The nucleation of cube grains during primary recrystallization of aluminum

M M Miszczyk$^{1,a}$ and H Paul$^{1,b,*}$

$^1$Polish Academy of Sciences, Institute of Metallurgy and Materials Science, Krakow, Poland
$^a$m.miszczyk@imim.pl, $^b$h.paul@imim.pl

*Corresponding author: h.paul@imim.pl

Abstract. The formation of cube grains during recrystallization has been analyzed in commercial AA1050 alloy and pure Al single crystal of S(234)<524> orientation. Samples of AA1050 alloy were deformed along two deformation modes to form different as-deformed texture components and then lightly annealed. One group of the samples was plane strain compressed (PSC) in a channel-die. The second group was deformed by equal channel angular extrusion (ECAE). The results obtained on polycrystalline AA1050 alloy were compared with recrystallization behavior of PSC single crystal of S{234}<524> orientation. The textures were measured by SEM/EBSD and X-ray diffraction. It was found that a very weak residual cube{100}<011> component was observed in the samples of AA1050 alloy after both deformation modes. After annealing cube-oriented grains were extensively formed only in PSC samples. These grains were situated preferentially inside or in between the S-oriented areas with local disorientations about <111> axes. Cube-oriented grains were not formed during annealing of the ECAE samples, where main as-deformed texture components close to two complementary orientations of {124}<651>-type were transformed towards two components of ~{100}<011> and ~{221}<114>, essentially by <110> rotations. In single crystalline samples cube grains were extensively formed during annealing despite (near)cube-oriented areas haven’t been observed in the as-deformed microstructure.

1. Introduction

Control of earing properties of thin sheets relies on a careful control of the texture. It is well-known that the minimal earing can only be obtained by well-balanced texture. In the case of Al and its alloys a key concern is to obtain a sufficient amount of cube{100}<001>-oriented grains. However, the origin of strong cube texture during annealing of cold-rolled face centered cubic (fcc) metals still constitutes an important scientific and technological problem, e.g. [1-13]. Most of the authors argue that {100}<001>-oriented grains evolve from ‘cube bands’, which are present in the deformed microstructure, often as thin (near)cube zones between high orientation gradients created by diverging rotations of unstable orientations [13] or fragmented leftovers of the original cube grains [8]. Two different cube grain nucleation models have been proposed. A first ‘nucleation’ model [2] proposes that the deformed cube-oriented bands recover faster due to the easy annihilation of two sets of pure screw dislocations with perpendicular Burger’s vectors, without any requirement of orientation relation across recrystallization front. The second ‘micro-growth’ model [3, 4] is based on the strong growth rate dependence of ~40°<111>-type between areas of the deformed state of S{123}<634>
orientation and cube recrystallized nuclei. Both models, which may be concurrent or in competition, or even superseded by some other mechanisms, assume presence of initial cube nucleus in the deformed state.

However, the statement that cube-oriented nucleus originate from fragmented leftovers of the original cube grains is strongly disputable since the cube-oriented crystallites are very unstable during room temperature rolling or plane strain compression (PSC). At the beginning stages of deformation the grains of this orientation splits up by transverse and rolling direction rotations and evolve, in the range of very high deformations (> 90%), towards four variants of ~S orientation, e.g. [1,13]. To solve this problem some of the authors assume, e.g. [8] that despite large volumes of cube crystallite are unstable (and rotates towards different variants of S orientation) but small fragments could be metastable because they are subject to a different stress state. These less deformed areas conserve orientations near the original cube orientation and they are the source of cube grains nucleation during recrystallization. But this description of the phenomenon leads to another difficulty - it is very difficult to accept the assumption that during annealing less deformed cube-oriented leftovers are more privileged sites for nucleation of new grains (areas of lower stored energy) as compared to highly deformed ones (areas of higher stored energy). Moreover, the experiments on deformed single crystals of fcc metals of stable orientations, deformed up to moderate strains clearly show that orientations of the as-deformed state are not retained in the new grains orientations during primary recrystallization [9, 10, 14, 15]. In these simple cases the orientation and the underlying microstructure of the as-deformed matrix are clearly defined due to the strong stability of such crystals. Since the orientations of new, recrystallized grains are not identified inside the as-deformed structure and the successful nuclei predominantly possess the ~25-55°(~<111>) -type orientation relationship with respect to the deformed matrix, e.g. [1-20], there must be a valid rotation mechanism which transforms the as-deformed state orientations into recrystallized one.

