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MONITORING GRAIN BOUNDARY MIGRATION DURING RECRYSTALLISATION USING TOPOTOMOGRAPHY

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ABSTRACT

The growth of a single grain during recrystallisation into a mildly deformed $\{001\}\langle 100 \rangle$ oriented single crystal has been monitored by synchrotron radiation using the topotomo technique. The formation and migration of individual facets is analysed using a new method which measures distances between grain boundary segments at different time steps along parallel lines normal to the facet plane. One facet is shown to move with a constant rate, while it remains planar and keeps the same boundary plane orientation. The formation of another facet, which is analysed in detail, reveals that first a planar boundary with a different orientation forms before it changes its boundary plane orientation into that of the final facet. It is argued that the local microstructural configuration in front of moving grain boundaries has a considerable influence on the kinetics of individual boundary segments and facets.

1. INTRODUCTION

Experimental techniques using synchrotron radiation have enabled non destructive characterisation of grains embedded in the bulk of the material (e.g. Schmidt, Nielsen, Gundlach, Margulies, Huang and Juul Jensen 2004, Ludwig, Lauridsen, Schmidt, Poulsen and Baruchel 2007). This has made it possible to obtain e.g. time resolved measurements of local grain boundary movements. Although the average behaviour of grain boundary segments

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during recrystallisation may be well described by the existing recrystallisation theory, it has been shown by earlier experiments (Schmidt et al. 2004) that the migration of individual boundary segments is far more complex than expected. Especially the role of the local deformation microstructure in front of a moving grain boundary is not yet well understood.

In order to study the local boundary migration during recrystallisation, it is chosen to monitor an individual grain during its growth into a deformed single crystal with such a non destructive 3D technique. Because of the growth of one grain within a deformed single crystal, all possible boundary plane orientations are present for one specific crystallographic misorientation. The single crystal is deformed by cold rolling, which results in fairly homogeneous deformation microstructures which are for the most important orientations well described in the literature.

2. EXPERIMENTAL SETUP

A single crystal of commercially pure aluminium (AA1050) with initial orientation $\{001\}\langle 100 \rangle$ (cube) has been deformed using conventional cold rolling to 30 % thickness reduction. A sample of 4.0 mm by 0.7 mm by 0.7 mm along the rolling, transverse and normal directions (RD-TD-ND) has been cut out of the larger cold rolled sample by spark cutting. In order to initiate recrystallisation at a specific location the same approach is followed as in the classical experiments of Beck, Sperry and Hu (1950). At one end of the specimen, an additional local deformation is imposed on one of the square TD-ND surfaces by a Vickers hardness indenter with an indentation load corresponding to 2 kg. The additional plastic deformation results in a locally higher stored energy just below the indentation. This position becomes the most likely place for recrystallisation nuclei to form and the zone affected by the indentation will be consumed first, before the nuclei grow into the deformation structure formed by solely cold rolling. If a moderate annealing treatment (depending on the orientation of the single crystal and the imposed deformation) is applied, only a few nuclei will consume most of the indentation zone and grow further into the more homogeneously deformed matrix. For the sample discussed in this paper the temperature during nucleation and initial growth has been 270 °C.

The growth during recrystallisation of an individual grain into the deformed matrix has been monitored by X-ray diffraction using the topotomo technique (Ludwig et al. 2007). The experiment has been carried out at beamline ID-11 at the European Synchrotron Radiation Facility. A monochromatic beam of dimensions 0.8 mm by 0.8 mm and an energy of 30 keV has been obtained by using a double Laue-Laue monochromator.

