Direct Observation of Grain Boundary Migration during Recrystallization within the Bulk of a Moderately Deformed Aluminium Single Crystal

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A single grain growing in the bulk of a mildly deformed (30% thickness reduction through cold rolling) aluminium single crystal with an [001]⟨100⟩ orientation (Cube orientation), is monitored during recrystallization with synchrotron radiation using topo-tomography. The formation and migration of planar boundary segments (facets) are analyzed using a method that determines the displacements of local boundary segments along parallel lines perpendicular to the facet plane. Facets are observed to form after a certain annealing time. They migrate at a constant rate for extended periods of time and remain planar during their migration. A change in the migration rate for one facet has been observed which is not related to changes in the experimental conditions and is most likely to be driven by the changes in grain orientation and/or the local deformation microstructure. The crystallography of the analyzed facets is not closely related to any crystallographic {111} plane of neither the growing grain nor the disappearing deformed matrix. DOI:10.2320/matertrans.M2013227

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1. Introduction

A trend which recently has received much attention is the ambition to combine materials science with materials engineering and design. The hope is thereby to speed up new innovations, for example enabling a faster route for a new alloy from idea to practical use in engineering components via predicting changes in mechanical properties based on compositional variations. A way towards such goals is via the development of advanced materials models and their implementation for engineering and design purposes. Such models are, however, not better than the data and physical assumptions on which they are based. Whereas many materials models focus on the average behavior or property of a given material, modern experimental techniques have paved the way for new advanced characterizations of local phenomena determined by the local structural variations within the materials. The aim of the present work is to contribute to the latter by a detailed direct investigation of boundary migration during recrystallization of a weakly deformed single crystal by synchrotron X-rays.

During recrystallization of a deformed material, the deformed microstructure is replaced by a new one containing a lower stored energy. This happens through the formation of almost defect-free nuclei whose boundaries subsequently migrate into the deformed matrix, hereby consuming the deformation structure and releasing stored energy. Recrystallization drastically alters the microstructure and may therefore significantly change the mechanical properties of the material. It is one of the basic treatments in the thermo-mechanical processing applied during the production of almost all metals used for a wide range of applications. Despite its technological importance, many fundamental aspects of recrystallization, in particular on the local scale, are not yet well characterized due to limitations of experimental techniques.

The migration rate \( v \) of boundary segments during recrystallization is usually expressed as:

\[
 v = MF,
\]

where \( M \) is the mobility of the boundary segment and \( F \) represents the driving force. The mobility is thought to be a function of the misorientation across the migrating grain boundary and the crystallography of the boundary plane. The driving force is considered to be mainly dependent on the stored energy in the deformation structure ahead of the migrating boundary, although the boundary curvature near local protrusions and retrusions on the migrating boundary has to be considered as well in some cases. 

For commonly used metals like low carbon steel, aluminium and copper, the stored energy is distributed heterogeneously on a local micrometer scale, because dislocations stored during deformation group themselves into dislocation walls or boundaries. Due to the heterogeneous distribution of the stored energy, conventional statistical analysis, based on an assumption of homogeneous growth, might be misleading. In order to better understand recrystallization it is necessary to study the phenomenon of grain boundary kinetics on the local (micrometer) scale.

One way to gather local data on grain boundary migration is by observing the traces of grain boundaries at the sample surface during heat treatment. This does not, however,
give the complete picture, because the orientation of the boundary planes cannot be fully characterized. The presence of the surface might also strongly affect the migration of the boundary segments.\textsuperscript{10} so it is not clear how representative results obtained with these surface techniques are for the bulk recrystallization behavior.

