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Abstract: A new method for quantitative analysis of mobilities for local recrystallization boundary segments in pure aluminum is presented. The analysis is based on microstructures characterized using electron microscopy and on quantification of migration velocities and driving forces for local boundary segments. The results show that even for the same recrystallization boundary different boundary segments migrate differently, and the differences can be understood based on quantitative information of mobilities and local deformed microstructures. The present work has important implications for understanding of recrystallization boundary migration, and suggests an experimental way forward for how to determine boundary mobilities during recrystallization.

misorientation axis

local stored energy

1 Quantification of local mobilities

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19 **Abstract:** A new method for quantitative analysis of mobilities for local recrystallization boundary segments
20 in pure aluminum is presented. The analysis is based on microstructures characterized using electron
21 microscopy and on quantification of migration velocities and driving forces for local boundary segments.
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The migration of recrystallization boundaries is typically expressed by the equation:

$$v = MF \quad (1)$$

where v is the migration velocity, M is the boundary mobility and F is the driving force for migration, which for recrystallization is the energy stored in the deformed matrix [1]. Recent new results show, however, that locally the migration of recrystallization boundary segments is very heterogeneous both in time and space: the recrystallization boundary is rough consisting of local protrusions/retrusions and migrates in a stop-go fashion [2-5]. Quantitative analysis of local boundary migration has shown therefore that the boundary mobilities for *local* boundary segments have to be known in order to understand this heterogeneous process [4, 5].

Some results have been published on boundary mobility during recrystallization [6-9]. However, these results are averaged over groups of grains, as the driving force used in these studies is that for the whole sample. It is now well recognized that the deformed microstructure varies from grain to grain [10, 11], and that variations typically also exist within individual grains. The local driving force is therefore expected to vary both on the grain scale and the subgrain scale. These variations have not yet been taken into account for analysis of boundary migration during recrystallization.

The aim of the present study is to address this issue by proposing a method to derive *local mobilities* of boundary segments at different places along a recrystallizing boundary and thus to understand the local boundary migration behavior. This method is based on quantitative analysis of the local deformation microstructures that provides the driving force for the local boundary migration, and of local migration velocities. The results confirm that it is absolutely necessary to study the *local* migration behavior, as even nearby boundary segments on the same boundary behave differently when migrating into the neighboring deformed matrix.

High purity aluminum (99.996%) with a large initial grain size of several millimeters was cold rolled to 50% reduction in thickness, followed by annealing at 250 °C for 10 min to create a partially-recrystallized microstructure. The sample was electropolished for scanning electron microscope (SEM) analysis to allow

selection of a recrystallization boundary. The sample was then annealed 6 times at 250 °C for 15 min in an air furnace to follow the migration of the selected recrystallization boundary. After each annealing step, the microstructure was characterized using the electron channeling contrast (ECC) technique in a Zeiss Supra SEM, without any additional surface treatment. The microstructure after the first ex-situ annealing step (i.e. the first 15 min annealing) was also characterized using the electron backscatter diffraction (EBSD) technique with a step size of 0.5 μm for quantification of local stored energies. During the EBSD scan thermal and mechanical drift of the electron beam and/or the sample as well as geometrical misalignment of the sample [12] induce spatial distortion in the EBSD maps. In order to calculate precisely the local stored energies that provide the driving force for the local boundary migration, these spatial distortions in the EBSD map were corrected using the thin plate spline method described in [13] (see details in section A in the supplementary material).

The ECC image and EBSD maps of the selected boundary are shown in Fig. 1a and 1b, respectively. The micrographs were taken after the first ex-situ annealing step at 250 °C for 15min, and this is referred to as the starting boundary position. The ECC image in Fig. 1a shows that the deformed microstructure consists mainly of two sets of dislocation boundaries, lying at $\pm 25\text{-}30^\circ$ to RD. The spacing of the two sets of dislocation boundaries is about 1-2 μm . A relatively coarse band ($\sim 12 \mu\text{m}$), representing large-scale subdivision [10] is marked by the white lines. In the EBSD map of the same region (Fig. 1b), the large-scale subdivision region appears orange/red, i.e. the crystallographic orientations along the sample normal direction (ND) are closer to $\langle 001 \rangle$, compared to the neighboring yellow and yellow green regions, for which crystallographic orientations along ND are closer to $\langle 012 \rangle$. The microstructure of the coarse band is heterogeneous, consisting of a large number of dislocation boundaries with misorientations $>10\text{-}15^\circ$, while the neighboring yellow and yellow green regions are more homogeneous, consisting of dislocation boundaries with misorientations mainly $<5^\circ$. The two sets of dislocation boundaries in the coarse band region are nearly parallel to those in the homogeneous regions. In the coarse band the set aligned at about $+25^\circ$ to RD is slightly more dominant than the other set, while the two sets of dislocation boundaries in the neighboring yellow and yellow green regions show no significant difference. The region to the right of the

image appears also orange/red, i.e. with ND close to $\langle 001 \rangle$, and consists of dislocation boundaries with misorientations in the range 10-15°.