In the case of cold-rolled and annealed polycrystalline fcc metals the situation is ambiguous. As an example, cold rolled commercial purity Al after annealing often develops a mixed cube and ‘R’ texture, where: ‘R’ means Retained component, close to S. Since the as-deformed texture ‘image’ after typical industrial rolling up to 70 - 90% reductions is composed of strong S-orientation of 4 variants (supplemented by 2 variants of brass{110}<112> and copper{112}<111> orientations) and weak cube orientation, there is not clear whether the presence of cube and S-oriented grains after annealing results from validity of the oriented nucleation model or the rotation around selected the <111> axes of S orientation. (It is important to remember that ~40° rotation of ~S orientation around selected the <111> axes is able to transform the S-oriented areas of given variant into (near)S-oriented grain of another variant or into (near)cube orientation).

We believe that the main factor which influences cube grain nucleation are the specific texture components of the as-deformed state. Therefore, the samples of AA1050 alloy were deformed along two deformation modes to form different as-deformed texture ‘images’ and then lightly annealed. The results obtained on polycrystalline samples were compared with recrystallization behavior of PSC single crystal of S{234}[524] orientation. High stacking fault energy (SFE) of Al practically excludes mechanical and recrystallization twinning and it facilitates identifying the mechanisms responsible for the texture transformation during the initial stages of annealing. The basic research technique employed is scanning electron microscopy equipped with an electron backscattered diffraction (SEM/EBSD) facility.

2. Experimental
The materials were a commercial AA1050 alloy and Al(99.998%) single crystal of the (234)[52-4] orientation defined here as ‘S’ orientation. (In fact the orientation was few degree rotated away from ‘classical’ positions of S orientation usually defined as: {123}<634> or {124}<211>). One group of the samples of AA1050 alloy was deformed in a channel-die to the logarithmic strain of 0.52 (41%), whereas the second group was equal channel angular extruded (ECAE) up to six passes along route A. In order to limit the friction each PSC sample was wrapped in Teflon™ tape, whereas in the case of
ECAE samples the billet and tooling were well-lubricated with MnS\(_2\) containing grease. Then the samples were annealed: AA1050 alloy (after PSC and ECAE) - for 1h at temperatures ranging between 100°C and 450°C with the purpose to obtain different stages of recrystallization, whereas single crystal of S orientation - for 30s at 420°C with the purpose to obtain final stage of primary recrystallization (95% of the sample surface is covered by recrystallized grains). The deformed and recrystallized microstructures were analyzed in the ND/ED section (where: ND and ED denote, the normal and the extension directions, respectively) with the use of SEM - QUANTA 3D FEG, equipped with a field emission gun and EBSD facility. At the sample scale, the textures were measured on the ED/TD plane (where: TD denote transverse direction) with the use of the Philips X Pert PW-1830 X-ray diffractometer.

3. Results and discussion

3.1. Microstructure and texture of the as-deformed state

3.1.1. PSC and ECAE of AA1050 alloy. The initial material was in the form of hot-rolled sheet taken from the medium stage of the technological process, i.e. after the reversible mill. At this initial stage, the material was characterized by a typical copper-type rolling texture composed of strong 4 variants of S\{123\}<634>, 2 variants of C\{112\}<111> (or copper) and brass\{110\}<112> orientations and minor cube\{100\}<001>. As expected, the texture of the initial material wasn’t essentially changed during further PSC up to 41\%. (After 41\% PSC the texture image is quite similar to that after large deformations during cold-rolling of many fcc metals of medium to high SFE), e.g. [10].

![Image](a) Orientation map and corresponding (b) the \{100\} pole figure showing microstructure and texture of homogeneously deformed regions of the S\{234\}\{52-4\}-oriented Al single crystal. (c) Disorientation line scan across the structure of elongated cells (red and blue lines show point to point and point to origin disorientations, respectively). Sample PSC up to 41\%. SEM/EBSD local orientation measurements with step size of 100 nm.

However, the application of relatively low cold deformation simplifies the analysis since it excludes the formation of plastic flow instabilities in the form of shear bands in the S- and C-oriented grains [21]). It is clear that it is impossible to apply exactly the same amount of deformation via ECAE processing. After 1-2 passes along route A the texture is almost random, without clearly marked components. A ‘clear texture image’, with two strong components is obtained after 6 passes of ECAE processing (up to 5 passes one still detects additional weaker components of the initial state). The texture after 6 passes is ‘stable’ during further processing, i.e. is not essentially changed up to very high deformations (up to 12 passes). The texture ‘image’ is composed of two nearly complementarily oriented groups of orientations close to \{124\}<651> with slight scattering towards the shear\{100\}<011> orientation. Moreover, in this strongly sheared sample there is also a weak residual cube texture - a component that is in fact closer to exact cube than in the PSC sample.
3.1.2. PSC of aluminum single crystals of S(234)[52-4] orientation. The crystal undergoes a macroscopic $\varepsilon_{ED/TD}$ shear, typical of plane strain compressed S or near S-oriented crystals. Optical microscopy revealed significant changes of the sample shape in the ND/ED section with clearly marked traces of thin bands of localized strain. Generally, the aim was to generate a homogeneous microstructure throughout the samples but this turned out to somewhat over-ambitious since in S-oriented single crystals the regions of both homogeneous and heterogeneous deformation structures were found. After 40% deformation the heterogeneities were thin layers of localized strain crossing the samples at 15-20° to ED. The rest of the sample underwent homogeneous deformation in which the disorientation between neighboring areas is <15°.