Experimental techniques using synchrotron radiation have enabled the characterization of grains embedded in the bulk of the material non-destructively. This makes it possible to obtain e.g., time resolved measurements of local grain boundary movements. It has been shown by earlier experiments\textsuperscript{11-13} that the migration of boundary segments is far more complex than expected. Boundaries generally do not migrate with a constant rate, but move by a stop-go motion. Often small boundary segments advance faster than the neighboring parts of the boundary for a short time thus forming protrusions. Some facets that are moving with a constant rate have also been reported.\textsuperscript{12}

In the present article, the growth of an individual grain during recrystallization in a deformed single crystal is monitored with synchrotron radiation using topo-tomography.\textsuperscript{14} The aim is to obtain directly measured growth rates of different grain boundary segments migrating in the bulk of the material. A deformed single crystal is used as the matrix material in order to have a similar microstructure throughout the growth process into which the different grain boundary segments migrate. Because the growth of one grain within a deformed single crystal is monitored, all possible boundary plane orientations are realized for one specific crystallographic misorientation. Topo-tomography is used to monitor the shape of the growing grain. In this manner, the spatial resolution is improved compared to earlier measurements of grain boundary migration during recrystallization in which 3DXRD was used.\textsuperscript{11} Also, a differently oriented single crystal is used which has a different deformation matrix for the recrystallizing grain to grow into. Based on the diffraction data the crystallography of migrating boundary facets will be analyzed and discussed.

2. Experimental Setup

2.1 Sample
A commercially pure aluminium (AA1050) single crystal with initial Cube orientation (001)[100] has been cold rolled to 30\% thickness reduction in a single rolling pass. Out of the rolled sheet, 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 by spark cutting. Orientation mapping by electron backscattered diffraction (EBSD) was conducted on a longitudinal (RD-ND) section on an area of 350 µm by 225 µm, using a Zeiss Supra 35 SEM equipped with a field emission gun and a Nordlys2 detector at an accelerating voltage of 20 kV. Channel5 software (HKL technology) was used to record and index the orientations. It is well known that the Cube orientation is unstable during rolling deformation\textsuperscript{15-22} and that it subdivides macroscopically. The present investigation of a relatively small 350 × 225 µm\textsuperscript{2} area is thus expected to only give part of the bigger picture. Figure 1 shows a disorientation contrast plot\textsuperscript{23} of the measured area. For every data point the disorientation from the average orientation of the whole map is calculated. The disorientation vector with the direction of the disorientation axis and the length of the disorientation angle of each data point is mapped into red-green-blue color space where half grey corresponds to a 0° disorientation and full intensity of a component to a 5° component of the disorientation vector. The red-green-blue components correspond to RD, TD and ND components of the disorientation vector expressed in the sample reference frame. A banded substructure is clearly revealed in Fig. 1. The predominant color tints are light green and purple (perfect TD rotations are either halfway between green and white (= light green) or halfway between magenta and black (= purple)) which show that the orientation spread is mostly due to disorientations with the vector close to the TD axis. The average disorientation angle is 1.9°.

By earlier SEM and TEM investigations\textsuperscript{20-22} of a 30\% cold rolled Cube oriented single crystal, it was found that the crystal broke up into 4 macroscopic bands parallel to the RD-TD plane with widths of several hundred micrometers each. The disorientations from one macroscopic band to its neighbors were high, between 10 and 25°. Within the macroscopic bands, the deformation microstructure is subdivided by one set of dislocation boundaries with relatively small disorientations (<5°). In the transition bands between adjacent macroscopic bands, two sets of boundaries are observed with similar disorientations to that shown in the micrograph in Fig. 1.

To initiate recrystallization at a specific location for the X-ray measurements, an additional local deformation is imposed on one of the two square TD-ND surfaces of the sample by a Vickers hardness indenter with an indentation load of 2 kg. This has a similar effect as scratching the sample used in the classical experiments of Beck and co-workers.\textsuperscript{24} The additional plastic deformation just below the indentation results locally in a higher stored energy. The zone affected by the indentation therefore becomes the most likely place for recrystallization nuclei to form. This zone will be consumed first, before the nuclei grow into the deformation structure formed solely by cold rolling.