The topotomo technique offers the possibility to monitor an undeformed single grain with a high spatial and time resolution. The principle of the technique is illustrated in Fig. 1. The sample is mounted on a set of three tilting stages and a rotation stage. By applying a specific combinations of tilts, the rotation axis of the rotation stage can be made collinear to a selected diffraction vector (g -vector) of the grain of interest. The combination of the two orthogonal tilts in between the rotation stage and the sample ensures the alignment of the rotation axis and the g -vector, while a base tilt below the rotation stage is needed to keep the grain in a diffraction condition for the particular g -vector. For the tilted position, the diffraction condition remains satisfied for all possible rotations of the rotation stage. The shape of the diffraction spot corresponds to the projection of the 3D grain shape. From a number of projections, obtained by rotating the sample, a tomographic reconstruction of the 3D grain shape can be calculated. Although it is also possible to use the contrast difference in the directly transmitted beam, it has been chosen to work only with the diffracted beam, because of its much higher signal to noise ratio. In order to make the 3D reconstruction, 20 different projections, 18° apart, have been obtained for each time step.

Monitoring grain boundary movements during recrystallisation using topotomography

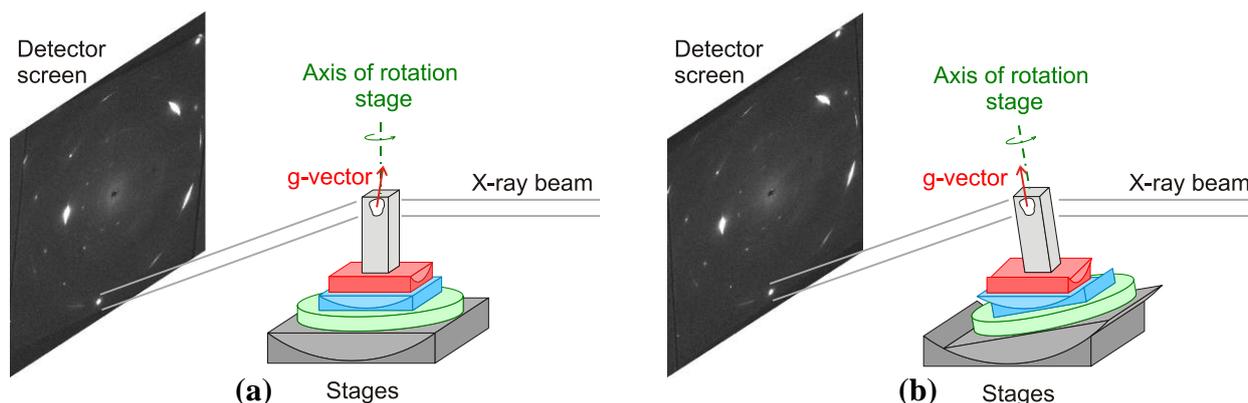


Fig. 1. Sketch of the experimental setup of the topotomo technique. (a) All stages in 0° tilt position. The g-vector for one diffraction spot of a particular grain is indicated. (b) A double tilt is applied to get the rotation axis of the rotation stage parallel to the g-vector.

The sample was mounted on the stages with the indented zone on top. A hot air blower furnace was used as the heating device. After 490 s of initial heat treatment at 270°C the largest nucleus was singled out to monitor its further growth into the deformed matrix with the topotomo technique. After recording a first snapshot, the heat treatment was continued first at 290°C and later at 300°C . The annealing treatment has been interrupted at regular intervals of 135 s, in order to cool down to room temperature and to record a snapshot of the current shape of the grain. It is difficult to assess how the intermediate cooling and heating cycles might have influenced the recrystallisation behaviour. Out of the observed stable growth rate of the main facet (cfr. section 3), it is however likely that the interruptions did not have a large effect on the recrystallisation behaviour.

For some parts of the boundary the tomographic reconstructions of the later snapshots unfortunately do not result in a shape determination with as a good a resolution as the earlier snapshots. This is indicated by the much deeper wrinkling of the boundary and the back and forward movement of some of the boundary segments between successive reconstructions. The reason for this may be due to a small misorientation within the grain for which the integration range of the base tilt was not sufficiently corrected. A correction routine has been applied on the intensity data of the reconstructions in order to determine the grain boundary with a single threshold value.