Fig. 2. Orientation map (a) and corresponding the {100} pole figure (b) showing microstructure and texture of heterogeneously deformed region of the S(234)[52-4]-oriented Al single crystal. Disorientation line scans across the matrix (c) and across the band (d). Red and blue lines show point to point and point to origin disorientations, respectively. Sample PSC up to 41%. SEM/EBSD local orientation measurements with step size of 100 nm.

In homogeneously deformed areas the SEM/EBSD local orientation measurements revealed microstructures composed of elongated microbands surrounded by low angle boundaries of one or two families (Fig. 1). The inclination of microbands with respect to the external directions depends on the analyzed area. However, strong coincidence with the traces of {111} planes is observed. The crystal lattice of the homogeneously deformed matrix (for larger distances) displayed rotation away from the initial crystal orientation around the axes lying close to TD. However, the disorientation angles in ‘pixel to pixel’ disorientation relation were below 15°. Consequently, the textures of the small areas corresponding to the homogeneously deformed microstructures are only slightly scattered, as shown by the {111} pole figures of the mapped areas. The crystal lattice of areas of the heterogeneous deformation undergoes strong ~TD rotation towards the orientation situated complementarily with respect to the initial orientation. The disorientation across the boundary separating the band and the less deformed matrix very often attains 40-50°. However, it is important to note that disorientation between particular cells inside the bands of localized strain is significantly lower and typically do not exceeds 15° (Fig. 2).

One of the most important conclusion which results from the detailed crystallographic characterization of the as-deformed sample is that the areas of (near)cube orientation are not present in the as-deformed samples PSC up to 40%.
3.2. Texture changes during annealing

Recrystallization behavior of commercial aluminum AA1050 alloy. The exact position of the main texture components of the PSC samples annealed at temperatures below 300°C were quite similar to those observed for the as-deformed state (Figs. 3a-c). At 300°C a radical textural changes occurs due to the development of a strong cube orientation, the retention of the 4 variants of the S orientation as recrystallized grains orientation (with lower intensity compared to the deformed state) and the disappearance of both C (copper) components in the fully recrystallized samples. It was observed that the volume fractions of all four S variants decreased, whereas the volume fraction of the cube component increased with annealing temperature. However, it was also noticed that similar area fractions of near cube-oriented areas, clearly observed in the as-deformed state were still-conserved during annealing up to 325°C. In the case of ECAE samples the most important textural changes were initiated at annealing temperature of 270°C (Figs. 3d-f). The intensity of the as-deformed components near the \{124\}<561> orientations strongly decreases with the progress of recrystallization and this process coincides with an increase of the \{100\}<011> and \{221\}<114> orientations which dominate in the recrystallized structure. Despite the presence of cube-orientation in the as-deformed state and over a wide range of annealing temperatures (of the same intensity) the cube texture development during annealing was not observed. This leads to the conclusion that cube-oriented bands in the as-deformed state are insufficient ‘factor’ to influence cube texture formation after annealing.

![Texture evolution during annealing: (a)-(c) PSC samples, (d)-(f) ECAE samples. The \{111\} pole figures showing essential texture changes. X-ray diffraction.](image)

As for (near)cube-oriented grains there are observed two characteristic groups of places of their nucleation and further privileged growth to the inside of the S-oriented areas (of different variants) (Figs. 4). First group of cube grains is growing near the cube bands (Figs. 4a, c), whereas the second one is growing without any contact with cube bands (Figs. 4b, d), as visible on two dimensional orientation maps. Figure 4a shows a cube-oriented grain growing preferentially into S-oriented areas of the as-deformed state. The bottom part of the grain sticks with a layer (cube band) composed of the
cells or (sub)grains of significantly larger diameter as those typically observed in the other as-deformed areas. In many cases the spread of the orientations of strongly recovered cells mark the transition from the near S to the (near)cube orientations and suggests that the rotation mechanism of $\sim40^\circ\langle111\rangle$-type play a decisive role in the transformation of the as-deformed area orientations ($\sim$S) into the recrystallized one ($\sim$cube). Since the recrystallized grain is elongated along the boundaries of the as-deformed/recovered flat grains, therefore, it is likely that new grain nucleate inside $\sim$S oriented areas of the as-deformed state and its preferred growth is connected with rapid migration of the high angle boundary. When the cube grain stick with cube band (marked as $\sim$cube) the high angle grain boundary across recrystallization front disappears. This explanation of the situation observed in Fig. 4a could be confirmed by the cases showing the growth of cube grains to the inside of the as-deformed areas of S orientation, without necessity to be in contact with cube bands, as observed in Fig. 4b.