2.2 Topo-tomography
With topo-tomography\textsuperscript{14} it is possible to monitor the 3D shape of a single undeformed grain with a high spatial resolution using high energy X-rays. Topo-tomography obtains the 3D shape of a grain by measuring different projections of one particular diffraction spot while the grain is rotated around the diffraction vector (g-vector, see Fig. 2) of that diffraction spot. The technique requires that the sample is mounted on a set of three tilting stages and a rotation stage. The purpose of the tilting stages is illustrated in Fig. 2. One diffraction spot of the grain of interest is brought into permanent diffraction condition by making the rotation axis of the rotation stage collinear to the g-vector of the diffraction spot. The combination of the two orthogonal tilts in between the rotation stage and the sample is needed to ensure alignment of the rotation axis and the g-vector, while the tilting stage below the rotation stage (base tilt) is needed to maintain the angle between the g-vector and the incoming beam and therefore the diffraction condition. In the tomo position (cf. Fig. 2(b)), the diffraction condition
remains satisfied for all possible rotations of the rotation stage. Each image of the diffraction spot seen on the near field detector corresponds to a projection of the 3D intensity distribution of the diffracted beam which is a function of the precise 3D shape of the grain. The shape and the intensity distribution of the diffraction spot therefore reflect the 3D grain shape. Different projections of the same diffraction spot, obtained by rotating the sample, can be used to make a tomographic reconstruction of the 3D intensity distribution caused by the diffraction of the incoming beam at the crystal lattice of the grain. This results in a reconstruction of the 3D shape of a single grain embedded in the bulk of a sample, obtained in a non-destructive way.

It would also be possible to use the contrast difference in the directly transmitted beam, but as these images have a much lower signal to noise ratio, it has been chosen to work only with the diffracted beam in the present work.

2.3 Measurement

The measurement has been carried out at beamline ID-11 at the European Synchrotron Radiation Facility. A double Laue-Laue monochromator was used to obtain a monochromatic beam of dimensions 0.8 mm by 0.8 mm and an energy of 30 keV. With this energy and beam dimensions the entire sample was illuminated.

The sample was mounted on a series of tilt and rotation stages (cf. section 2.2) with the indented zone on top. A hot air blower furnace was used as heating device. To initiate nucleation and initial growth, the sample was heated to a temperature of 270°C for 489 s. The largest nucleus was found using conventional synchrotron diffraction with the sample in the upright position (cf. Fig. 2(a)). Its further growth into the deformed matrix was monitored with topo-tomography (cf. section 2.2). For simplicity, this growing nucleus will in the following be referred to as a grain because no strict definition is available to express when a nucleus becomes a grain.

The 3D shape of the grain was reconstructed based on 20 different projections each 18° apart obtained from a 311 diffraction peak. In order to account for a possible small orientation spread within the grain, each projection of the diffraction peak has been recorded as the integrated intensity over a 0.3° interval of the base tilt. For these measurements, a near field detector was used.

Diffraction data were obtained on a far field detector (at a distance of 20 cm from the sample) containing the full 111, 200, 220, 311 and 222 diffraction rings, during a 360° degree scan of the rotation stage with a step size of 1°. This was done before and after the heat treatment with the sample in upright position (cf. Fig. 2(a)) and enables to analyze the crystallographic orientation of the matrix and the growing grain.

2.4 Annealing treatment

After recording the 20 different projections of the 3D shape in the topo-tomo position (further called a snapshot) of the initial shape of the growing grain, the heat treatment was continued first at 290°C and later at 300°C. Since heating could not be continued during the recording of snapshots with topo-tomography, the heat treatment was interrupted at regular intervals of 135 s, during which the sample was cooled down to room temperature and a snapshot of the current grain shape was recorded. During heating, the evolution of a single projection was however monitored by saving a detector image every 12 s. Whether the intermediate cooling and heating cycles have influenced the recrystallization behavior, is difficult to assess but as shall be discussed...
later, it is not expected to radically change the typical bulk growth. In total 13 snapshots have been recorded: one initial condition after 489 s of heating at 270°C, 4 snapshots during the heat treatment at 290°C and 8 snapshots during the treatment at 300°C.

### 2.5 3D Reconstruction

The tomographic reconstructions are done using a standard arithmetic reconstruction (ART) algorithm. One complication of this otherwise standard ART reconstruction was that the intensity of the lower part of the grain was observed to be lower in the raw data of the diffraction spots (see Fig. 3). This indicates that the reason for this lower intensity reflects a small orientation change within the sample. A possible explanation might be that a small disorientation forms within the grain during its growth, which the integration range of the base tilt of 0.3° was not sufficient to capture.

