Three-dimensional EBSD Analysis and TEM Observation for Interface Microstructure during Reverse Phase Transformation in Low Carbon Steels

Kengo HATA,^{1)*} Kazuki FUJIWARA,¹⁾ Kaori KAWANO,¹⁾ Masaaki SUGIYAMA,^{1,2)} Takashi FUKUDA²⁾ and Tomoyuki KAKESHITA²⁾

1) Advanced Technology Research Laboratories, Nippon Steel & Sumitomo Metal Corporation, Hyogo, 660-0891 Japan.

2) Department of Materials Science and Engineering, Osaka University, Osaka, 565-0871 Japan.

(Received on October 27, 2017; accepted on December 25, 2017)

For the development of advanced steels, reverse phase transformation from ferrite is essentially important to control the austenite phase in the heating process. Formation of austenite during the initial stage of reverse transformation from the recrystallized ferrite in low carbon steel has been studied from the view-point of the orientation relationships and the interphase boundary structure. At high temperature, the *in situ* electron backscattering diffraction (EBSD) measurement of austenite grain growth during the reverse transformation indicates that the different migration behaviors according to different α/γ interfaces, deriving from the interfacial coherency with the specific orientation relationships. The orientation and microstructure of the interface between ferrite and austenite have been investigated using the 3D crystal orientation analysis and transmission electron microscopy (TEM) observations. When the crystal orientation relationship between ferrite and austenite grain are close to the Kurdjumov–Sachs relationship, the grain boundary normal itself is also close to the $\{111\}_{\gamma}$ and $\{011\}_{\alpha}$, respectively. The microstructure of these interfacial planes is revealed to be flat using 3D-EBSD and TEM analysis. These coherent planes are strongly connected to the formation of the austenite phase on heating and also affect the slow migration of the grain-growth process.

KEY WORDS: phase transformation; crystal orientation relationship; interface boundary plane; three-dimensional EBSD analysis; TEM observation; low carbon steel.

1. Introduction

In the steel production process, the phase transformation from ferrite to austenite during heat treatment is one of the key factors for controlling the complex microstructure of the high strength steel products with good mechanical properties. The amount and morphology of the austenite grains in the temperature of the two-phase region often influence the subsequent martensite and bainite transformations. The coherency of the interface boundary of an austenite grain will relate to the mobility of the interface boundary, which changes the size and morphology of the austenite phase. Based on this view-point, the understanding of the formation mechanism of austenite during the reverse phase transformation from ferrite is required to realize various applications field and also for the scientific interests.^{1–6)}

The coherency of the boundary between ferrite and austenite has been studied in connection with specific orientation relationships^{7–17)} such as the Kurdjumov–Sachs¹⁸⁾ (K–S) and the Nishiyama–Wasserman (N–W) relationship.¹⁹⁾ A computer simulation of the atomic structure predicted the

existence of the partially coherent atomic structure on the boundary which is related with the specific crystal orientation relationships. Aaronson *et al.* studied the geometrical atom matching on the interphase boundary between the bcc and fcc phase and demonstrated the different cases of the K–S relationship or the N–W relationship, and the necessity of partial coherent atomic matching areas facing on $\{011\}_{\text{bcc}} // \{111\}_{\text{fcc}}$ were discussed.

After considering the orientation relationship between the ferrite and austenite phases in low carbon steels, it has been suggested that product phases of phase transformation satisfy the specific orientation relationships at plural interfaces to parent phase.^{20–31)}

Lischewski *et al.* observed the $\alpha \rightarrow \gamma$ phase transformation in the C–Mn steel containing microalloying elements such as Niobium by *in situ* electron backscattering diffraction (EBSD) measurement, and analyzed the orientation relationship between the parent ferrite and transformed austenite grains.^{28,29)} They found that a considerable number of transformed austenite grains are related to two adjacent mother grains by the approximate K–S relationship and proposed that the variant selection rule is due to the preferential nucleation of the K–S variant with low interfacial energy at a grain boundary.

