Analysis of Inhomogeneous Crystal Rotation in a Grain after Plastic Deformation of a Polycrystalline Low Carbon Steel

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In the case of metals, it is considered that the crystal grain refinement due to plastic deformation is caused by the operation of various slip systems in a grain. In order to understand the initial stage of the grain subdivision, a simple shear test was performed on a low-carbon steel polycrystal having an average grain size of approximately 150 μm.

After the application of 10% shear, several regions having different slip lines were observed in a grain. The slip lines were explained by (110)(111) slip-system of body-centered cubic iron, and the operated Burgers vectors were estimated. The crystal orientations were different for the subdivided regions, and misorientation angles of the crystal rotation were approximately 10°. The rotation axes of the crystal rotation across the subdivided regions were determined. The rotation axes were almost perpendicular to either of the estimated Burgers vectors in the subdivided regions. The directions of the rotation axes are discussed.

Keywords: crystal rotation, dislocation density tensor, lattice curvature tensor, grain subdivision, axis/angle pair

1. Introduction

Grain refinement is an effective strengthening mechanism for combining high strength with good toughness. In recent years, ultrafine grain metals having grain sizes of 1 μm or less have been produced by severe plastic deformation1–3). It is known that grain refinement occurs due to crystal orientation change within a grain during plastic deformation. The microstructural evolution in metals progresses by grain subdivision within a grain during plastic deformation. Using the logarithm of the rotation matrix, the three-dimensional EBSD technique was employed9). Normally, such experimental works are performed using bicrystals or tricrystals focusing on the crystal rotation neighboring existence grain boundaries.

In this study, to investigate the initial stage of grain subdivision during plastic deformation, we analyzed the crystal orientation change across the subdivided regions in a grain of a low-carbon-steel polycrystal. The crystal orientations and slip systems were estimated from observations of the slip line and EBSD measurements on the surface after plastic deformation. Using the logarithm of the rotation matrix10–12), the axes of the crystal rotation caused by the plastic deformation are divided into the rotation about the coordinate axes of the crystal system. The changes of crystal rotation axes across the subdivided regions are discussed.

2. Experimental Procedure

The chemical composition of the polycrystalline low-carbon steel is shown in Table 1. The polycrystalline low-carbon steel was prepared by vacuum melting followed by hot rolling and acid pickling. The hot rolled sheets were cold rolled with a reduction of 60%. In order to obtain relatively coarse and equiaxed recrystallized grains, the cold rolled sheets were heat treated at 973 K for 100 s followed by die quenching to room temperature. They were perfectly recrystallized using heat treatment, and the average grain size was approximately 150 μm.

A planar simple shear test technique13) was employed for applying plastic deformation. Figure 1 shows the geometry of a simple-shear-test specimen. The coordinate frame of the simple shear test is also indicated.

Table 1 Chemical composition of the test material, low-carbon steel.

<table>
<thead>
<tr>
<th>Compositions (mass%)</th>
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<tbody>
<tr>
<td>C</td>
<td>0.0016</td>
<td>&lt;0.1</td>
<td>&lt;0.1</td>
<td>0.0003</td>
<td>0.0002</td>
</tr>
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Fig. 1 Schematic representation of a simple-shear-test specimen. The coordinate frame of the simple shear test is also indicated.
of the simple shear test specimen with coordinate system, the observation direction (OD//x₁-axis), shear direction (SD//x₂-axis), and shear plane normal (SN//x₃-axis). In the simple shear test frame, the components of the deformation gradient tensor \( \mathbf{F} \) by applying a simple shear strain are given by

\[
\mathbf{F} = \begin{pmatrix}
1 & 0 & 0 \\
0 & 1 & \gamma \\
0 & 0 & 1
\end{pmatrix},
\]

where \( \gamma \) is amount of shear calculated by \( \gamma = \delta / h \). \( \delta \) and \( h \) are simple shear displacement and gauge length (3 mm) respectively. After a 10% shear, the slip-line directions and crystal orientations were observed using a field emission scanning electron microscope (FE-SEM) and EBSD respectively. To obtain these observations, the specimens were mechanically polished followed by electrical polishing to eliminate the deformation layer introduced by the mechanical polishing. The EBSD observations were obtained at a step size of 0.2 \( \mu \)m.