For quantitative analysis of local boundary migration, the deformed microstructure in front of the migrating boundary is divided into five regions, as marked by the white 25- μm -wide boxes in Fig. 1d. The selection of the boxes is based on the textural (i.e. color in the EBSD map) and microstructural differences: boxes I, III and IV are from the yellow and yellow/green regions where microstructures are relatively homogeneous, while boxes II and V are from orange/red regions where microstructures are more heterogeneous. The orientation variations within the five regions are listed in Table 1.

Table 1. Orientation variation for each of the deformation regions marked by the five boxes in Fig. 1d. Only areas that have been consumed during the 6 annealing steps within each box are included in the calculation.

Boxes	I	II	III	IV	V
Orientation variation	2.0°	10.1°	1.7°	1.8°	4.0°

The position of the recrystallization boundary after the final (6th) annealing step is shown in Fig. 1c. The traces of the recrystallizing boundary after each annealing step are sketched in Fig. 1d based on the ECC images. It is seen that the boundary has migrated mainly along RD. The recrystallizing boundary segments in the central homogeneous regions (in boxes III and IV) are relatively flat and the boundary migration is quite uniform, while the boundary segments in the left heterogeneous region (in box II) are rougher, consisting of many local protrusions and retrusions, and the migration there is non-uniform. A large retrusion is developed in the orange/red region to the right (in box V) during the last 2 annealing steps, while a large protrusion is developed in the region to the left (in box I).

For quantification of local boundary mobilities using Eq. 1, the migration velocity, v , and driving force, F , of local boundary segments within each of the five boxes in Fig. 1d was quantified. Following previous studies [14,15], both the stored energy, F_s , in the deformed microstructure and the driving force attributed to the boundary curvature, F_σ , are quantified and included in total driving force F . For the calculation of the stored energy using the method described in [16,17], the EBSD map was filtered using a 3x3 Kuwahara filter to reduce the orientation noise [18]. After filtering, most of the dislocation boundaries in the deformed microstructure can be seen (see Fig. S2). Using this filtered EBSD map, all dislocation boundaries with misorientations $\geq 1^\circ$ were taken into account in the calculation of F_s . The contribution to the stored energy from the loose dislocation density within the subgrains is not considered, as this is typically very small compared to that from dislocation boundaries [19]. The variations of v and F for each local segment were also estimated as described in section B in the supplementary materials.

The results are shown in Fig. 2. It is evident that the local boundary segments in the five boxes migrate quite differently. Two general trends can, however, be summarized. Firstly homogenous regions lead to relatively homogenous migration and vice versa: the variation of v (the vertical error bars in Fig. 2) is relatively small for boundary segments in boxes I, III and IV (in the range 0.02-0.07 $\mu\text{m}/\text{min}$), compared to those for boundary segments in boxes II and V (in the range 0.08-0.13 $\mu\text{m}/\text{min}$). The observed differences are directly related to the fact that the variation of F (the horizontal error bars in Fig. 2) is relatively small within the homogenous regions (boxes I, III and IV), in the range 0.02-0.05 MJ/m^3 , compared to those for heterogeneous regions (boxes II and V), in the range 0.07-0.15 MJ/m^3 . Secondly, on a local scale both average migration velocity and driving force vary from time to time, even for the boundary segments within the homogenous regions: The average value of v is in the range of 0.10 – 0.41 $\mu\text{m}/\text{min}$, and the average value of F is in the range of 0.07 – 0.3 MJ/m^3 .

For quantification of the mobilities, a linear fitting to Eq. 1 has been performed for the data in each region. The fitting is forced to go through the origin. As shown in Fig. 2, after taking the variations of measured v and F into consideration, Eq. 1 fits well the v - F data for the boundary segments in boxes III and IV, and less well for boundary segments in box I, but does not fit very well for those in boxes II and V.