* Corresponding author: E-mail: hata.pq3.kengo@jp.nssmc.com
DOI: <http://dx.doi.org/10.2355/isijinternational.ISIJINT-2017-639>

The authors of the present study investigated the frequency of the orientation relationship between the austenite and adjacent ferrite grains during the same $\alpha \rightarrow \gamma$ transformation in low carbon steels.^{30,31)} The orientation of the reverse transformed austenite satisfies the orientation relationships close to the K–S relationship (within 6°) with one or two ferrite grains. Furthermore, another neighboring interface is still close to the K–S relationship within 12° . That is, the nucleation process of austenite is related to not only one adjacent ferrite grains but to plural adjacent ferrite grains at the grain corner.

On the basis of these observations, there is a possibility that the specific orientation relationships tend to be satisfied at plural interfaces between the ferrite and austenite phases during the initial stage of the reverse transformation. The interfaces with the specific orientation relationships may restrict the behavior of the subsequent grain-growth of austenite because the mobility of the α/γ interface is influenced by the coherency between the two phases.^{32–36)} These characteristic orientation relationships and subsequent grain-growth behavior of austenite transformed at a grain corner are likely to relate with the interface structure between ferrite and austenite. By applying the method of reconstruction of three-dimensional microstructure comprising ferrite and austenite, the interface properties have been investigated in this study.

To investigate the influence of the orientation relationship and interfacial planes on the formation of austenite microstructure during the reverse transformation, the crystallographic analysis on the microstructure in the early stage has been conducted using three-dimensional (3D) EBSD measurements and transmission electron microscopy (TEM) observations in this study. The relevance between the grain-growth of austenite and the crystal orientation relationship between ferrite and austenite has been discussed based on the mechanism of the microstructure formation during the reverse transformation.

2. Experimental Procedure

Fe-1.0Mn-0.1C (mass%) low carbon steel was melted and cast as a 50-kg ingot in a vacuum induction furnace. The ingot was hot-rolled to 2 mm in thickness and cooled to 710°C by a water spray, and then the rolled plate was cooled to room temperature at a cooling rate of $20^\circ\text{C}/\text{h}$ in a furnace. The plate was then cold-rolled to 1 mm in thick-

ness, and test pieces were cut from the middle part of the plate with dimensions of $200 \times 10 \times 1 \text{ mm}^3$. After that, the test pieces were heated at 730°C at a rate of $5^\circ\text{C}/\text{s}$, and they were immediately quenched at a rate of greater than $1\,000^\circ\text{C}/\text{s}$ using a high-pressure water spray system.

The samples for TEM observation were cut from the heat-treated test piece, mechanically thinned to a thickness of less than 0.1 mm, and punched to 3 mm discs in diameter. The conventional electropolishing method was done using an electrolyte solution containing 5% perchloric acid and 95% acetic acid. The observations were conducted using a Hitachi HF-2000 (FE-TEM) operating at 200 kV.

The samples for 3D EBSD measurement were prepared from the same test piece. The surface was first mechanically polished and then chemically polished with a colloidal-silica suspension. The sample was tempered at 400°C for 1 h to obtain clear EBSD patterns of martensite. It was confirmed that no change of the crystal information of the lath martensite occurred by the low temperature tempering at 400°C for 1 h.

The chemical composition of the steel used for *in situ* EBSD measurement is selected to be Fe-0.2C (mass%) to avoid evaporation of manganese at high temperature. A $5 \times 7 \text{ mm}$ sample was cut from a cold-rolled sheet and the surface plane was mechanically grinded to a thickness of 0.8 mm, and then electropolished. High temperature EBSD measurement was conducted using a FE-SEM (FEI Quanta 200 FEG) equipped with the heating stage made by TSL Solutions Japan in the temperatures range from 680 to 840°C .

