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New insights into Cube nuclei in deformed aluminium

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Abstract The formation of Cube oriented elements in plane strain compressed aluminium has been studied by EBSD for both hot and cold deformations. By following the orientation changes of the same set of 176 grains deformed at 400 °C up to a strain of 1.2 using a split sample, it is shown that about 15% of the grains can break up into several regions of very different orientations, characterized by very large orientation gradients. In particular those grains oriented within about 30° of Cube develop Cube oriented zones in contact with other rolling texture components. Finite element crystal plasticity simulations confirm this mechanism of creation of Cube by plastic deformation. The same type of microstructure can also be observed after heavy cold rolling (strain of 2.3), but at a scale that is much finer by at least an order of magnitude. In this case the micron-sized Cube fragments are located along many grain boundaries or in some particular grains. When the cold deformed sample is annealed, EBSD observations of the same areas reveal that the intergranular Cube fragments are very efficient recrystallization nucleation sites, apparently since they possess mobile high angle boundaries with the local environment.

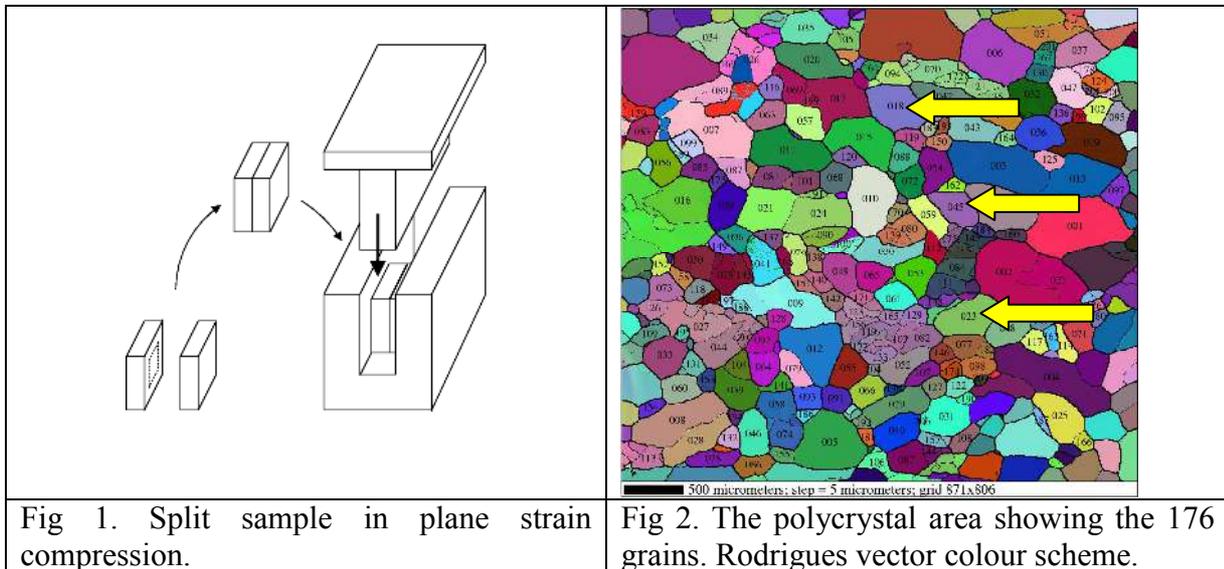
Introduction

Both the formation of Cube oriented elements in rolled fcc metals and their development into Cube grains during subsequent annealing have long been the subject of extensive theoretical and experimental studies. Most present analyses consider that the Cube grains of a polycrystal are unstable during cold rolling, breaking up into bands [1-3] and then fragments which, during subsequent annealing, become very active cube recrystallization nuclei to favour the Cube recrystallization texture. However, despite work for over 50 years, many details of these important physical mechanisms are still basically unknown. The present work describes new experiments involving studies of the same areas during deformation and annealing to show how Cube fragments can be formed during rolling type deformation and how they become potent cube nucleation sites. For hot rolling deformation the split sample method has been perfected by Quey et al [4] to follow the real reorientations of grains in a deformed polycrystal. Following this experimental work, a finite element crystal plasticity (FECP) model has been applied to the deformation of the same “polycrystal”, with the grains finely meshed, to see if one can satisfactorily predict orientation fragmentation in specific grain orientations [5]. Finally, the local evolution, during heating, of a cold rolled sample containing Cube fragments will be presented. In particular it will be shown that the important parameters are the size, density and spatial locations of the Cube fragments for which only extensive EBSD orientation mapping can provide relevant experimental data [6].

