ECAE) to accumulated strains of between 1 and 10 were annealed at different temperatures for a time of 2 h. The microstructural evolutions of both the deformed and the annealed materials were studied by electron back-scattered pattern (EBSP) analysis. At high strains the average cell size is only slowly refined, whilst the average cell boundary misorientation increases more quickly. Examination of the subgrain morphology during annealing suggested that whilst for low strains a recrystallisation occurred in a discontinuous manner, at high strains the samples showed traits of both discontinuous and continuous annealing behavior.

Keywords: ECAE; ECAP; EBSP; EBSD; Recrystallisation; Continuous coarsening

1. Introduction

Nanometer and submicron grain-sized materials fabricated by material scientists have attracted much attention from material scientists owing to their high tensile strength at room temperature and their potential for superplasticity in the medium temperature range [1–4]. Amongst the methods to fabricate fine-grained materials by severe plastic deformation (SPD), equal channel angular pressing (ECAP) has the advantage of being applicable, producing a deformation with no net shape change. Recently, ECAP-deformed samples of different sizes (extrusion diameter varying from just several millimeters to several centimeters) were studied [5], and it was concluded that the initial size of the extruded material has no effect on the mechanical properties and microstructure.

Whilst SPD samples have high strength, it is known, however, that they also exhibit a low ultimate tensile strain. It is important therefore to study the annealing behavior of SPD materials with the objective of improving ductility whilst minimizing the loss of tensile strength. Until now, the stability of ECAP-deformed materials during annealing [6] has been little studied, especially in the low strain regime. Several studies have shown that for dislocation cell-forming materials, during ECAP deformation, both the dislocation cell size and the average misorientation angle across the dislocation cells increase with accumulated strain [7–9]. It should be very interesting therefore to study the annealing of such materials over a wide strain range, to investigate the effect of the initial deformed

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microstructure on the annealing behavior. Humphreys [10] has suggested that for materials with a very large fraction of high angle boundaries, annealing should result in a process of continuous coarsening rather than discontinuous recrystallisation.

Optical microscopy and scanning electron microscope (SEM) techniques can only be used to reveal the grain morphology of the microstructure and do not provide qualitative information concerning crystallite orientations. The transmission electron microscope (TEM) can be used to characterize the grain morphology and provide at the same time information concerning crystallographic orientations. However, semi-automatic TEM techniques for determination of orientations using a Kikuchi pattern analysis are still time-consuming and not well suited for studies with a large matrix of experimental conditions. Recently, the electron back-scattered pattern (EBSP) technique has been developed, and has been widely used for crystallographic microstructural characterization, owing to the high speed of orientation data collection and applicability of the technique for use on bulk samples. The EBSP technique is therefore appropriate for studies of annealing behavior, where a statistical description of microstructural evolution is needed for a thorough understanding of the annealing behavior.