A 3D EBSD measurement was performed using a FEI Quanta 3D FEG equipped with a Ga ion beam column and an EBSD detector.³¹⁾ The geometry of the measurement system is schematically depicted in Fig. 1. The sample is installed on the pretilted-holder at 54° and mounted on a stage reversely tilted by 16° from the horizontal position. This setup realizes grazing-incidence ion milling by the ion-beam column, which is mounted at an angle of 38° from the vertical. The movement from the milling position to the EBSD position is accomplished by rotating the stage by 180° , followed by the automatic adjustment of the stage to the eucentric position by the detection of a fiducial marker that is milled next to the measured area. The plane of the sample was sliced by the sputtering of a Ga^+ ion beam accelerated at 30 keV at a step of $0.1\text{-}\mu\text{m}$ thickness. On each of the sputtered planes, EBSD measurement was performed

Fig. 1. Schematic of the geometry of the 3D EBSD measurement setup.

in a $27 \times 40 \mu\text{m}^2$ area with a step size of 80 nm in a rectangular scan grid. The acquired EBSD dataset includes a slight displacement among adjacent sections due to sample drift in the position during the measurement. For precise alignment of the EBSD maps, the deviation of each image is corrected by a calculation method using the Euler angles and its position (X, Y) in the EBSD maps.³⁷⁾ The differences of Euler angles at the corresponding positions between any two EBSD image data are calculated and compared to each other. The position of each EBSD image data is corrected as the exact position with a minimum difference of Euler angles among adjacent sections.

3. Experimental Results

3.1. *In situ* EBSD Measurement of Austenite Grain Growth during Reverse Transformation

The microstructure change of the Fe-0.2C alloy owing to ferrite to austenite transformation from 730 to 840°C was observed using *in situ* EBSD measurement in the same way as the previous study³¹⁾ to confirm the repeatability. The grain-growth of austenite with increasing temperature of the sample is indicated in Figs. 2(a)–2(d). In the figures, the ferrite grains are shown as the gray scale images representing the quality of EBSD band patterns and austenite grains are presented in red. The microstructure obtained at 730°C comprises ferrite and austenite, and the austenite grains appeared at the triple junction of ferrite grains. With further heating, the austenite grains grew into the adjacent ferrite grains, and finally completely covered the grains in the observation area at 840°C. The crystal orientation relationship between ferrite and austenite at some boundaries was analyzed by the difference of the misorientation. The, $\Delta\theta^{\text{KS}}$ is defined as the deviation angle from the measured misorientation one to the exact K–S relationship one at the boundary. The $\Delta\theta^{\text{KS}}$ is also noted by the equation in the next section. Example of the analysis for grains A to D shown in Fig. 2(a) is summarized in Table 1. In the table, the distances of the austenite boundary migration between 730°C and 790°C are also shown. As is seen in Table 1, Interface 2 in Austenite grain D migrated by less than $1 \mu\text{m}$, and the orientation relationship is close to the K–S relationship ($\Delta\theta^{\text{KS}} = 1^\circ$). The Interfaces 1, 3, 4 and 5 of Austenite grain D migrated by more than $1 \mu\text{m}$, and the orientation relationships are deviated from the K–S relationship by over 9° . The interfaces satisfying the K–S relationship are difficult to migrate smoothly. The interfaces with the orientation rela-

tionship deviated from the K–S relationship with more than 9° misorientation migrate quickly. It must be noted here that the true migration speed in three-dimension is not measured, resulting in the appearance of slow migration interfaces with a large deviation from the K–S. However, these results shown in Table 1 are consistent with previous study.³¹⁾

3.2. Interphase Boundary Plane Analysis by 3D EBSD Measurement

The boundary planes of the interfaces with the orientation relationship close to the K–S relationship were investigated in the Fe-1.0Mn-0.1C alloy using 3D EBSD measurement. During the heating process to 730°C in the two phase temperature region at a heating rate of 5°C/s, initial austenite grains appear at the triple junctions. The appearance of the austenite grains is clearly confirmed from the observation that martensite exists at the triple junction in the sample immediately quenched at 730°C at a rate of more than 1 000°C/s. The microstructure obtained by quenching is confirmed to be the same as that observed by the *in situ* EBSD observation of Fe-0.2C alloy at 730°C, as seen in

Table 1. Misorientation from the K–S relationship and distance of interface migration between 730°C to 790°C.