3. Results

3.1 Slip-line observation

Figures 2(a) and (b) show the FE-SEM micrograph and inverse pole figure (IPF) map for the same area after the application of a 10% simple shear. The original high-angle grain-boundaries that existed before the simple-shear deformation are identified using the IPF map in Fig. 2(b) and are indicated by white dashed lines in Fig. 2(a). In Fig. 2(a), slip lines are clearly identified on the sample surface. As shown in Fig. 2(a), the central grain in the figure was divided into several regions with parallel slip lines. Figure 3 shows a magnified FE-SEM image of the area of the white solid square in Fig. 2(a). Between the two arrows from (i) to (ii) and (iii) to (iv), there exist transition regions where the slip lines of both sides were observed.

3.2 Analysis of crystal rotation

The objective of this study is to consider the initial stage of the grain subdivision within a grain where the crystal orientation was originally the same. As shown later, the changes of crystal orientation due to a 10% simple-shear deformation are up to approximately 10°. We have analyzed such changes of crystal orientation using the following method.

By using conventional software to treat the EBSD results, we can obtain the Euler angles corresponding to the crystal orientations. However, even if the crystal orientations of two neighboring points are near each other and, for example, the angle between \( \{100\} \) of them is approximately 10°, the Euler angles may not always cause the result that these \( \{100\} \) are the same \( [100] \). Therefore, in this study, in order to discuss the changes of crystal orientations, we have corrected the Euler angles by considering the symmetry of the cubic structure so that near \( \{100\} \) can be expressed as the same \( [100] \).

Using the corrected Euler angles, we obtained the rotation matrices. The rotation matrix indicating the crystal orientation at each position in the observation region is defined as shown in Fig. 4 with respect to the simple-shear test coordi-
Fig. 4 Definition of rotation matrices $\mathbf{R}$ and $\Delta \mathbf{R}$ showing crystal orientations obtained from the EBSD measurements. $\mathbf{a}_1$ and $\mathbf{a}_2$ show the locations at which the EBSD measurements are made. The reference frame used is the simple-shear-test coordinate frame shown in Fig. 1.

\[
\Delta \mathbf{R} = \mathbf{R}(\mathbf{a}_2)\mathbf{R}^{-1}(\mathbf{a}_1),
\]

where $\mathbf{R}(\mathbf{a}_1)$ and $\mathbf{R}(\mathbf{a}_2)$ are the rotation matrices that give the crystal orientations at $\mathbf{a}_1$ and $\mathbf{a}_2$ respectively, and $\mathbf{R}^{-1}(\mathbf{a}_1)$ is the inverse matrix of $\mathbf{R}(\mathbf{a}_1)$.

The rotation matrix $\Delta \mathbf{R}$ can be expressed by using a rotation angle $\Phi$ around a unit vector $\mathbf{n} = (h, k, l)^{14}$ called the axis/angle pair. The result is shown in Appendix A.1. The logarithm $\ln \Delta \mathbf{R}$ of $\mathbf{AR}^{10–12}$ is a skew symmetric matrix having three independent elements $(\omega_1, \omega_2, \omega_3)$. On using the axis/angle pair, $\ln \Delta \mathbf{R}$ is given by$^{10–12}$

\[
\ln \Delta \mathbf{R} = \begin{pmatrix} 0 & -\omega_3 & \omega_2 \\ \omega_3 & 0 & -\omega_1 \\ -\omega_2 & \omega_1 & 0 \end{pmatrix} \begin{pmatrix} 0 & -l\Phi & k\Phi \\ l\Phi & 0 & -h\Phi \\ -k\Phi & h\Phi & 0 \end{pmatrix},
\]

The elements $\omega_1, \omega_2$ and $\omega_3$ are considered to be the sums of the divided rotation angles around the coordinate axes of the reference frame$^{10}$. By using $\ln \Delta \mathbf{R}$, we can divide a three-dimensional rotation into sets of rotations about the reference frame$^{11}$. For $\mathbf{AR}$ with elements of numerical values, $\ln \Delta \mathbf{R}$ can be calculated using recent mathematical computing programs such as Mathematica 9 or MATLAB.