The difference in intensity makes it impossible to define the grain boundary with a single threshold value for the entire whole grain. However, in spite of this correction, the reconstruction of the lower part of the boundary suffers from a lower signal to noise ratio in the raw data, which means that the obtained boundary in the reconstructions of the lower parts does not have as good a resolution as the upper parts of the grain. Indications of this are deeper wrinkling of the boundary and the back and forward movement of some of the boundary segments between successive reconstructions. These effects are considered artifacts and shall not be analyzed further.

### 3. Results

#### 3.1 Overall growth and crystallographic orientations

The individual snapshots of the reconstructed 3D grain shape are shown in Fig. 4, from which the overall growth can be analyzed. Similar characteristic features as found in the earlier experiment are observed for this grain, although it is growing into a different deformed matrix: most boundary segments migrate in a stop-go fashion and several protrusions can be found. However, different from the earlier experiment is that most of the growth of the grain happens through the migration of one planar boundary segment (facet) in the direction almost parallel to RD. Also a second facet is observed to form late during the annealing and this second facet moves in a direction almost perpendicular to RD.

The average orientations of the growing grain and the deformed matrix are represented in different parameterizations in Table 1. The disorientation between the two is shown in Fig. 4, from which the overall growth can be calculated in Fig. 7.

### Table 1 Crystallographic orientation of the growing grain and average orientation of the matrix.

<table>
<thead>
<tr>
<th>Euler angles</th>
<th>G</th>
<th>Miller indices</th>
</tr>
</thead>
<tbody>
<tr>
<td>Growing grain</td>
<td>133.2° 46.4° 15.9°</td>
<td>[−0.80, 0.57, 0.20], [−0.30, −0.65, 0.70], [0.53, 0.50, 0.69]</td>
</tr>
<tr>
<td>Matrix</td>
<td>206.5° 13.6° 327.7°</td>
<td>[−0.99, 0.09, −0.13], [−0.11, −0.97, 0.20], [−0.11, 0.21, 0.97]</td>
</tr>
</tbody>
</table>

### Table 2 Disorientation between the growing grain and the matrix.

<table>
<thead>
<tr>
<th>Euler angles</th>
<th>G</th>
<th>Axis and angle</th>
</tr>
</thead>
<tbody>
<tr>
<td>Disorientation</td>
<td>145.2° 44.0° 234.4°</td>
<td>[0.81, 0.15, −0.56], [−0.43, 0.81, −0.40], [0.40, 0.57, 0.72]</td>
</tr>
</tbody>
</table>

#### Fig. 3 Images of the same diffraction spot at different time steps. (a) 489 s, (b) 1044 s, (c) 1449 s, (d) 1719 s, (e) 1921 s, (f) 2124 s. The dotted line indicates the position along which the growth rate is calculated in Fig. 7.
3.2 Facets

3.2.1 Method

The main focus of the further data analysis will lie on the characterization of the two facets. To analyze the movement of the boundary segments making up these facets a method\(^{(25)}\) is used which is schematically illustrated in Fig. 5. At a certain reference position when the facet has considerable lateral dimensions (typically one of the later snapshots) a number of points are placed at different positions on the facet. The normal to the facet plane can be approximated by the normal to the plane obtained by least square fitting through the chosen points. Through the points on the facet a set of lines is then defined parallel to the normal of the facet plane (cf. Fig. 5(a)). For all previous and subsequent snapshots the points where the parallel lines intersect with the grain boundary are obtained and the distance from these points to the corresponding reference points is calculated (cf. Fig. 5(b)). The change of these distances along the respective parallel lines with time characterizes the migration of the facet.

Fig. 4 Snapshots of the reconstructed 3D grain at different time steps during its growth. The length of the legs of the tripod indicating the reference frame is 42 µm. Colored dots indicate the positions where a set of parallel lines intersect the grain, the arrows indicate the direction of the parallel lines. This direction is parallel to the normal of the plane derived by least square fitting through the intersection points at snapshot (j). The intersection points and corresponding parallel lines are selected to illustrate the formation and movement of a facet growing in a direction almost parallel to RD.