Austenite grain	No. of interface to adjacent ferrite grain	Misorientation from the K–S relationship, $\Delta\theta^{\text{KS}}$ (deg.)	Distance of interface migration between 730°C and 790°C (mm)
A	Interface 1	3	0.4
	Interface 2	14	0.7
	Interface 3	1	0.9
B	Interface 1	15	1.6
	Interface 2	15	1.7
	Interface 3	24	2.5
	Interface 4	31	1.8
C	Interface 1	3	0.4
	Interface 2	1	0.5
	Interface 3	15	0.7
D	Interface 1	9	1.3
	Interface 2	1	0.4
	Interface 3	26	2.5
	Interface 4	9	8.4
	Interface 5	19	1.8

Fig. 2. Microstructure measured by *in situ* EBSD at different temperatures during the reverse transformation in Fe-0.2C alloy (the ferrite microstructure is indicated by gray scale image representing the quality of EBSD band patterns and the austenite grains are shown in red color).³¹⁾

Fig. 2(a). Both experimental results show that the reverse transformation from the ferrite to the austenite phase occurs at the triple junction in low carbon steels.

The typical local areas including a martensite region and the adjacent ferrite grains were investigated using 3D EBSD measurement in the sample quenched from 730°C. The procedure for the 3D reconstruction of the crystal structure is similar to that in the previous paper,³¹⁾ and one example is indicated by a series of cross sectional maps in Fig. 3. The total number of slice images is 70 in this case, and each crystal orientation map in Fig. 3 corresponds to the crystal orientation of the slice at a depth of 0.4, 0.8, 1.2, 1.6, 2.0, and 2.4 μm . Inside the martensite region, several types of color pixels are observed, in which correspond to different martensite blocks. The black dots in Fig. 3 are the region

wherein clear Kikuchi patterns were not obtained. The interfaces between the region of martensite and the adjacent ferrite grains are clearly defined with reliable accuracy.

Using the orientation data of martensite blocks in the 3D microstructure, the prior austenite orientation was calculated. The details of the analysis method were described previously.^{31,38–44)} In our previous study,³¹⁾ the misorientation between the reconstructed austenite grain and each surrounding ferrite grain was investigated. In the present analysis, we focus on the characteristics of crystallography of interfaces itself. First, the orientation relationship is evaluated as the misorientation from the K–S relationship, $\Delta\theta^{\text{KS}}$, by the following Eqs. (1) and (2).

$$\mathbf{M} = \mathbf{g}^{\alpha} \left(\mathbf{R}_j \mathbf{V}_i^{\text{KS}} \mathbf{g}^{\gamma} \right)^{-1} \dots\dots\dots (1)$$

Fig. 3. A series of orientation mappings of martensite in Fe-0.1C-1Mn alloy quenched from 730°C in the heat treatment, measured at a depth of (a) 0.4 μm , (b) 0.8 μm , (c) 1.2 μm , (d) 1.6 μm , (e) 2.0 μm , (f) 2.4 μm (the color indicates the crystal orientation, which corresponds to the attached standard stereo triangle).

Fig. 4. (a) A reconstructed austenite grain (martensite region) in Fe-0.1C-1Mn alloy reconstructed from the EBSD data of martensite blocks shown in Figs. 3(a)–3(f) (the broken black lines are superimposed along the grain edge of the prior austenite to clarify its morphology), (b) definition of the sample coordinate system, (c) the color code on the surface indicating the grain boundary normal in the sample coordinate system, (d) schematic for analysis method of boundary plane normal based on the voxel model, displaying the voxel of interest and eight voxels in first neighbor on surface as well as eight vectors for calculating the normal vector.