Results

Hot Plane Strain Compression

(a) Experiments. In a first set of experiments a split sample of high purity polycrystalline Al-0.1%Mn was deformed in channel-die (plane strain) compression at 400 °C by successive passes up to 1.2 [4]. This set of experiments was conducted at high temperature in order to develop relatively simple, homogeneous, deformation microstructures that could be more easily characterized by EBSD. The sample configuration is shown in Figure 1. The channel-die equipment [7] is designed, and the sample suitably lubricated, to minimize friction effects and therefore avoid major macroscopic strain heterogeneities. It also enables rapid quenching after hot deformation to avoid microstructure change by recrystallization. The orientations of 176 grains were followed by EBSD from the un-deformed state through strains of 0.19, 0.42, 0.77 to 1.2 (termed microtexture tracking). A very large sample area (4 x 4 mm) was examined but the fairly coarse grain and sub-grain microstructures meant that the EBSD step size of 5 μm was quite adequate for this purpose. Fig 2 shows the initial grain distribution and highlights 3 grains of particular interest for the present study. They are average sized grains with no special environment.



During hot deformation most grains showed unimodal rotations (average rotation with some orientation spread) as described in more detail by Quey et al. [4]. However, 19 grains underwent orientation fragmentation, i.e. different regions of the grain rotated in significantly different directions. This phenomenon has been characterized quantitatively by studying the orientation spreads represented in the fundamental region of Rodrigues orientation space [5]. Of this set of fragmenting grains, the three grains highlighted in Fig. 1, initially 20-30° rotated Cube orientations, happened to break up into Cube/Cu or Cube/S regions to generate Cube/ β fibre interfaces. These grains are numbered 018, 023 and 045, and had the following initial orientations: 018 = 27°/ND rot. Cube, 023 = 28°/(0.89xTD + 0.46xND) rot. Cube, and 045 = 25°/(0.39xRD + 0.43xTD + 0.81xND) rot. Cube (in Rodrigues vector convention: 018 = (0, 0, 0.240), 023 = (0, 0.221, 0.114), 045 = (0.087, 0.096, 0.180)). Their development into Cube / β fibre component structures can be revealed by pole figures and texture component maps. In Fig 3, the orientations of the three grains at a strain of 1.2 are illustrated by {111} pole figures, showing very wide orientation spreads and their tendency to split towards two different orientations. In Fig 4 the orientations in the grains are mapped out at the same strain by affecting an orientation to a coloured texture component, using the standard 15° disorientation criterion. The orientation maps reveal that the grains develop Cube / β fibre component regions during deformation (note that, at $\epsilon = 0$, the grain orientations did not align with the texture components and so would have been coloured white).

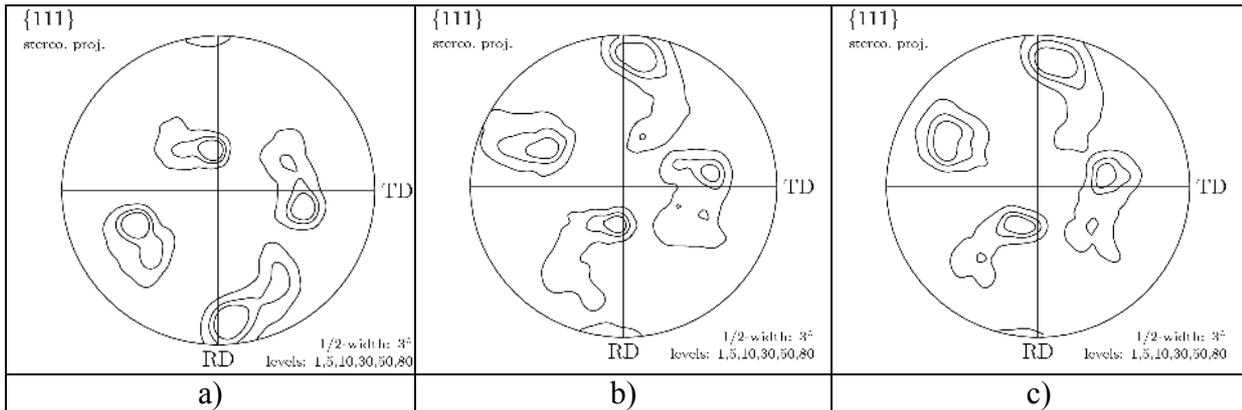


Fig 3 $\{111\}$ Pole figures of grains 018, 023 and 045 as measured by EBSD maps over the entire surface of each grain. The very wide spreads include some Cube and a much higher proportion of β fibre components. a) grain 018, b) grain 023 and c) grain 045.

