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Oriented growth during recrystallization revisited in three dimensions☆

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The two surfaces of a 40% cold-rolled tricrystal of aluminium were scratched to stimulate recrystallization nucleation. Serial sectioning combined with electron backscatter diffraction was used to characterize the nuclei in three dimensions. It was found that the largest nuclei have a 40°⟨1 1 1⟩ relationship to the matrix, but there are also many nuclei of this orientation relationship which do not grow to large sizes. It is shown that local variations in the deformation microstructure determine where preferential growth occurs.

Keywords: Tricrystal; Aluminium; Recrystallization; Electron backscatter diffraction (EBSD); Nucleation

Growth during recrystallization is determined by the orientation of the available nuclei (i.e. the nuclei that have formed) and by the deformation microstructure in which they grow. Barrett [1] was the first to suggest that grain boundary mobility depends on the misorientation across the boundary. The experimental verification of this idea was provided by the classical Beck experiment [2], in which growth of artificially stimulated nuclei into a lightly deformed single crystal was studied. Later several groups performed experiments of this type and for face-centred-cubic (fcc) metals it was generally found that nuclei with a misorientation of ~40° around a common ⟨1 1 1⟩ axis grew the fastest [2–6]. Also analysis of texture change during recrystallization tends to support a preferential 40°⟨1 1 1⟩ growth [7]. The magnitude of the preferential growth advantage was observed to depend on materials and process parameters such as purity and solute content [8–10] as well as annealing temperature [11,12]. The actual grain boundary plane was also found to be of importance, and Kohara et al. [13] and Parthasarathi and Beck [14] observed that tilt boundaries move faster than twist boundaries in fcc metals.

The aim of the present work is to revisit this classical work on preferential growth during recrystallization. The novel aspect of this work relates to two factors:

1. A large tricrystal is used instead of a single crystal. This allows for an investigation of the importance of the deformed microstructure morphology, including stored energy variations which exist between the three different crystals, on the growth of artificially stimulated nuclei under otherwise identical conditions in one experiment.
2. The investigation is done in three dimensions. By having the full three-dimensional (3-D) picture, possible pitfalls from the previous more limited two-dimensional (2-D) investigations are avoided. Compared to the texture investigations the present work benefits from knowing the 3-D shapes of the nuclei, and the direct relationship to the local deformed matrix.

It is found that both these factors are important for interpreting the preferential growth results.

A high-purity (99.99%) tricrystal of aluminium with the growth direction triple line along the crystallographic ⟨1 1 0⟩ axes was prepared by directional solidification. A sketch of the sample is shown in Figure 1a. The three crystals are labelled as A, B and C, and their orientations

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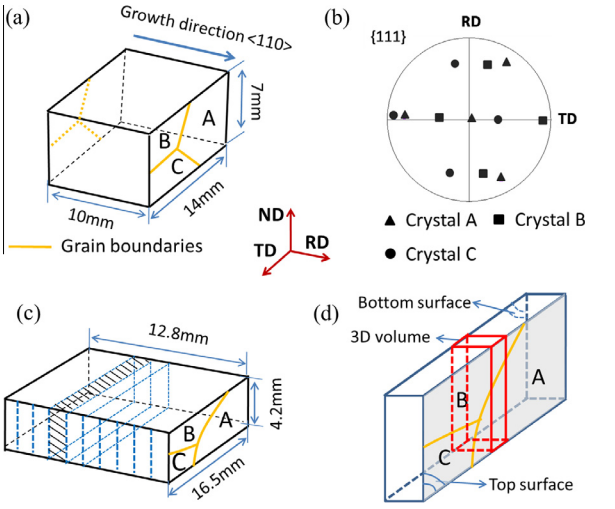

Figure 1. Sketches of the aluminium tricrystal sample: (a) the geometry of the tricrystal with the growth direction along the crystallographic ⟨1 1 0⟩ axes and (b) a {1 1 1} pole figure showing the orientations of the three crystals; (c) schematic drawing of rolled sample which was divided into ten slices; (d) the geometry of slice No. 4 as highlighted in (c). The "top surface" is indicated in grey and the sketch shows the details of the serial section volume (red box).

are ∼(1 1 –1)[1 0 1], ∼(1 3 –1)[1 0 1] and ∼(−3–1 1)[0 1 1], respectively (Fig. 1b), which correspond to "medium hard", "soft" and "soft" orientations according to Taylor factor calculations, (∼3.7 for A and ∼2.4 for B and C).