3. RESULTS AND DISCUSSION

In Fig. 2, 3D reconstructions of the grain shape are shown at different time steps during the growth of the grain. The successive grain shapes show that the growth occurs both by a constant migration of flat boundary segments (which here are called facets) and by irregular shooting out of boundary segments forming protrusions which are later caught up by the neighbouring segments (Schmidt et al. 2004). In this particular case most of the growth happens by migration of a single facet in a direction almost parallel to the rolling direction (RD).

To analyse the formation and movement of facets, a new analysis method is applied, which is illustrated in Fig. 3. At the final snapshot, the boundary segment that forms a facet is characterised by a number of points lying on the facet. The facet plane is calculated by means of a least squares fitting procedure through the chosen points. It is possible to define a new reference frame $x_f - y_f - z_f$ on the boundary facet plane, where the direction of z_f is normal to the plane. A set of parallel lines is then defined through the points in the direction of x_f . The

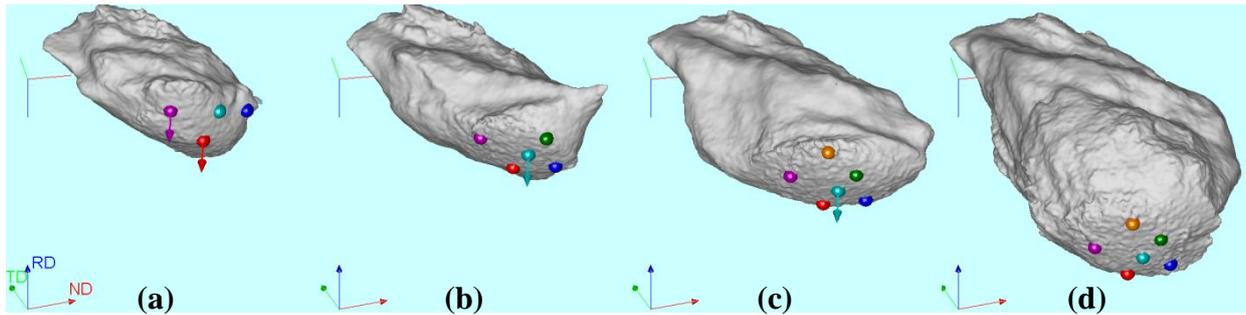


Fig. 2. Snapshots from the 3D reconstructed grain at different times during its growth, after (a) 490 s, (b) 1044 s, (c) 1584 s and (d) 2124 s. The coloured dots indicate the intersection points used to define the facet in (d). For an explanation of the arrows see text.

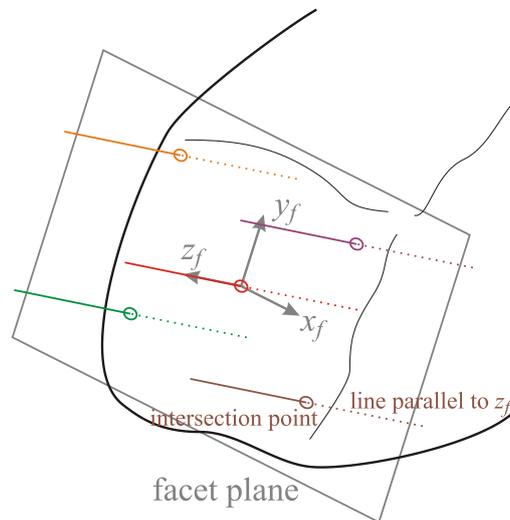


Fig. 3. Schematic illustration of the method applied to analyse the formation and growth of grain boundary facets.

intersection points of this fixed set of lines with the grain boundary surfaces of the previous snapshots are furthermore calculated. The successive distances of these points to their final position along the lines describe how individual segments of the facet migrate towards their final position on the facet. Another method has been applied before to characterise grain boundary facets (Rowenhorst, Gupta, Feng and Spanos 2006, Juul Jensen, Rowenhorst and Schmidt 2007). An advantage of the new method is that it can be directly applied on the reconstructed data, without the necessity of applying a smoothing filter. In this new method however, the facets have to be identified by the user and do not automatically follow from the data analysis.