Figures 5(a) and (b) show the variation of the point-to-origin misorientation angle along the two arrows in Fig. 3 from (i) to (ii) and from (iii) to (iv), respectively. In the line scan, the position $\mathbf{a}_1$ in the right-hand side of the eq. (2) is set as (i) or (iii), which is the origin of the line scan, and the position $\mathbf{a}_2$ is set as the analysis point on the line scan. As shown in Fig. 5, the scanned lines can be divided into single-slip-line regions and transition regions of $[\mathbf{A}]$–$[\mathbf{C}]$ as shown in Fig. 5(a) and $[\mathbf{D}]$–$[\mathbf{F}]$ as shown in Fig. 5(b) by comparing them with the slip line observations shown in Fig. 3. In the figures, the locations of the representative orientations$^{15}$ in each region are also indicated by open arrows. The representative orientation is defined as the orientation at a certain point where the sum of the misorientation angles between the certain point and those of other points in the region becomes the minimum$^{15}$. Orientation changes along the line scans mainly occurred in the transition regions $[\mathbf{B}]$ and $[\mathbf{E}]$ in Figs. 5(a) and (b) respectively.

3.3 Operated slip system

In the case of body-centered cubic (bcc) iron, the slip direction is well confirmed as the closest packed $\{111\}$ direction, and its Burgers vector is $\mathbf{a}/2(111)$ where $\mathbf{a}$ is a lattice constant. Moreover, there are three postulated possible slip planes $\{110\}$, $\{112\}$ and $\{123\}$ for bcc iron. In order to estimate the slip system for each region, the directions of three possible slip plane traces obtained from the EBSD measurement are compared with the slip lines that were experimentally observed using the FE-SEM in Fig. 3. Figure 6 show the stereographic projections of the representative orientation in regions $[\mathbf{A}]$, $[\mathbf{C}]$, $[\mathbf{D}]$ and $[\mathbf{F}]$. The slip plane traces observed using the FE-SEM are indicated by black solid lines with arrows. In all the cases, the slip planes coincided well with the trace of $[110]$. On the $[110]$ slip plane, there are two possible (111) slip directions. By considering the direction of the applied simple shear, we estimated that the operated slip systems in the regions $[\mathbf{A}]$ and $[\mathbf{F}]$ were $\{011\}[111]$, and that in the regions $[\mathbf{C}]$ and $[\mathbf{D}]$ were $\{101\}[111]$ respectively. The majority of the slip systems estimated in this study was the $\{110\}(111)$ system, and thus,
we analyzed only the \{110\}/\{111\} slip system for the analysis of the crystal rotation axis.

### 3.4 Crystal rotation axis

The rotation axes of the crystal rotation in each region from [A] to [F] shown in Fig. 5(a) and (b) are discussed. The changes of crystal rotation axis in each region along the line scans were analyzed using eqs. (2) and (3). Here, \( \Delta R \) at a certain point in each region was calculated with a \( t \) of the right-hand side of eq. (2) as the position giving the representative orientation in each region. The rotation axes in each region were obtained in this manner. In Fig. 7, the rotation axes of \( \mathbf{R}, \mathbf{n} = (h, k, l) \), are plotted as small black dots in the stereographic projections showing the representative orientation of each region [A], [B] and [C]. In the figures, the operated slip planes are indicated using black solid lines with solid arrows. Possible slip planes are also indicated using a black broken line for \{110\}, gray dotted line for \{112\} and black dotted line for \{123\}.

In the initial stage of grain subdivision caused by plastic deformation, transition regions were generated by operation of various slip systems in a grain\(^4\,\,^5\). As a result, these regions rotate towards different orientations, and the dislocations on the operated slip plane become GNDs around the
transition region in order to accommodate the incompatibility of the different orientations. The accumulation of GNDs around the transition region is illustrated in Fig. 9. Analysis of the crystal rotation axis in each region shows that the rotation axes in the transition region were located between the directions orthogonal to the SPN and BV in both sides of the subdivided regions. The GNDs are necessary for accommodating the difference in directions of rotation. Based on Figs. 7 and 8, the generation of the transition region can be explained as the result of the accumulation of GNDs from both sides of the subdivided regions.