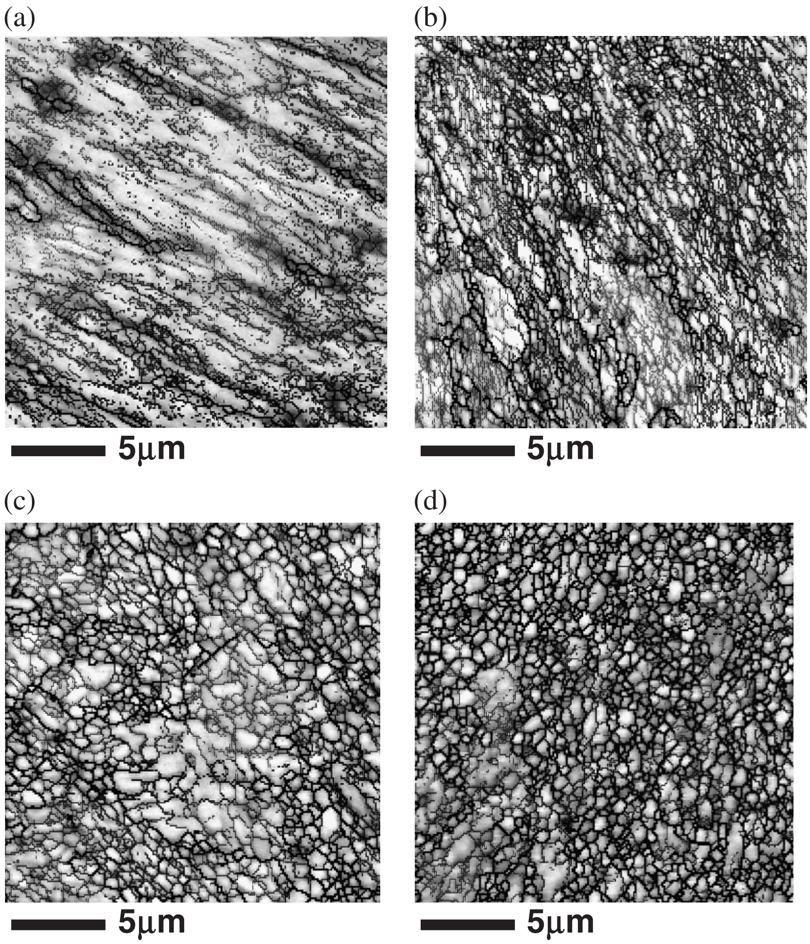

Fig. 1. Microstructural evolution of commercial purity aluminum during ECAE processing (route Bc). Misorientations between adjacent pixels of >1.5° and >15° are shown as thin and thick black lines, respectively. The misorientation data is superposed on a map of Kikuchi band contrast (white corresponding to high pattern quality). The EBSP map step size in each case is 0.1 μm: (a) 1 pass, (b) 2 passes, (c) 4 passes and (d) 10 passes.

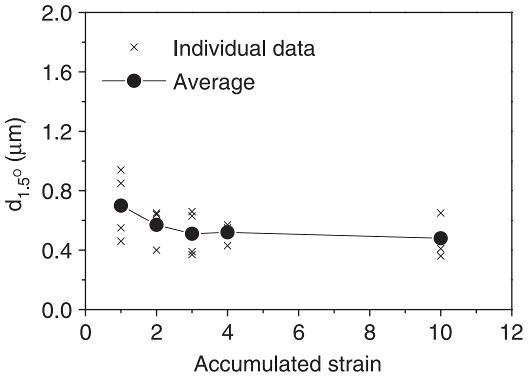

Fig. 2. Evolution during ECAP deformation of cell size (using a grain reconstruction method with misorientation threshold of 1.5°). Individual measurements are marked with a cross to show the spread of values obtained.

In this study the EBSP technique was used to study the microstructural evolution during annealing of an ECAE-deformed material as a function of both accumulated strain and annealing temperature, with the objective of characterizing the effect of the deformed microstructure on the annealing behavior.

2. Experimental

Commercial purity aluminium (99.5%, AA1050) with initial grain size about 50 μm was deformed by ECAP at room temperature using route Bc (clockwise rotation of 90° about the extrusion direction after each pass). Samples were taken after 1, 2, 3, 4 and 10 passes (approximate accumulated strains of 1, 2, 3, 4 and 10). The deformed materials were annealed at temperatures of 210, 240, 270, 300, 420 and 500 °C in air for 2 h.

For the annealing, discs with diameter of 8 mm and thickness of 3 mm were cut from the middle part of the ECAP-extruded rods, with cutting plane perpendicular to the extrusion axis. Microstructural characterization was also carried out in this sample section. For the examination of the microstructure by EBSP, the cut plane was ground to SiC4000, before final electropolishing in a 10:90 $HClO_4$/CH4CO solution at −30 °C and 30 V for 45 s. The microstructures were examined in the LEO1530-SEM equipped with an EBSP system, using 20 kV accelerating voltage with a working distance of 24 mm, and 70° sample tilt angle. For the deformed samples, the microstructural parameters are the average for four investigations in different regions of the samples. For the annealed samples, one EBSP investigation was used for each combination of strain and annealing temperature.

For EBSP data, in order to define a dislocation cell size (subgrain size), the misorientation required to define a boundary must be chosen. The minimum value must be chosen above the angular resolution of the measurements to avoid convolution of the data with misorientation resulting from orientation noise in the data. Whilst for undeformed single crystals the orientation noise can be close to 1° [11], for deformed materials it may be higher than this [12]. In this study a misorientation definition of 1.5° was used for both the deformed and the annealed data. Dislocation cells were identified using a grain (area) reconstruction method [11]. The average misorientation of the dislocation cell structure was determined by considering all adjacent site misorientations of angle greater than 1.5°.