The intersection points of the boundary surface with the set of parallel lines, as obtained by the method described in the previous paragraph, are plotted on the snapshots in Fig. 2. The direction of the lines is indicated by small arrows on some of the points. The distances of the boundary segments at different snapshots along their lines to the position of the boundary surface in the final snapshot are given in Fig. 4. Since the parallel lines do not necessarily intersect an early grain shape, some intersection points will only appear at a later point into the growth. The curves of Fig. 4 show how the facet migrates as well as visualise how boundary segments become part of the facet.

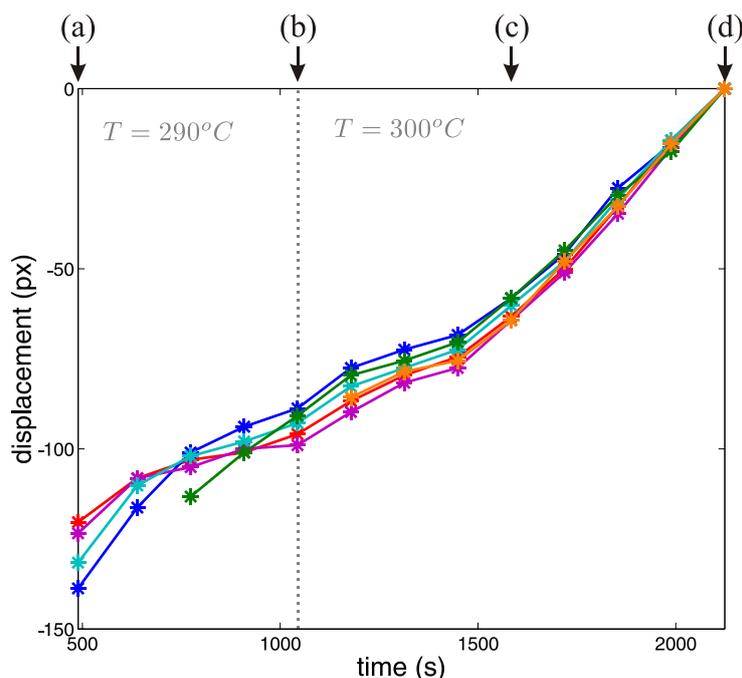


Fig. 4. Displacement of grain boundary segments forming a facet with reference to their final position along several parallel spatial lines as indicated in Fig. 2. The timings of the snapshots of Fig. 2 are indicated on top of the figure.

The displacement curves (Fig. 4) along the red and purple parallel lines are almost coincident, illustrating that the facet already exists between these 2 points from the start of the heat treatment. The fact that these curves fall right on top of each other shows that the facet during its migration remains planar and keeps its orientation. The boundary segments at the light and dark blue intersection points are initially somewhat behind compared to the red and purple points, and are therefore not a part of the facet in the initial snapshots (cfr. Fig. 2 (a)). They however catch up early on in the heat treatment. This happens first for the light blue point (at snapshot 3), followed by the dark blue one (at snapshot 4). This demonstrates that the facet gradually extends itself in its lateral direction. From the moment of catching up, the displacement curves almost coincide, which means that the larger facet remains flat during the further growth of the grain. The lateral growth can be either accommodated by the catching up of boundary segments which are induced by protrusions or by the extension of the facet because of the growth of another facet into another direction. The boundary segment along the green spatial line is an example of the former mechanism (cfr. Fig. 2 (b)), while that along the orange spatial line is a result of the latter (cfr. Fig. 2 (c)).