(a) 489 s  (b) 639 s  (c) 774 s  (d) 909 s
(e) 1044 s  (f) 1179 s  (g) 1314 s  (h) 1449 s
(i) 1584 s  (j) 1719 s  (k) 1854 s  (l) 1989 s
(m) 2124 s

Fig. 5 Schematic illustration of the method applied to analyze the formation and growth of grain boundary facets. (a) Defining the facet plane and the parallel lines at a reference position. (b) Calculating the distance along the line to intersection points with earlier snapshots.
3.2.2 Facet number 1

In Fig. 4, the positions of the intersection points, which trace the evolution of the facet that moves along a direction close to RD, are indicated. The facet plane and the parallel lines are defined based on the reconstruction recorded after 1719 s of heat treatment (cf. Fig. 4(j)). The obtained normal direction corresponds to [0.996 0.090 0.025] in the RD-TD-ND reference frame, which is approx. 5° from RD. The direction of the parallel lines is indicated with arrows for some of the intersection points (see Fig. 4). Not all of the parallel lines do necessarily intersect the grain at the shortest annealing times, which implies that some intersection points appear only later in the growth.

In Fig. 6 the displacements of the intersection points along the parallel lines are shown. The curves illustrate how the facet is migrating as well as how individual boundary segments become a part of the facet. The fact that the curves for the different intersection points coincide from the fifth snapshot onwards demonstrates that the facet remains planar from this time on and keeps its orientation throughout the heat treatment at 300°C. During the growth, the red and purple curves are coinciding from the beginning (see Fig. 6), which means that the facet was already present in the first recorded snapshot. The larger distances to the reference positions of the blue, cyan and green intersection points compared to those of the red and purple points in the initial snapshots indicate that the boundary segments at these points are initially not a part of the facet. These boundary segments gradually catch up with the facet and in this way extend the lateral dimensions of the facet.

The growth rate of the facet can be deduced from the slope of the curves. The growth rate of the facet remains constant for extended periods of time. From the second to fifth snapshot (290°C), the migrating rate of the facet equals 0.04 µm/s. With the increase of temperature to 300°C, the migration rate increases to 0.07 µm/s. From the snapshot at 1449 s onwards the rate increases again to 0.15 µm/s and this second increase in migration rate is not associated with any temperature increase.

As the second increase in growth rate almost coincides with the point when the threshold level is changed in the data analysis, the second increase in the growth rate is checked and confirmed based on the analysis of the unprocessed diffraction spot images recorded during the heat treatment (cf. Fig. 3). These images have been obtained during the heat treatments in between the recordings of the different snapshots. During each heat treatment at least 10 images have been measured, resulting in a time resolution with an order of magnitude higher than that of the 3D reconstructions. Since the previously discussed facet migrates along the rolling direction, it can be found at the bottom of the diffraction spot. The displacement of this bottom part is characterized by the distance from the first pixel along a vertical line (indicated in Fig. 3) above a certain threshold value to a reference position (the position in the first analyzed image). The displacement curves for different values of the threshold are shown in Fig. 7. For the lower threshold values, displacement curves are obtained which exactly match the displacement curves in Fig. 6 (for overlay curve see Fig. A1). The increase in the growth rate at about 1500 s is present in all curves and is thus not an artifact of the chosen threshold for the full 3D reconstruction of the grain but must be considered as a real effect.

3.2.3 Facet number 2

The formation of the second facet is analyzed using the same method as before. A number of 3D reconstructions, seen from a different viewing angle as in Fig. 4, is shown in Fig. 8. The facet is characterized by a number of points placed on the facet in the final snapshot. To avoid problems with the lower spatial resolution of the reconstruction in the bottom part of the grain, all points are selected in the upper half of the facet. The direction of the facet plane normal is parallel to [0.056 −0.595 0.802] in the RD-TD-ND reference frame. Figure 9 shows the displacement curves of the boundary segments finally making up the facet at about 1989 s of annealing. The displacement curves indicate that the boundary segment growing along the red parallel line is already at the bottom part of the grain, all points are selected in the upper half of the facet. The direction of the facet plane normal is parallel to [0.056 −0.595 0.802] in the RD-TD-ND reference frame. The growth rate of the facet can be deduced from the slope of the curves.