3. Results

3.1. Evolution of the deformed microstructure during ECAP processing

Fig. 1 gives examples of the change in deformation microstructure with increasing strain. In these EBSP maps, misorientations between adjacent pixels of 1.5° and 15° are drawn in gray and black, respectively. In

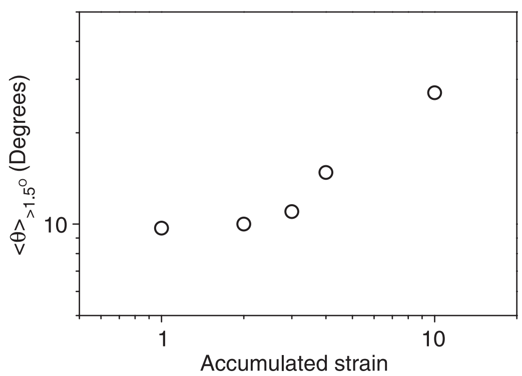

Fig. 3. Variation of average misorientation (only considering values >1.5°) with increasing ECAE strain. Individual values are marked with a cross.

each case, the EBSP grid step-size is sufficiently small that these misorientations should represent the dislocation cell structure. A cellular structure begins to form after 2 ECAP passes. After 10 passes, almost 70% of the boundaries have misorientation angles of greater than 15° and the microstructure can be considered to be submicron grain-sized [13]. As seen in Fig. 1, however, some heterogeneity exists in the distribution of the high angle boundaries, even after 10 ECAP passes. Fig. 2 shows the evolution of the dislocation cell size during deformation process. The cell size changes rapidly initially, but only a slow refinement of cell size is beyond 2 ECAP passes. Fig. 3 describes the evolution of the average cell boundary misorientation with increasing ECAP strain. The average misorientation initially increases slowly, and then, after 3 ECAP passes, increases more rapidly.

3.2. Evolution of microstructure during annealing

Fig. 4 gives examples of the microstructures of samples of different accumulated strain annealed 300 °C. In these figures, the grayscale corresponds to the Kikuchi pattern quality, such that, in general, light

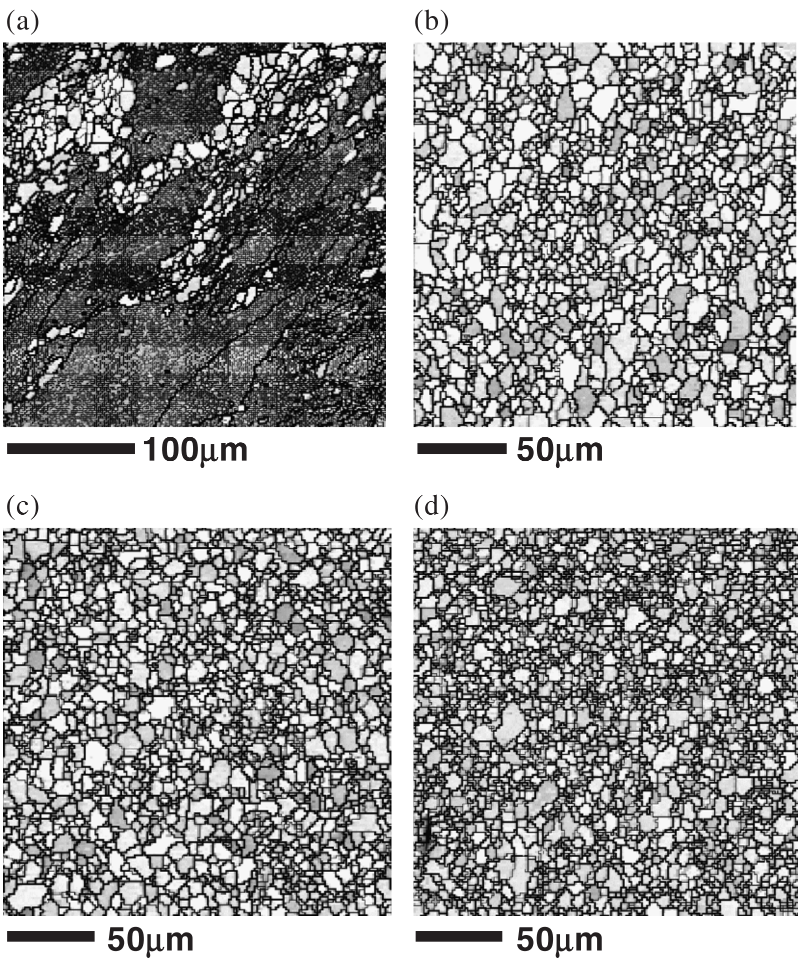

Fig. 4. Microstructural evolution of ECAP processed samples after annealing for 2 h at 300 °C. Misorientations between adjacent pixels of >1.5° and >15° are shown as thin and thick black lines, respectively. The misorientation data is superposed on a map of Kikuchi band contrast (white corresponding to high pattern quality). The EBSP map step size in each case is 0.1 μm: (a) 1 pass, (b) 2 passes, (c) 4 passes and (d) 10 passes.