$$\Delta\theta^{\text{KS}} = \cos^{-1} \left(\left(M[1,1] + M[2,2] + M[3,3] - 1 \right) / 2 \right) \dots (2)$$

M represents the rotation matrix from the ideal orientation of ferrite in the exact K–S relationship to the measured ferrite orientation. $\mathbf{V}_i^{\text{KS}}$ ($i = 1 \dots 24$) represents the exact K–S relationship, which is expressed by the 90° rotation around the $\langle 112 \rangle$ axis of the austenite crystal coordinate system. The indices $[i, i]$ ($i = 1 \dots 3$) express diagonal components of the 3×3 matrix. $\mathbf{g}^\gamma$ and $\mathbf{g}^\alpha$ are the orientations of the prior austenite and the adjacent ferrite. $\mathbf{R}_j$ ($j = 1 \dots 24$) represents the symmetry of the bcc crystal system.

In the reconstructed microstructure, most of the austenite grain is adjacent to four or five ferrite grains, which indicates that austenite nucleates at grain corners of ferrite matrix. The 3D austenite grain is extracted from the 3D crystal microstructure. A representative austenite grain is analyzed in **Fig. 4**, which is reconstructed from the martensite region shown in Figs. 3(a)–3(f). The austenite grain has five interfaces among ferrite grains in the case of **Fig. 4(a)**, and the misorientation angles, $\Delta\theta^{\text{KS}}$, of these interfaces are 2°, 6°, 14°, 20° and 28°, respectively. The each color of the voxel on the interface in **Fig. 4(a)** represents the grain boundary normal at each voxel, which is defined in the sample coordinate system of Figs. 4(b) and 4(c).³⁵⁾ The calculation method is briefly explained as follows.

The normal vector of the interface in the 3D microstructure is evaluated as the average of plural cross products of pairs of vectors that are set on the interface in the sample coordinate system. As depicted schematically in **Fig. 4(b)**, to determine the normal direction of a center voxel of interest in the sample coordinate system, $\mathbf{n}_s$, eight vectors $\mathbf{x}_1$ – $\mathbf{x}_8$ that start from the center voxel and extend to the neighboring voxels are taken into account. Every cross product of two adjacent vectors among $\mathbf{x}_1$ – $\mathbf{x}_8$ is calculated, and the average of the vectors is given as follows.

$$\mathbf{n}_s = \frac{1}{8} \sum_{i=1}^8 \frac{\mathbf{x}_i \times \mathbf{x}_{i+1}}{\sqrt{|\mathbf{x}_i|^2 |\mathbf{x}_{i+1}|^2 - |\mathbf{x}_i \cdot \mathbf{x}_{i+1}|^2}} \dots (3)$$

The vector $\mathbf{x}_9$, which appears in the case of $i = 8$, is equal to $\mathbf{x}_1$. Although in the above explanation, only the first neighbor voxels to a center voxel of interest are used, the second voxels are taken into account to calculate the normal vector at the center voxel in the present study for more precise evaluation of the grain boundary normal.

The accuracy of the grain boundary normal by 3D EBSD analysis is still not high, but a number of local normal vectors at voxels on the interface and their average will provide the mode in the statistics of the normal directions as well as the curvature of the interface. Therefore, it is possible to compare the mode of grain boundary normal and the crystal orientations of ferrite and austenite.

The results of the analysis on interfacial boundary planes for a region of martensite are shown by the pole figures in **Fig. 5**. The distribution of the grain boundary normal is expressed by the contour map in each pole figures of **Figs. 5(d)–5(f)**. In these figures, $\{001\}_\gamma$, $\{011\}_\gamma$ and $\{111\}_\gamma$ poles are indicated by crosses with red, green and blue colors, and the poles of $\{001\}_\alpha$, $\{011\}_\alpha$, and $\{111\}_\alpha$ of each neighbor ferrite grains are indicated by colored circles. The vertical

and horizontal axes of the pole figure are parallel to the two edges of the rectangle sample, respectively.