The relative positions of the intersection points on the boundary surface of the snapshots as shown in Fig. 8 combined with the displacement curves of Fig. 9, show that the facet forms first on one plane and then rotates to a second one. The initial facet slowly migrates at a constant rate of around 0.03 µm/s along the normal of the final facet. At 1719 s the boundary segment represented by the red line comes to a complete halt, and a new facet with a different orientation is formed. This happens by the fast 0.13 µm/s migration of the rest of the segments until all segments form the new facet. In further snapshots (at a temperature of 310°C, not included in this paper), the facet moves with a constant rate along its new normal direction.

3.2.4 Crystallography of facet planes

In Table 3, the crystallographic orientation of the discussed facet planes is expressed in the crystallographic reference frames of the growing grain, of the deformed matrix and of the intermediate lattice of the grain boundary. In none of the reference frames are the calculated plane orientations close to any crystallographic {111} plane, which may be assumed to be a crystallographic plane likely to form facets. The angular difference to the closest {111} plane is given for all possible representations. In all cases, a large angular difference is found.

Facet number 1 is observed to be closer to a [100] plane (minimum deviation 11.8°) and to move almost directly along RD (within 5°).
4. Discussion

4.1 Technique

Topo-tomography has been selected to characterize the growth of an individual grain, which is a different technique as that used in a previous experiment of this type by Schmidt et al.\(^\text{11}\) using 3DXRD.\(^\text{27}\) Some details regarding the samples and the experimental set-ups for the two experiments are summarized in Table 4.

The advantage of topo-tomography over 3DXRD in regard to the specificity recrystallization experiments lies mainly in the better spatial resolution of the former. In topo-tomography, the detector optics and exposure time can be fine-tuned to optimal conditions for the collection of one particular diffraction spot, while for 3DXRD, spots of different diffracting planes having different average intensities need to be collected to reconstruct the grain in 3D. The number of projections of the grain shape from different viewing angles can be set by the user in topo-tomography (20 in the present experiment), while this depends on the crystallography and...
the grain orientation for 3DXRD. Because topo-tomography uses a square beam illuminating always the entire grain, compared to the layer by layer scanning in the experiment of Schmidt et al., the spatial resolution is identical for all directions in topo-tomography, unlike 3DXRD.

The present experiment has a 1.4 µm pixel size of the 3D reconstructions which is about an order of magnitude better than the average pixel size value in the experiment of Schmidt et al. (see Table 4).

Another important difference between the two experiments is that the heat treatment in the present experiment has been interrupted in order to make snapshots at room temperature, while all measurements have been made in-situ in the experiment of Schmidt et al. The latter is of course preferential. In principle in-situ measurements should also be possible using topo-tomography by using a furnace better suited to the topo-tomography setup than the air blower used.

One might speculate whether the interrupted heating had a significant influence on the results, e.g., leading to thermal groovings, which are often observed using surface techniques. For the present experiment, however, no features can be found in the data, which would indicate an influence of the cooling down and reheating on the migration of boundary segments. In the displacement curve derived from the projections measured during heating (cf. Fig. 7), a constant migration rate is obtained in a time span covering multiple cycles of cooling and heating. There is no indication that the boundary migrates faster or slower just after the cooling and reheating compared to its migration in the rest of the heating interval.

### 4.2 Facet orientation

For none of the characterized facets in any of the relevant reference frames a crystallographic {111} plane is observed (cf. Table 3); actually the facet plane orientations are largely different from any dense packed {111} plane. This may appear surprising in view of {111} facets frequently being observed in micrographs of partly recrystallized samples. It however has to be noted that the present facets are highly mobile (observed in-situ during their motion) whereas rather immobile {111} facets are found to form by lateral migration of neighboring boundary segments (ledges) moving faster.

The present facets are very large, more than 100 µm. Their migrations are therefore likely to relate in a complex way to the deformation microstructure. There are significant local (micrometer scale) variations in the local dislocation arrangement and density and thus also crystallographic orientation in the neighboring matrix which the facets have to “deal with” during their migration. Detailed microstructural investigations of the local deformation microstructure in front of a given macroscopic facet and the subsequent migration of the facet is outside the scope of the present work, but will be of interest for further studies.