In Figs. 7 and 8, the crystal rotation axes were analyzed by considering \( a_1 \) in Fig. 4 as the location giving the representative orientation in each region. The stereographic projections in Figs. 7 and 8 show the results for various regions in Fig. 5 with reference to the fixed simple-share test frame. However, the crystal orientation and its slip system continuously rotate as a function of the distance along the line scan. To discuss the crystallographic relation between the changes of the crystal rotation axes and their slip systems including the continuous rotation, the crystal coordinate systems corresponding to the crystal orientations at various locations should be considered. When we use the crystal coordinate system at a certain location \( a_1 \) to express the additional rotation from the orientation at \( a_1 \) given by \( R(a_1) \) to that at \( a_2 \) given by \( R(a_2) \), the additional rotation \( \Delta R_C \) is given by

\[
\Delta R_C = R^{-1}(a_1) R(a_2). \tag{4}
\]

Figure 10 shows the definition of \( \Delta R_C \) when we consider various crystal orientations expressed by the \( x'_1-x'_2-x'_3 \) crystal coordinates at various \( a_1 \). In the following analysis, the \( x'_1-x'_2-x'_3 \) coordinate is constructed by the axis orthogonal to BV and SPN (ORD//\( x'_3 \)), BV//\( x'_2 \) and SPN//\( x'_3 \) as shown in Fig. 10.

Using eq. (4), we calculated \( \Delta R_C \) and obtained the changes of crystal rotation axes at various locations along the line scan at 10 \( \mu \)m intervals. Using \( \Delta R_C \), the crystal rotation axes can be shown in the same crystal coordinate system. Figure 11(a) shows the results along the line scan of (i)–(ii) as a function of the distance from the origin (i). In Fig. 11, subscripts \([A]\) and \([C]\) of ORD, BV and SPN indicate the referenced slip system operated either in region \([A]\) or \([C]\) that is used to determine these axes’ directions. Their crystallographic directions are also indicated in the figure. As shown in Fig. 11(a), the referenced crystal coordinate frame is the slip system in region \([C]\) in this case. In the case of the line scan in region \([A]\), the rotation axes are

![Fig. 10](image_url)

**Fig. 10** Schematic of the calculation of the rotation matrices in the region \( \Delta R_C \) of eq. (4) with respect to the crystal coordinate frame. The crystal coordinate frames at each \( a_1 \) are also indicated.

![Fig. 9](image_url)

**Fig. 9** Schematic of a wall of GNDs caused by the motion of dislocations of various slip systems. Cubes labelled \([A]\), \([B]\) and \([C]\) show the variation of crystal orientation along the arrow.

![Fig. 11](image_url)

**Fig. 11** Analysis of crystal rotation at 10-\( \mu \)m intervals along the line scan from (I) to (II) in Fig. 3. (a) Changes of crystal rotation axes shown in the stereographic projection. Gray and black chain lines indicate the planes orthogonal to the Burgers vector for regions \([A]\) and \([C]\) respectively. The crystal coordinate frame is also indicated. (b) Changes of ratio of \( \omega_{ORD}/\Phi \), \( \omega_{BV}/\Phi \) and \( \omega_{SPN}/\Phi \) as a function of the distance from (i).
close to a plane that is orthogonal to \( \mathbf{B}_V \mid \alpha \), and the axes then move toward a plane that is orthogonal to \( \mathbf{B}_V \mid \beta \).

By using the logarithm \( \ln \mathbf{AR} \) of \( \mathbf{AR} \), the rotation can be divided into a crystal coordinate system. A set of rotation angles, \( \omega_{\text{ORD}}, \omega_{\text{BV}}, \omega_{\text{SPN}} \), component of \( \Phi \) around ORD, BV, and SPN, respectively, can be obtained by using \( \ln \mathbf{AR} \). Figure 11(b) shows the changes of the rotation angle \( \Phi \) for the total rotation angle \( \Phi \) as a function of the distance from the origin (i). The rotation of \( \omega_{\text{SPN}} \)/\( \omega_{\text{ORD}} \) becomes the main component of the rotation in regions [B] and [C].