areas represent to recovered/recrystallized material whereas dark areas correspond to places where the EBSP images were poor (in deformed regions and near grain/cell boundaries). Absolute values should not ascribed to the data as the Kikuchi pattern quality can also vary with surface preparation and microscope operating conditions. Misorientation angles between adjacent map pixels of >1.5° are also marked.

At this annealing temperature, only the 1-pass sample shows a microstructure typical of a discontinuous recrystallization process, with a clear separation in the microstructure between the recrystallized and still deformed areas. In the 2-, 4- and 10-pass samples annealed at 300 °C/2 h, an approximately equiaxed microstructure is seen, though the average cell size is fairly small with respect to the average cell size in the deformed state. Examples of the microstructural evolution in the 2- and 10-pass samples annealed at lower temperatures (240 and 270 °C) are shown in Fig. 5. Here it is seen that in the 2-pass sample annealed at 240 °C, the microstructure suggests a discontinuous (nucleation and growth) type recrystallization process. For the 10-pass sample, the interpretation of the microstructure is more complicated. After annealing at 270 °C, in places some large grains (20–40 μm) are seen, whereas elsewhere the microstructure has also

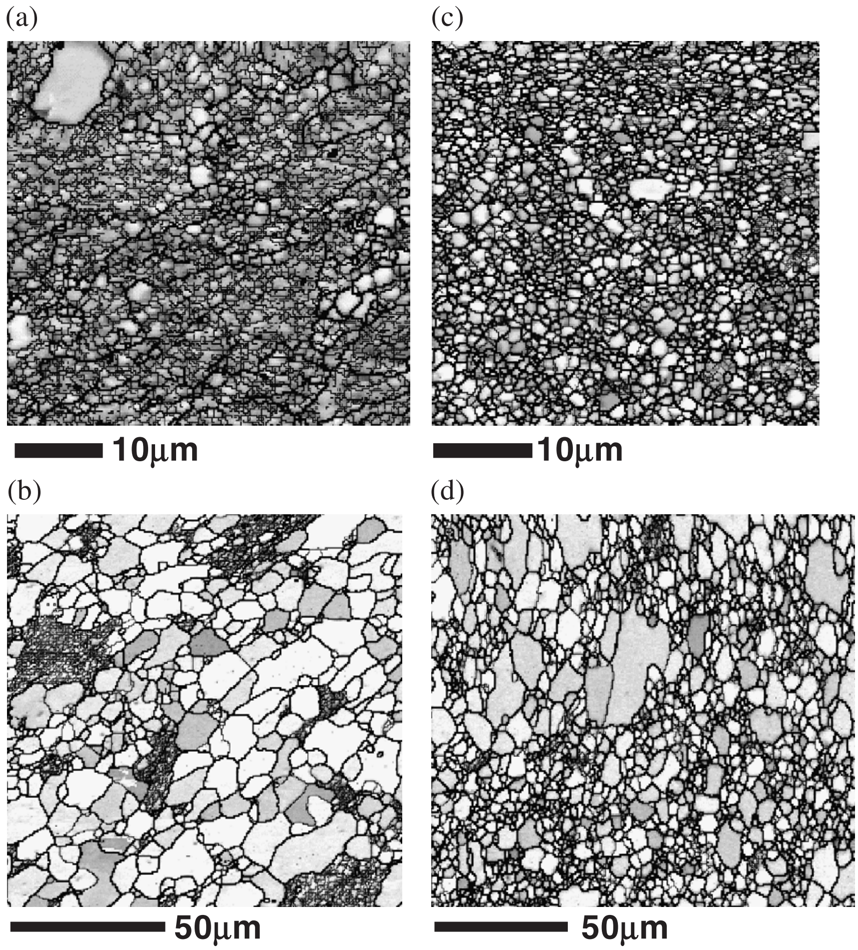

Fig. 5. Microstructural evolution of 2- and 10-pass ECAP processed samples after annealing for 2 h: (a) 2 passes, 240 °C; (b) 2 passes, 270 °C; (c) 10 passes, 240 °C; and (d) 10 passes, 270 °C. Map plotting conditions are the same as for Fig. 4.

coarsened, though by lesser amount with respect to the deformed state. The observation of a coarsening in all places of the deformed microstructure is confirmed by the 10-pass sample annealed at the lower temperature of 240 °C, where the average cell size is 0.80 μm (cf. 0.48 μm in the as-deformed condition).