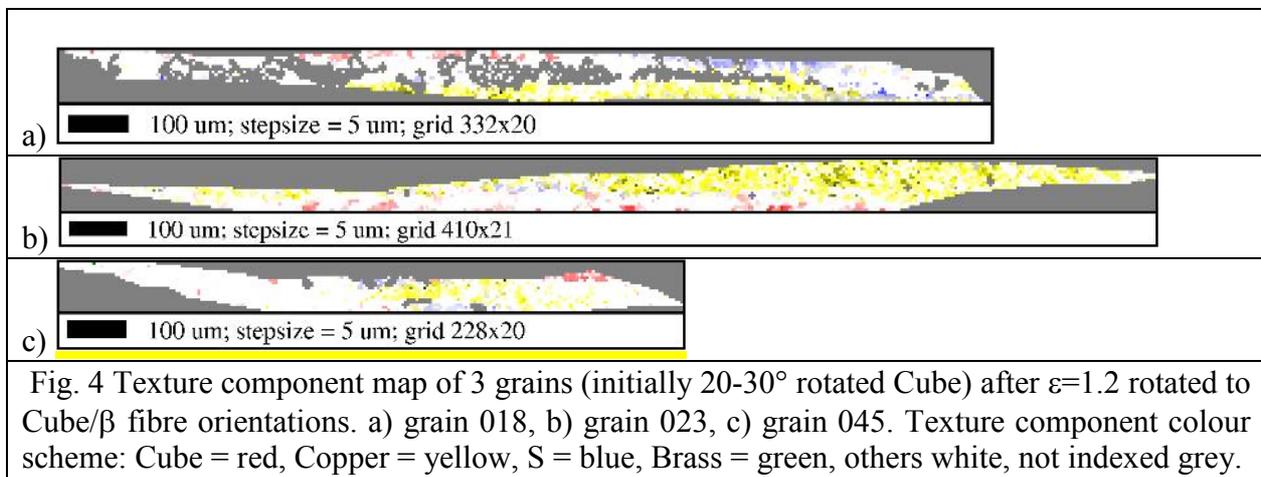
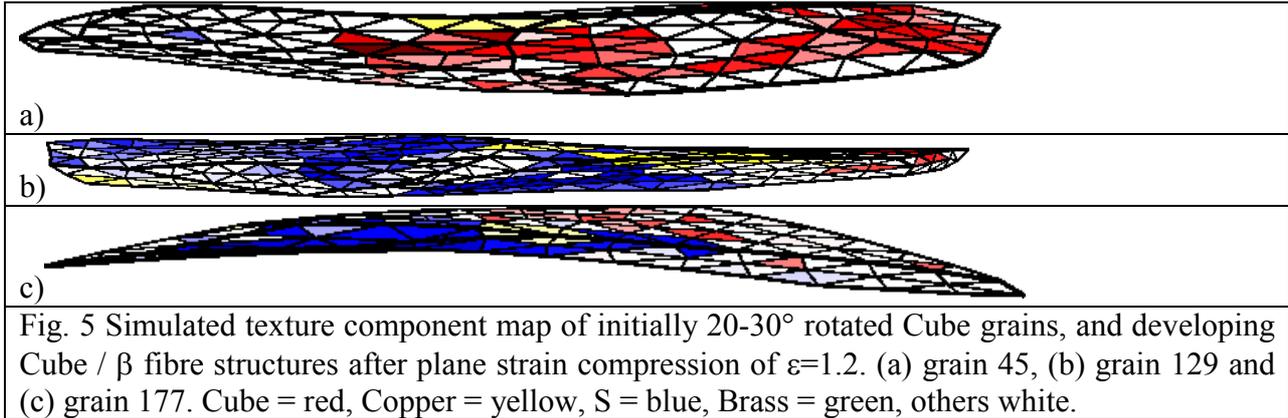


Fig. 4 Texture component map of 3 grains (initially $20-30^\circ$ rotated Cube) after $\epsilon=1.2$ rotated to Cube/ β fibre orientations. a) grain 018, b) grain 023, c) grain 045. Texture component colour scheme: Cube = red, Copper = yellow, S = blue, Brass = green, others white, not indexed grey.

The fragmented grain microstructures are quite complex, composed of small ($10-50 \mu\text{m}$) fragments of Cube with larger quantities of Cu and S fragments, together with off-texture components. Despite this microstructural complexity, it is clear that some parts of the grains have rotated towards Cube and other parts have rotated away towards β fibre components. As recognized by several authors this orientation fragmentation is of major interest for recrystallization nucleation and it would be very useful to be able to model the process.