The relation between the dislocation density tensor and lattice curvature tensor\(^7\) explains that the crystal rotation axis caused by arrays of dislocation becomes orthogonal to the Burgers vector. The detailed information can be expressed using the lattice rotation, \( \Phi \), and number of dislocation lines per unit area normal to the lines \( n \),

\[
\alpha_{ij} = n b_i r_j \quad (i, j = 1, 2, 3).
\]

Furthermore, \( \kappa_{ij} \) can be expressed using the lattice rotation, \( \phi_i \), about the \( x_i \) axis,

\[
\kappa_{ij} = \partial \phi_i / \partial x_j \quad (i, j = 1, 2, 3).
\]

By substituting eqs. (A4)–(A6), the relation between the Burgers vector and crystal rotation axis is given by

\[
\alpha_{ij} = \begin{pmatrix} nb_1 r_1 & nb_2 r_2 & nb_3 r_3 \end{pmatrix}
\begin{pmatrix} \alpha_{11} & \alpha_{12} & \alpha_{13} \\ \alpha_{21} & \alpha_{22} & \alpha_{23} \\ \alpha_{31} & \alpha_{32} & \alpha_{33} \end{pmatrix}
\begin{pmatrix} nb_1 r_1 \\ nb_2 r_2 \\ nb_3 r_3 \end{pmatrix}
\]

In case of all of the edge dislocation components, i.e., \( \alpha \neq \beta \), the relation between the Burgers vector \( \mathbf{b}_h \) and lattice rotation about the \( j \) axis \( \phi_i \) is orthogonal. However, for the screw dislocation,

\[
\kappa_{ij} - \alpha_{ij} - \frac{1}{2} \delta_{ij} \alpha_{kk} = \begin{pmatrix} \kappa_{11} & \kappa_{12} & \kappa_{13} \\ \kappa_{21} & \kappa_{22} & \kappa_{23} \\ \kappa_{31} & \kappa_{32} & \kappa_{33} \end{pmatrix}
\begin{pmatrix} 1/2(a_{11} + a_{22} - a_{33}) & a_{21} & a_{31} \\ a_{12} & 1/2(a_{22} - a_{33} - a_{11}) & a_{32} \\ a_{13} & a_{23} & 1/2(a_{33} - a_{11} - a_{22}) \end{pmatrix}
\]

An example of an array of parallel screw dislocations in a small volume \( (\delta x_1, \delta x_2, \delta x_3) \) on assuming \( a_{11} = a \) while the other values of \( a_{ij} = 0 \) is illustrated in Fig. A-1. The lattice curvature tensor in this assumption can be written as

\[
\kappa_{ij} = \begin{pmatrix} 0 & 0 & 0 \\ 0 & 0 & 0 \\ 0 & 0 & 0 \end{pmatrix}
\]

From this relation, \( \Phi \) and \( \mathbf{n} = (h, k, l) \) are calculated as follows,

\[
\Phi = \cos^{-1}\left(\frac{\Delta R_{11} + \Delta R_{22} + \Delta R_{33} - 1}{2}\right),
\]

and

\[
(h, k, l) = \left(\frac{\Delta R_{32} - \Delta R_{23}}{2 \sin \Phi}, \frac{\Delta R_{13} - \Delta R_{31}}{2 \sin \Phi}, \frac{\Delta R_{23} - \Delta R_{12}}{2 \sin \Phi}\right).
\]
When the line scan direction is parallel to the $x_1$-axis, the crystal rotates about the $x_1$-axis. However, when the scan direction is parallel to the $x_2$-axis and $x_3$-axis, the crystal rotation axis becomes the $x_2$-axis and $x_3$-axis respectively. Consequently, the rotation axis is not orthogonal to the slip direction only when the Burgers vector of the screw dislocation is parallel to the scan direction.

REFERENCES