The average cell sizes after annealing for 2 h at each temperatures of 210–300 °C are summarized in Table 1. For these data (using a 1.5° misorientation criterion), an attempt was made to separate the discontinuous and continuous components in a semi-quantified manner. First the average cell size was calculated for all cells (d_{av}). Next the data were divided in two subsets, those with sizes $d \leq 2d_{av}$ and those with sizes $d > 2d_{av}$, and the average size within each of these was subsets calculated ($d^*_{av}(<2d_{av})$ and $d^*_{av}(>2d_{av})$). If the ratio of these two averages is larger than 2.7, then the average values are given separately. The choice of the value 2.7 was made after a visual inspection of the EBSP maps, to identify those cases where the annealing behavior had a clear mixed behavior.

Fig. 6 shows the evolution of average misorientation with temperature during annealing. For the 10-pass ECAP sample, the average misorientation remains approximately constant with annealing temperature, whereas for the samples deformed to accumulated strains of 2 and 4, an increase of average misorientation is observed with increasing annealing temperature. For the 1-pass sample, there is little change in the average misorientation, reflecting the fact that after 2 h at 300 °C this sample has only partly (discontinuously) recrystallized.

Table 1
Cell sizes (using a 1.5° misorientation definition) for the ECAP material after annealing at temperatures between 210 and 300 °C for 2 h

ECAE strain	As deformed (μm)	210 °C (μm)	240 °C (μm)	270 °C (μm)	300 °C (μm)
1	0.70	–	0.88	1.3 (5.1)[a]	2.7 (8.9)[a]
2	0.57	0.76	0.96[b]	1.3 (6.9)[a]	5.8
4	0.52	0.64	0.77	2.1 (7.5)[a]	5.2
10	0.42	0.55	0.80	2.4 (8.0)[a]	4.3

Samples with ratio $d^*_{av}(<2d_{av})$:$d^*_{av}(>2d_{av})$>2.7 are treated as having both normally and abnormally coarsened (discontinuously recrystallized) components, and average values are given for each part separately.

[a] Average size of discontinuous/abnormally coarsened cells (see text for criterion used to separate the data).

[b] Just one very large recrystallized grain seen in this sample (d_{ECD} = 8.9 μm).

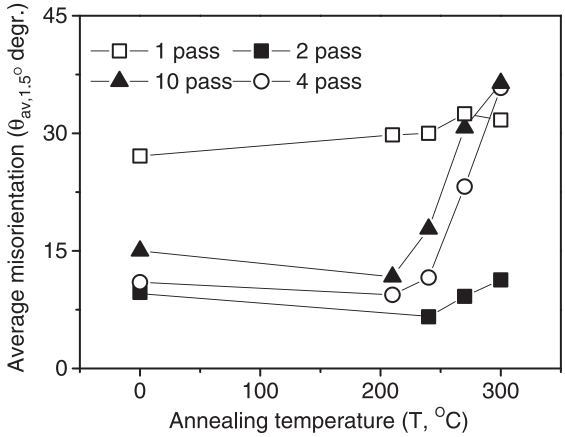

Fig. 6. Variation of average misorientation (only considering values >1.5°) as a function of annealing temperature (2-h duration) and ECAE strain.

4. Discussion

4.1. Evolution of microstructure during deformation

The microstructure resulting from cold-rolling has been described as being comprised of cell blocks delineated by the parallel geometrically necessary boundaries (GNBs), which contain cells defined by incidental dislocation boundaries (IDBs) [14,15]. Many studies have shown that the evolution with increasing strain of both spacing and misorientation is different for each of these two boundary types [16,17]. In the microstructure resulting from ECAP processing, it is not possible to distinguish the GNBs and IDBs on a morphological basis, as it is for cold-rolled microstructures. However, it is known that the average IDB misorientation is low even at high strains, with a saturation possibly at around 3° [18,19]. By considering only misorientations in the EBSP data of 1.5° or greater, the average misorientation and spacing data can therefore be considered as being mostly for boundaries comparable to the GNBs in cold-rolling. It is shown elsewhere [13] that a higher cutoff of 2° makes little change to the form for the evolution of the data, except for after just 1 ECAP pass.