![Image of microstructure and orientations](image)

**Fig. 4.** Cube grains nucleation in the as-deformed state of $\sim$S orientation: (a) cube grain growing to the inside of S-oriented areas (of different variants) without any contact with cube bands, and (b) cube grain growing near cube bands (marked as $\sim$cube). The new grain orientations and their nearest deformed/recovered neighborhood are presented in (c) and (d) as the $\{111\}$ pole figures; they corresponds to the orientation maps presented in (a) and (b), respectively. PSC sample up to 40% and annealed for 1 h at 300 °C. SEM/EBSD local orientation measurements with step size of 100 nm.

However, in the case of polycrystalline metals the situation is always ambiguous, since ‘there is a plenty room under bottom...’ and we never know if the cube bands are placed up/bottom observed section or not. The problem can be solved by the analysis of single crystalline samples of S orientation which is semi-stable during PSC up to moderate strains.

3.2.1. Recrystallization behavior of aluminum single crystals of S(234)[52-4] orientation. In the case of single crystalline samples the recrystallized grain orientation were analyzed inspects whole sample area. Figures 5a and b show the microstructure and orientations of recrystallized grains after annealing at 420 °C for 30 s. This recrystallized state can be described as final stage of primary recrystallization

![Image of microstructure and orientations](image)
(~95% of the recrystallized fraction) as analyzed on the sample section perpendicular to TD. Generally, the recrystallized grain orientations are symmetrically situated with respect to the external directions. This symmetry results from the positive and negative rotations of the recrystallized grain orientations around the axes grouped near all normals of the \{111\} planes. The disorientation angles are ranged between 25° and 55° with the maximum near the 40°, as presented many times in the past, e.g. [16, 18, 22-27].

Fig. 5. Cube grains nucleation in the $S(234)[52-4]$-oriented Al single crystal. (a) Orientation map presented as a ‘function’ of grain boundaries (>15°) and texture component (20° scattering of ideal cube \{100\}<001> orientation, where: yellow – ideal cube, red – 20° away from cube) and corresponding (b) the \{111\} pole figure. Sample PSC up to 41% and then annealed at 420°C for 30s. SEM/EBSD local orientation measurements with step size of 5 μm.

The high resolution orientation maps made in the deformed state well-documented that (near)cube-oriented areas were not identified in the as-deformed state and despite this the (near)cube grains nucleate intensively during annealing. This confirm the validity of the mechanism of the recrystallized grain orientations formation based on $-\alpha<111>$-type rotation. It is clear that anti-clockwise rotation of $-S$-oriented areas of the as-deformed/recovered state around common for $-cube$ and $-S$ orientations the $<111>$ axis directly leads to (near)cube grains. This also confirm the thesis that presence of cube nuclei in the as-deformed structure is not necessary condition for (near)cube grains nucleation.

The calculation of disorientation relations between the recrystallized grains of cube orientation and the average orientation of the as-deformed/recovered state shows that most of the disorientation angles across the migrating recrystallization front are within the range of 25 and 55°, whereas the disorientation axes are concentrated around all the $<111>$ poles ‘describing’ the orientation of the as-deformed/recovered state. However, the axes lying near the two highly stressed slip planes during deformation are preferred. It was also noticed that the scattering of the disorientation axes is larger than that of the orientations in the deformed state.

4. Conclusions
The present results provide detailed information about the development of cube texture in a commercial AA1050 alloy and a model Al single crystal of $S(234)<524>$ orientation. Based on local orientation measurements and X-ray diffraction it was found that:

(i) A very weak residual cube texture component was observed in the samples of AA1050 alloy after both PSC and ECAE deformation modes. Despite this cube-oriented grains were extensively
formed during annealing only in PSC samples. These grains were situated preferentially inside or in between the S-oriented as-deformed areas with local disorientations about <111> axes.

(ii) Cube-oriented grains were not formed during annealing of the ECAE samples. During annealing main as-deformed texture components of ECAE samples close to \{124\}<651> were transformed to two components of \{100\}<011> and \{221\}<114>-type, essentially by <110> rotations.

(iii) In the case of Al single crystal of S[234]<524> orientation, the cube grains were extensively formed during annealing despite no (near)cube-oriented areas were observed in the as-deformed microstructure. The strong cube texture after recrystallization results from the ~40°(~<111>)-type rotation of the as-deformed orientations.

(iv) Finally, it is concluded that during annealing of Al samples the cube-oriented grains can nucleate from S-oriented areas of the deformed state free of cube-oriented nuclei.

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