The tricrystal was cold rolled to 40% reduction with the triple line parallel to the rolling direction (RD). Rolling was conducted applying intermediate passes under conditions of $l/h \sim 2$, where l is the length of contact between the rolls and the specimen and h is the mean sample thickness. After rolling, the sample was sectioned perpendicular to the RD into ten 1 mm thick slices as shown in Figure 1c. The surfaces on the normal direction–transverse direction (ND–TD) planes of slice No. 4, i.e. top and bottom surfaces in Figure 1d, were rubbed with emery paper as randomly as possible, in order to stimulate artificial preferential nucleation. Slice No. 4 was then annealed at 300 °C for 20 min in an air furnace to initiate recrystallization. The top surface of slice No. 4 (see Fig. 1d) was electropolished, while the bottom surface was preserved as the originally scratched surface.

A sample from slice No. 4 near the triple junction (red box in Fig. 1d) was serial sectioned along the TD. In each section an area of 3 mm × 0.8 mm was characterized by electron backscatter diffraction (EBSD) with a step size of 2 μm using a Zeiss Supra 35 thermal field emission gun scanning electron microscope. A detailed description of the serial sectioning and alignment procedure can be found in the Supplementary material and in Refs. [15,16].

The partially recrystallized microstructure seen within a ND–RD section is shown in Figure 2. Focusing on the non-recrystallized microstructure, it is evident that crystal A is significantly subdivided, consisting of some narrow deformation bands with large misorientations (maximum misorientation angle ∼45°) near the grain boundary (between crystal A and B), a large transition band and wide deformation bands as marked in Figure 2 (see also Figs. S3 and S4 in the Supplementary material). The crystal B is far less subdivided, consisting of mainly diffuse transition bands. These differences in microstructures of the crystals will be described in more detail in Ref. [17].

As seen in Figure 2, the recrystallizing nuclei/grains are found mainly in crystal A, at the scratched surfaces and within the deformed matrix, in particular at the narrow deformation bands and transition bands. Only seven very small nuclei/grains are found in crystal B at the bottom scratched surface (see Figs. 2 and S3). These seven nuclei/grains have no apparent preferential orientation relationship to the matrix.

For crystal A we shall in this paper only analyse the nuclei stimulated by scratches which can best be seen on the bottom surface, which was not electropolished, and thus also the smallest nuclei are visible. Nuclei formed within the deformed matrix of crystal A will be investigated in Ref. [17]. For simplicity, in the following the

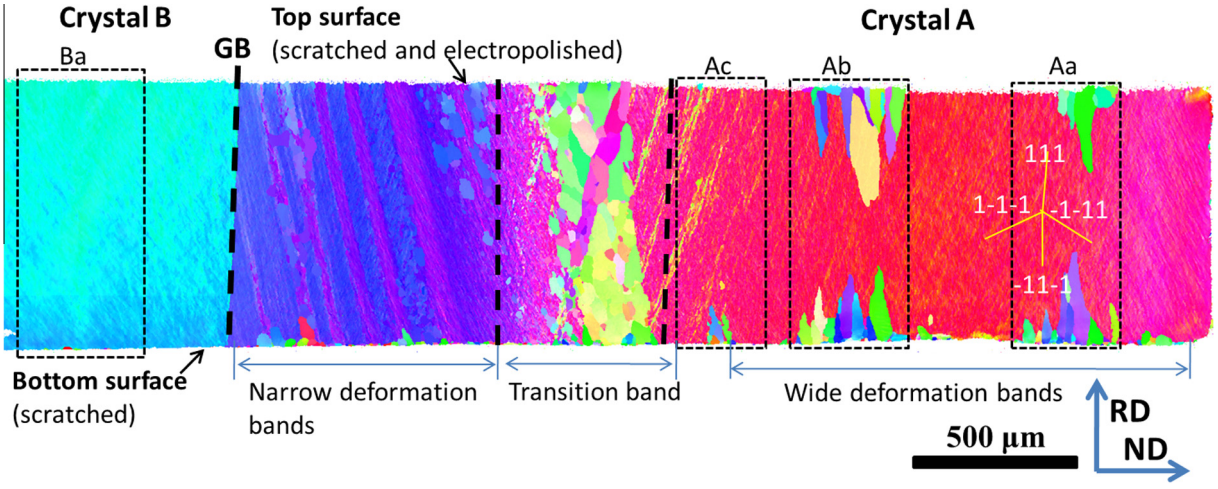

Figure 2. (a) EBSD map showing the partially recrystallized microstructure on the ND–RD section of the first sectioned layer. The colours represent the crystallographic direction of the TD (see Fig. S2).