The facet has a constant migration rate for extended periods of time, but this rate changes twice. The first change in migration rate coincides with the increase of the temperature (after snapshot (b)), while the second change (just before snapshot (c)) is not related to any known change in the experimental environment. A possible reason might be a change in the crystallography of the deformation microstructure, which would change the mobility of the facet. Since it is expected that the grain at that position already has grown out of the zone which is affected by the hardness indent, this inhomogeneity should come from the cold rolling process. A further characterisation and analysis of the substructure in front of the facet will have to be carried out, in order to obtain indications on this issue. A reorganisation of the substructure driven by recovery processes could be another possible explanation for the increase in the migration rate. Such a reorganisation reduces on the one hand the overall stored energy and therefore diminishes the driving force for boundary migration, but on the other hand it could also strongly

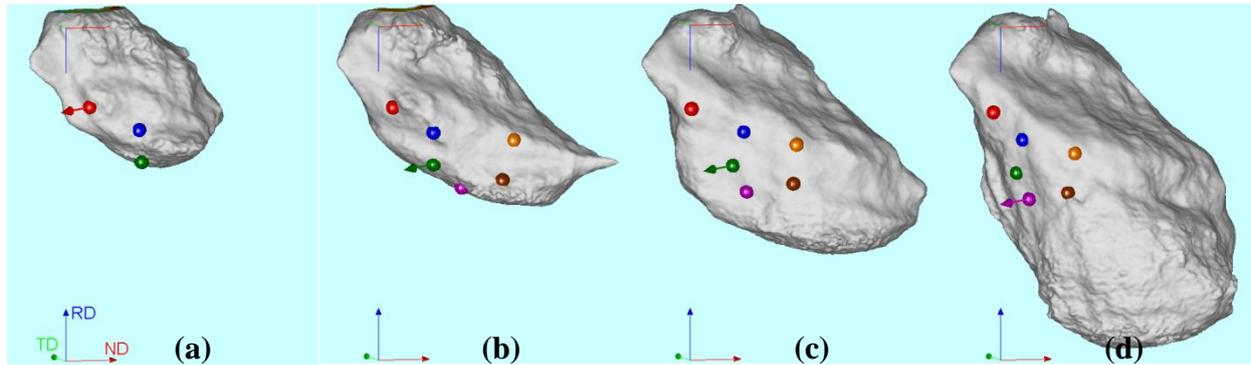


Fig. 5. Snapshots from the 3D reconstructed grain at different times during its growth, after (a) 490 s, (b) 1044 s, (c) 1584 s and (d) 2124 s. These are the same snapshots as in Fig. 2, but viewed from a different angle. The coloured dots indicate the intersection points with a set of parallel lines following the emergence of the second facet.

improve the mobility of this particular facet, leading to an overall increase of its migration rate. However, the rather abrupt increase in the migration rate does not seem to correspond with such an explanation.

The analysis with the parallel lines is also used to study how a facet can form. For this purpose a facet is selected which has a normal almost perpendicular to RD. The same snapshots as shown in Fig. 2, but from another view point, are shown in Fig. 5. This facet forms during the presented time span. It migrates at a constant speed in the direction normal to the facet plane in subsequent snapshots (not presented in this paper). Fig. 6 shows the displacement curves of the intersection points along the parallel lines during the formation of the facet.

The red line follows the evolution of a boundary segment that is already sticking out of the boundary surface in the snapshot of the initial grain shape. This boundary segment slowly migrates at a fairly constant rate until it remains stationary for the last four snapshots. The boundary segments along the dark blue and green spatial lines follow a very similar growth path as the boundary segment along the red line, but from snapshot (c) onwards they speed up drastically and catch up with the red segment after two more snapshots to become momentarily stationary as well. The boundary segment along the purple line is formed at a later stage. Its formation can be attributed to the migration of the facet discussed in Figs. 2 and 4. It first catches up with the boundary segment along the green line. Afterwards it proceeds concurrently with the green and dark blue boundary segments. The boundary segments along the orange and brown spatial lines are formed at a somewhat later stage and follow a similar growth path, ending up at the facet in the very last snapshot. As can be seen in Fig. 5, all intersection points along the different lines fall at snapshot (c) on the same large planar boundary segment which has a different orientation from the final facet. This segment only gradually changes its orientation towards the growth direction of the facet between snapshots (c) and (d). From snapshot (d) onwards the then fully formed facet, migrates in a similar way as the facet discussed in Figs. 2 and 4.