### 5. Concluding Remarks

(1) The growth of a single grain within the bulk of a 30% cold rolled aluminium single crystal of Cube orientation has been monitored during recrystallization by synchrotron radiation using topo-tomography. With this technique it is possible to characterize the 3D shape of

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**Table 3** Indices of the facet planes in the crystallographic reference frames of the growing grain, the matrix and the intermediate lattice.

<table>
<thead>
<tr>
<th>Reference frame of growing grain</th>
<th>Facet 1</th>
<th>Facet 2 start</th>
<th>Facet 2 end</th>
</tr>
</thead>
<tbody>
<tr>
<td>[−0.736 0.336 0.587]</td>
<td>[−0.340 0.857 0.388]</td>
<td>[−0.226 0.931 0.288]</td>
<td></td>
</tr>
</tbody>
</table>

**Table 4** Comparison of the present experiment with the experiment of Schmidt et al.\(^{11}\)

<table>
<thead>
<tr>
<th>Technique</th>
<th>3DXRD</th>
<th>Topo-tomography</th>
</tr>
</thead>
<tbody>
<tr>
<td>Orientation of deformed matrix</td>
<td>Goss (110)(001)</td>
<td>Close to cube</td>
</tr>
<tr>
<td>Orientation of growing grain</td>
<td>42% cold rolled</td>
<td>30% cold rolled</td>
</tr>
<tr>
<td>Disorientation between grain and matrix</td>
<td>(214)(201)</td>
<td>(133)(312)</td>
</tr>
<tr>
<td>Heat treatment</td>
<td>8° from Σ9</td>
<td>48° (221)</td>
</tr>
<tr>
<td>Time for recording a snapshot</td>
<td>7.5 min/snapshot</td>
<td>12 min/snapshot</td>
</tr>
<tr>
<td>Time resolution*</td>
<td>24.5 min</td>
<td>2.25 min</td>
</tr>
<tr>
<td>Pixel size of 3D reconstructions</td>
<td>6 µm along RD</td>
<td>1.4 µm in all directions</td>
</tr>
<tr>
<td>Approx. volume of initial grain shape</td>
<td>60 µm by 80 µm by 130 µm</td>
<td>170 µm by 170 µm by 100 µm</td>
</tr>
<tr>
<td>Approx. volume of final grain shape</td>
<td>280 µm by 360 µm by 440 µm</td>
<td>340 µm by 210 µm by 140 µm</td>
</tr>
</tbody>
</table>

*Time between the start of successive snapshots during which the sample was heated.
a single grain non-destructively with a high spatial resolution.

(2) Most of the growth of the grain is realized through the migration of a facet along a direction close to the rolling direction. The facet remains planar during the growth and migrates with a constant rate for extended periods of time. The rate increases with an increase of the temperature, but also changes at one other instance in the heat treatment. This latter rate change may be related to the slight change in crystallographic orientation which is observed to develop within the bottom part of the growing grain at the time when the second rate change is observed and/or to changes in the deformation microstructure into which the grain grows at that time. Actually both aspects may be coupled.

(3) The formation of a second facet is analyzed, which shows that an initial facet at some point in time stops migrating and subsequently changes its growth direction and facet plane normal, i.e., a type of stop-go motion is observed for this facet.

(4) None of the observed facets can be related to a crystallographic \{111\} plane in either the reference systems of the growing grain, nor the deformed matrix.

(5) The observed grain boundary migration behavior during recrystallization of grains embedded in the bulk of a specimen could not have been obtained in any other way than with non-destructive 3D characterization techniques as discussed in this article. A statistical analysis of boundary segments in the bulk obtained by 2D or 3D destructive characterization methods of partially recrystallized samples lacks the possibility of revealing the growth mechanism of individual boundaries, while the 2D \textit{in-situ} characterization of recrystallization at the specimen surface cannot circumvent the influence of possible surface effects and misses one dimension to characterize the boundary plane orientation. All information obtained in different types of experiments can be complementary to understanding the full picture, but non-destructive 3D characterization seems, however, compulsory in unrevealing the kinetics of recrystallization and grain boundary migration.

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Fig. A1 Overlay of curves from Fig. 6 and 7 for comparison purposes.