The interface boundary between the ferrite and austenite grain in **Fig. 5** are analyzed as follows. The orientation relationship at Interface 1 is related by the K–S relationship with $\Delta\theta^{\text{KS}} = 1^\circ$. The nearest normal of close-packed planes $\{111\}_\gamma // \{011\}_\alpha$ is indicated by an arrow in **Fig. 5(d)**. The distribution of the grain boundary normals, which is shown as a contour map, is very close to the pole of the close-packed planes. The deviation of the mode of the grain boundary normals from the close-packed plane is estimated $\sim 5^\circ$. That is, the interface satisfying the close K–S relationships is also close to the close-packed planes of $\{111\}_\gamma // \{011\}_\alpha$. In the case of Interface 2, the orientation relationship between the grains is related by the K–S relationship with $\Delta\theta^{\text{KS}} = 3^\circ$. The mode of the grain boundary normals in **Fig. 5(e)** is in the vicinity of the close-packed planes indicated by the arrow with a deviation of $\sim 6^\circ$. At Interface 3 and 4 in **Figs. 5(f)** and **5(g)**, the orientation relationships are deviated from the K–S relationship by 8° and 22°, respectively. The interfacial boundary largely deviated from both of the close-packed planes of the ferrite and austenite phase.

A similar analysis was made for other austenite grains and an example of the analysis is shown in **Fig. 6**. For the austenite region in **Fig. 6**, Interface 1 and 2 are close to the K–S relationship with misorientation $\Delta\theta^{\text{KS}} = 6^\circ$ and 11° , respectively. As is shown in **Figs. 6(d)** and **6(e)**, these grain boundary normals are close to the close-packed planes with a deviation of $\sim 17^\circ$ and $\sim 24^\circ$. Contrarily, at the interfaces with the orientation relationship deviated from the K–S relationship in **Figs. 6(f)** and **6(g)**, the coincidence between the grain boundary normals and the poles of the close-packed planes is not found. These results are consistent with those in **Fig. 5**.

The frequency of coincidence among the grain boundary normal and the close-packed planes of austenite and ferrite are analyzed for 97 interfaces related by the K–S relationship and approximate K–S relationship in the same way indicated above. Among those interfaces, the number of interfaces with the orientation relationships between the K–S relationship and the N–W relationship ($\Delta\theta^{\text{KS}} < 5.3^\circ$) is 41, and the other interfaces that are deviated from the K–S relationship by 6–12° is 56. Concerning the former interfaces, 83% of the interfaces are in the vicinity of the close-packed planes $\{111\}_\gamma // \{011\}_\alpha$. Concerning the latter interfaces, 60% of the interfaces are in the vicinity of $\{111\}_\gamma$ or $\{011\}_\alpha$.

3.3. TEM Observations for the Interface Microstructure

The morphology of the austenite interface at the grain corner satisfying the close orientation relationships to the K–S one is a key factor to investigate its initial formation and growth mechanism during the reverse transformation. Using the sample after quenching at 730°C, the microstructure of martensite dispersed in the ferrite phase has been examined at room temperature. Under the TEM observations, most of the martensite regions exist at the grain corners in the ferrite matrix, and no cementite is observed in the inside or boundary region of martensite. Most of cementite

Fig. 5. A three-dimensional morphology of an austenite grain (martensite region in the sample quenched at 730°C) and the analysis on the grain boundary normals at different interfaces in Fe-0.1C-1Mn alloy: (a)–(c) a reconstructed austenite grain observed from different three directions (the broken white lines are superimposed along the grain edge of the prior austenite to clarify the interfaces to different ferrite grains), (d)–(f) pole figures indicating the grain boundary normal as the contour map and the orientations for ferrite and austenite, each contour line corresponds to the pole density of the boundary normal vectors by 2.5 times to random distribution of an assumed spherical interface.