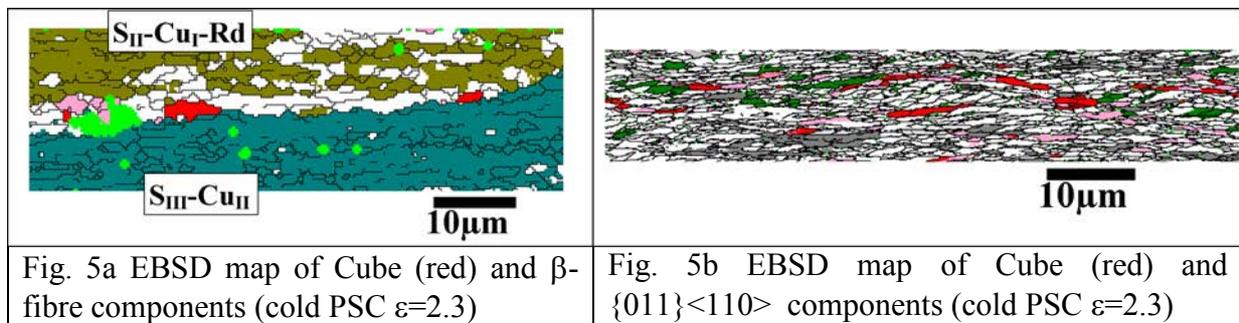
(b) Simulations. A FECP analysis has therefore been carried out for detailed comparison. The FE meshing procedures are described in [8], and the implementation is detailed in [9, 10]. In the present simulation [5], the polycrystal was modelled as a Voronoi tessellation, and the grains were attributed the experimental crystal orientations and finely meshed to enable in-grain orientation spreads (and potentially fragmentation) to develop. The constitutive model uses the experimentally observed slip systems and appropriate hardening laws of Al-Mn single crystals deformed at 400°C [11]. The results have been analysed in terms of orientation fragmentation, as for the experiments, and similar trends for the $20-30^\circ$ rotated Cube grains to develop Cube / β fibre component structures were observed. Here, the predictions can be processed, as in the experiments, in terms of texture component development. A well-developed Cube / β fibre component structure has been reproduced only for one of the three experimental grains (N° 045), but this can be reasonably explained by the fact that the simulations do not use exactly the same polycrystal morphologies as in the experiments so that each grain can undergo different grain interactions. This is confirmed by the fact that, in the simulations, other grains of initially $20-30^\circ$ rotated Cube orientations developed Cube / β fibre components. The predictions for three of these grains are shown in Figure 5, including grain 045, providing good agreement with the experimental observations.



Note that the reorientations are not quite those expected of the classical Dillamore and Katoh model of Cube splitting for which initially ND rotated cube grains split up into transition bands composed of \pm RD rotated cube and cube segments. On the contrary, the original and important feature of the present work is the fact that the resulting components are not symmetrical.

Cold Plane Strain Compression

(a) Deformation. In a second experiment a sample of the same alloy was channel-die compressed at room temperature up to a strain of 2.3 (90% thickness reduction). A slice was then taken from near the centre on the RD/ND section and the same selected areas examined by EBSD before and after light annealing (microhardness indents of about 20 μm diameter were used to identify the areas). In this case, the much finer cold deformation microstructure required EBSD step sizes of 0.1 μm over areas of 150 x 300 μm .



Cube fragments could be clearly identified and possessed typical sizes of a few microns in length and about 1 μm thickness. They basically occurred in two locations: i) along grain boundaries between grains of the β fibre components particularly S and Cu (e.g. Fig 5a), and ii) as transgranular segments in a few specially oriented grains (e.g. Fig 5b). The latter could often be loosely described as TD rotated cube $\{011\}\langle 110\rangle$ grains in which apparent local shear had rotated banded elements back towards Cube. The origins of the intergranular Cube segments are less obvious. Firstly, they do not appear to have been clearly identified in previous studies and, secondly, models for their formation are not yet satisfactorily developed. However, we feel that the type of orientation fragmentation characterized above by the hot deformation tests can, in principle, be extended to the cold deformation process (but at a scale almost 2 orders of magnitude finer). The hot PSC simulations demonstrate that off-Cube grains can break up into Cube + S components which must have interfaces corresponding to grain boundaries.

(b) Annealing. The behaviour during annealing of the same cold deformed microstructure is illustrated by Figures 6 and 7 clearly showing that the residual Cube fragments can become Cube grains on annealing. However, this is not automatic in the sense of a rate of transformation of 100%.