1 According to Eq. 1, good linear fitting of the ν - F plots for the boundary segments within boxes III and IV
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3 suggests that these migrating boundary segments have similar mobilities at each annealing step. The average
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5 apparent mobilities calculated based on the fitting are $6.6 \pm 0.4 \times 10^{-14} \text{ m}^4\text{s}^{-1}\text{J}^{-1}$ and $5.8 \pm 0.4 \times 10^{-14} \text{ m}^4\text{s}^{-1}\text{J}^{-1}$
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7 for the boundary segments within box III and IV, respectively. The difference between the average apparent
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9 mobilities for these two boxes is relatively small, $< 12\%$, which is in agreement with the fact that the
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11 boundary segments within these two boxes have very similar misorientations to the deformed matrix that
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13 they consume (see Fig. 3c and d).
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17 Although a less good fitting is seen for the boundary segments in box I, the apparent mobilities for the
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19 boundary segments during individual annealing steps are not so different from the average apparent mobility
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21 of the boundary segments during the whole annealing period. The gradual decrease in apparent mobility (Fig.
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23 2a) for these boundary segments during annealing may be related to the gradual change in misorientation
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25 characteristics to the deformed matrix (see Fig. 4a and b). The average apparent mobility calculated based on
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27 the fitting is $5.3 \pm 0.8 \times 10^{-14} \text{ m}^4\text{s}^{-1}\text{J}^{-1}$, which is similar ($\sim 20\%$ difference) to those for the boundary segments
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29 in boxes III and V. The mobility difference between the boundary segments within box I and box III/IV is
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31 likely to be related to the small orientation difference of 3.4° between the deformed matrix within box I and
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33 box III/IV (which leads to a small misorientation difference for the boundaries, see Fig. 3a and c/d).
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38 On the other hand, poor fitting of the ν - F plots for the boundary segments within boxes II and V suggests
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40 that these boundary segments have different mobilities at each annealing step. A simple estimation of the
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42 mobility variation, by calculating mobilities for individual ν - F points, shows that the average apparent
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44 mobilities for the boundary segments within boxes II and V can be in the range 6.0×10^{-15} – $3.4 \times 10^{-14} \text{ m}^4\text{s}^{-1}\text{J}^{-1}$,
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46 i.e. a ~ 6 times difference in magnitude. The mobility differences between the non-migrating and migrating
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48 segments within these boxes are of course even bigger than this.
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52 The large mobility variations for the segments in boxes II and V may be related to the large variation in
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54 misorientation to the deformed matrix (see Fig. 3b and e). It is, however, difficult to establish a one-to-one
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56 correlation between the mobility and misorientation for these regions. This difficulty is illustrated in Fig. 4a
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1 and 4b, where the boundary traces have been overlaid on top of the EBSD maps, which are recolored
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3 according to the misorientation angles and axes, respectively. The color for each pixel of the map represents
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5 the misorientation (angle/axis) at the recrystallization boundary when the recrystallized grain reaches that
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7 pixel. Within the heterogeneous regions (boxes II and V), both the misorientation angles and axes vary
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9 significantly from location to location, both horizontally and vertically.
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12 Nevertheless, some qualitative information can be obtained from Fig. 4 for those segments that halt during
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14 annealing. From the misorientation axis map (Fig. 4b), it is evident that most of the pinning points (e.g. A-D
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16 in Fig. 4b) coincide with $\langle 110 \rangle$ rotation axes (green in Fig. 4b), while the misorientation angles are quite
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18 scattered (Fig. 4a). The local stored energies at these points are relatively high (Fig. 4c). It is therefore very
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20 likely that the boundary segments with misorientation axes close to $\langle 110 \rangle$ have comparatively lower
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22 apparent mobilities than those with other rotation axes. This result agrees well with those obtained based on
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24 grain growth of bicrystals [20]. This is interesting as the driving force for grain growth is different from that
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26 of recrystallization.
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31 The present study shows evidently that local boundary migration heterogeneities are directly related to the
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33 local heterogeneities in the deformation microstructure and boundary mobility. Local quantitative analysis is
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35 essential for understanding the boundary migration behavior, and the results show that using this approach
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37 mobilities can be readily obtained for the local boundary segments that migrate into homogenous
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39 deformation regions. A relation between misorientation and mobility can then be established. For the present
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41 boundary with misorientation of $50^\circ \langle 221 \rangle$ the apparent mobility is in the range $5.8\text{--}6.6 \pm 0.4 \times 10^{-14} \text{ m}^4 \text{ s}^{-1} \text{ J}^{-1}$
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43 at 250°C . The experimental approach presented here suggests a way to determine many more mobilities for
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45 different recrystallization boundaries, which can be the basis for evaluating mobilities obtained by
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47 simulation, e.g. molecular dynamics [21,22], and moreover can be used as an input for modelling of
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49 microstructural development during recrystallization.
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Figure1

Fig. 1. (a) and (c) are ECC images showing the investigated microstructure at the starting state and after 6 steps of annealing at 250°C for 15 min each. The black arrows in (a) mark the groove developed during electropolishing, while the white lines mark the large-scale subdivision region. (b) EBSD map showing the same microstructure as that in (a). The colors in (b) represent the crystallographic direction parallel to the sample ND (see the inset). (d) Traces of the recrystallization boundary after each annealing step. Boxes I-V mark five selected regions for local quantitative analysis. The groove seen in (a) and (c) is shown by a dotted line in (d) for reference.

Figure2

Fig. 2. Migration velocity v for the boundary segments in the five boxes marked in Fig. 1b for each annealing step as a function of total driving force, $F = F_s + F_\sigma$. (a)-(e) are for boundary segments in the boxes I-V, respectively. The error bars represent standard deviations. Numbers are used to identify annealing intervals.

Figure3

Fig. 3. Misorientation (angle/axis pair) distributions for all boundary segments. (a)-(e) are for boxes I-V, respectively.

Figure4

Fig. 4 Boundary traces overlaid on top of the EBSD map. In the EBSD map, each pixel in the deformed matrix is colored according to the misorientation angle (a) and misorientation rotation axis (b) between the pixel and the recrystallized grain, and to the local stored energies (c). A-D mark pinning points. The local stored energy at each pixel is calculated from a subset of 11×11 pixels centered at the selected pixel using the method described in [6].

Supplementary Material

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