It is seen in Fig. 2 that the cell size formed during ECAP deformation initially decreases rapidly, but that after 2 passes significantly less refinement is observed. This behavior is more similar to that seen during cyclic extrusion–compression, where a stable cell size is quickly developed [20]. During cold-rolling, on the other hand, a continual decrease in GNB spacing with increasing strain has been reported [14,15,17–19]. The average misorientation however formed in the ECAP process increases slowly at first, then after 3 passes increases more quickly, at high strains following a power law relationship with strain with an exponent closer to that seen in cold-rolling than CEC [13].

The differences between the three processes may be explained by the deformation conditions. Cold-rolling is a unidirectional deformation process, and the CEC process is a pure cyclic process (with a full strain reversal). The ECAP-Bc process is, however, only partly cyclic, with a rotation of 90° about the extrusion direction between each pass.

4.2. Annealing of ECAP material

Annealing of the 1 ECAP pass samples results in a straightforward discontinuous recrystallization process, with the nucleation and growth of recrystallized grains surrounded by high angle boundary. With increasing ECAP deformation, however, the annealing behavior is more complex, tending towards a situation where the entire structure undergoes a coarsening. This coarsening is, however, not uniform, as can be seen in the microstructures. In a few cases the size distributions of the uniformly coarsened and abnormally coarsened (or discontinuously recrystallized) parts are sufficiently separated that an average size for each component can be readily estimated. However, in many cases, particularly for the 4- and 10-pass samples, although it can be seen that the spread in cell sizes is larger than expected for uniformly coarsened structures, it not possible to separate clearly the two cell size distributions. In the present study we have separated the data based on the ratio between the cell sizes below and above the average for all cells $d^*_{av}(<2d_{av})$:$d^*_{av}(>2d_{av})$ (where d_{av} is the average for all cells, and d^*_{av} are averages for subsets of the data). This method of separating the data in only approximate, as d_{av} depends on the fraction of any larger size component, as well as its magnitude. Based on visual inspection, a value of the ratio $d^*_{av}(<2d_{av})$:$d^*_{av}(>2d_{av})<2.7$ was taken to define a normally coarsening structure. For comparison, for an ideal Rayleigh distribution of cells sizes the value of this ratio would be 2.41.

Work on more sophisticated techniques to perform a separation of the size distribution data is currently underway. It should be noted in this regard that even the overall change in grain size (using a 15° misorientation definition) during annealing is only small, particularly for the 10-pass ECAP material (Table 1), where after 2 h at 300 °C the grain size is only 6.4 μm.

The data suggest that the annealing response occurs in two ways. In some places a locally discontinuous recrystallization process is seen, whereas elsewhere a gradual coarsening of the microstructure is seen. Two interesting and important questions are why in some places recrystallization occurs discontinuously, and why these regions do not grow rapidly to consume the microstructure. Other studies have noted that at high deformation annealing results in a continuous coarsening (sometimes referred to as continuous recrystallization) rather than a discontinuous recrystallization process [10,21]. Recently, Morris and Muñoz [22] have also noted that for an ECAP-deformed Al–3.6% Mg alloy, some regions of the sample appeared to undergo conventional discontinuous recrystallization, whereas in other regions only a coarsening was seen. In their study [22], they suggested that the difference in behavior was due to heterogeneities in the deformed microstructure leading to regions with different amounts of high angle boundaries. In detailed studies of the deformed microstructure, a heterogeneous pattern has indeed been observed. However the length scale of the differences is only small, as also seen in the annealed microstructures, so that the recrystallization response must be regarded as being mixed of two processes. For a better understanding of the annealing behavior, it will be necessary to develop techniques to identify and separate the discontinuous and continuous recrystallized components, particularly for low-temperature annealed samples, where the differences in cell sizes are small, but may provide information about local discontinuous nucleation events.

5. Conclusion

Commercial purity aluminum processed by ECAE technique was characterized using the EBSP technique. The annealing behavior of the deformed material was studied based on an analysis of the cell and grain morphology. It was found that whilst for samples deformed to only 1 ECAP pass, annealing resulted in a conventional discontinuous recrystallization process, with increasing ECAP deformation, a mixed process was observed, with some part of the microstructure showing discontinuous recrystallization, and elsewhere a continuous coarsening. The grain size of the 10-pass ECAP sample after annealing at 300 °C for 2 h was only 6.4 μm.

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