Fig 6 shows that the intergranular cube fragments become Cube nuclei with a fairly high success rate, and in the absence of other nuclei orientations. On the other hand the transgranular fragments develop into Cube plus randomly oriented grains, and both appear to have about the same growth rates in these circumstances. A more detailed analysis of the efficiency of the nucleation sites shows that about 40% of the intergranular Cube fragments become true Cube grains as opposed to only 20% for the transgranular fragments. Moreover, the principal local orientation relations of the successful intergranular cube nucleation sites is usually close to the well-known 30-40° <111> corresponding to Cube-S misorientations.

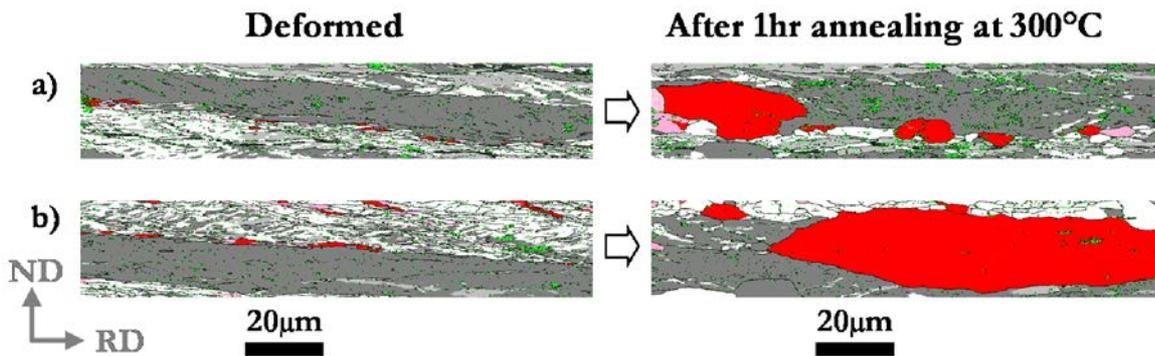


Fig 6 Two examples of intergranular Cube fragments developed at a strain of 2.3 and their evolution during annealing at 300°C into recrystallized Cube grains.

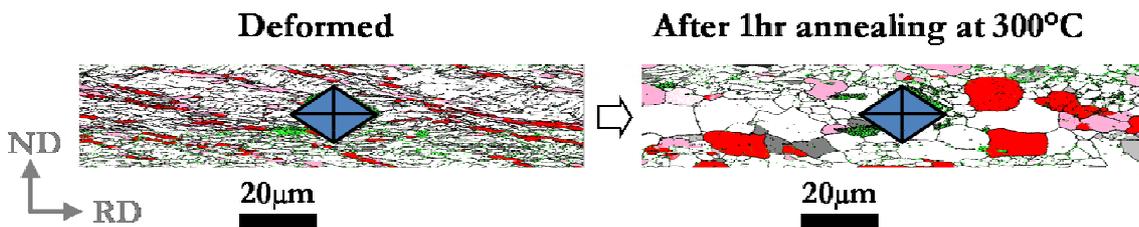


Fig 7 An example of transgranular Cube fragments after PSC and the evolution of the fragments and surrounding areas during annealing into recrystallized Cube grains plus non-Cube grains.

Concluding remarks

It is well known that the Cube recrystallization texture is strongly developed in pure fcc metals rolled to high strains then annealed. Apart from the case of Cube bands that are retained during hot rolling [12, 13] it is not immediately obvious how the Cube nuclei are retained, or formed, during plastic deformation to develop the Cube annealing texture. It is thought that they must exist in sites of very heterogeneous deformation, but the details are basically unknown. This work has therefore concentrated on some specific deformation structures that develop at large plastic strains in rolled aluminium, both at room and high temperatures, and their evolution during recrystallization annealing in the case of cold deformation. The deformation structures of great interest to recrystallization nucleation are those characterized by large orientation gradients that develop in certain grain orientations particularly original near-Cube grains.

The results of the hot deformation “microtexture tracking” study demonstrate that initially off-Cube grains can be deformed by plane strain compression to generate small quantities of Cube fragments paired with β fibre texture components such as the S and Cu orientations. As shown by the finite element crystal plasticity analysis, this behaviour is often observed for grains that are initially 20/30° off Cube. Moreover, by detailed EBSD experiments it is shown that during cold rolling to large reductions (90%) small micron-sized Cube fragments survive the rolling process, and are often located at the grain boundaries. These intergranular fragments are particularly efficient cube recrystallization nucleation sites since they frequently occur adjacent to S or Cu/S grains with which they possess a near 40° <111> orientation relation.

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