The formation of this facet is quite peculiar because it happens in two stages. First the boundary segments form an intermediate planar boundary by catching up with a part that is already sticking out of the original boundary surface (probably a protrusion). The second stage is the gradual change of this segment towards a new orientation, where a part of the boundary (segment at the red line) remains stationary. The direction of the grain boundary normal changes by about 21° . A possible explanation for this change might be that the newly oriented

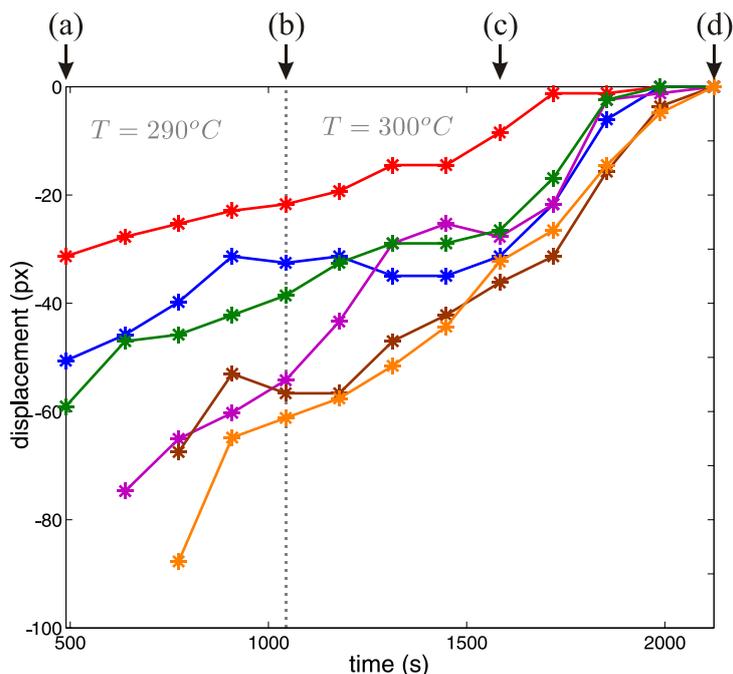


Fig. 6. Displacement of the grain boundary with reference to its final position along a set of parallel spatial lines indicated in Fig. 5. The timings of the snapshots of Fig. 5 are indicated on top of the figure.

boundary plane results in a lower energy state, but further analysis of the grain boundary misorientation over the facet has to be carried to confirm this. This does however not explain why the initial planar boundary (cfr. Fig. 5 (c)) forms in the first place. The local microstructural arrangement in front of the moving boundary is most likely responsible for the specific way in which this facet gets formed.

The observations reported in this paper support the earlier findings based on the analysis of a similar experiment using 3DXRD (Schmidt et al. 2004, Juul Jensen and Schmidt 2009). The growth of a grain during recrystallisation happens in a more irregular fashion than assumed in classical recrystallisation theories. Grain boundaries do not generally migrate at a constant rate and local protrusions often precede the migration of a certain segment. The formation of these protrusions is most likely caused by the local inhomogeneities in the deformation microstructure on the microscale. Facets which migrate at a more constant rate can, however, be found as well and are the dominant mechanism for the growth of the grain under investigation in this paper.

4. CONCLUSIONS

The evolution of the 3D shape of a single growing grain in a deformed matrix of a cube oriented aluminium single crystal during recrystallisation has been characterised by synchrotron radiation using the topotomo technique. A new analysis method to study the migration and the formation of grain boundary facets has been presented.

The analysis of a growing facet shows that during its migration it remains planar and keeps the orientation of its normal direction. Moreover, it moves with a constant rate. The facet can during its movement also become extended into its lateral directions. The formation of a facet in a two stage process is observed as well. First a planar boundary is formed, which gradually changes its orientation to turn into a facet that migrates along its normal direction.

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