Fig. 6. A three-dimensional morphology of an austenite grain (martensite region in the sample quenched at 730°C) and the analysis on the grain boundary normals at different interfaces in Fe-0.1C-1Mn alloy.

particles precipitate at the intragrain or the grain boundary of ferrite. Concerning the dispersion of cementite particles in the present sample, the microstructure before the reverse transformation was observed in the previous study.³¹⁾ It has been shown that the cementite particles disperse randomly in the recrystallized ferrite matrix, and the cementite particles are not localized to the grain boundaries of ferrite in the present sample. Thus, it is considered that the initial austenite grain has been formed at the grain corner. It seems that there is no relation between the cementite distribution and nucleation of austenite in the present sample.

The morphology of martensite is lath structure with dislocations. The typical microstructure including the martensite and the surrounding ferrite matrix is shown in Fig. 7. The interface between martensite and Ferrite grain 1 is flat, which satisfies the edge on condition under the $[311]$ incident beam, as seen in Fig. 7(a). The dark field image taken by the $01\bar{1}$ reflection of Ferrite grain 1 is indicated in Fig. 7(b), and the trace of interface is perpendicular to the $[01\bar{1}]_{\alpha}$ direction in Ferrite grain 1. Taking into account the other interfaces, the interfaces to Ferrite grain 2 and 3 are also almost flat and the interface to Ferrite grain 4 is complex with large curvature.

4. Discussion

When the austenite phase appears at just above the A_{c1} temperature, most of the initial austenite grains are formed at the grain corners in the ferrite matrix. Since the heat treatment of the low carbon steels are mainly performed in the temperature region between A_{c1} and A_{c3} , the formation mechanism of the initial austenite is very important to control the subsequent microstructure.

In the present study, the interface boundary planes between the ferrite and austenite grains were investigated by the 3D EBSD measurement and the determination method of the normal direction vector of the voxel has been proposed. When the interface structure of martensite is not smooth, it is difficult to predict the morphology of austenite

boundary. Since the lath martensite in the low carbon steels nucleated at a grain boundary of austenite, it is considered that the smooth interface structure of austenite remains at room temperature after the martensitic transformation. The nature of the flat interface is also confirmed by the TEM analysis, as shown in Fig. 7(a). Under the TEM observations, lots of martensite regions having flat interfaces were observed at the corner of ferrite. These observation results support the crystallographic analysis for the interfaces using 3D EBSD. For the austenite grains shown in Figs. 5 and 6, the deviation of grain boundary normal from the close-packed planes are summarized in Table 2. The Interface 1 of austenite grain 1 in Fig. 5 is considered to be the habit plane, and formed at the initial stage of the reverse transformation. The close relationship between the grain boundary normal and the coincident point of $(111)_{\gamma} // (011)_{\alpha}$ are also close each other in the case of Interface 2 as seen in Fig. 5(e). The deviation of the grain boundary normal from the close-packed planes is about 6° .

It is concluded that the interface with the orientation relationships between the K–S relationship and the N–W relationship is in the vicinity of the close-packed planes of ferrite and austenite ($\{111\}_{\gamma} // \{011\}_{\alpha}$). The interface with the orientation relationship that is deviated from the K–S relationship by $6\text{--}12^{\circ}$ also tends to be near $\{111\}_{\gamma}$ or $\{011\}_{\alpha}$. It is important that the grain boundary normal is a little bit deviated from the close-packed plane of $\{111\}_{\gamma} // \{011\}_{\alpha}$, as seen in Figs. 6(d) and 6(e).

In the literatures, the interface boundary structures with specific orientation relationships, such as the K–S relationship and N–W relationship, were studied by microscopic observations.^{9,12,14)} It was observed that the boundaries contain arrays of misfit dislocations and parallel structural ledges, in which the apparent habit planes deviate significantly from $\{011\}_{\text{bcc}} // \{111\}_{\text{fcc}}$.

In the *in situ* EBSD measurement, the interfaces related by the K–S relationship hardly migrated. To clarify the details of their interface nature, two types of misorientations are measured. One is the misorientation between the

Fig. 7. TEM micrograph of the ferrite-martensite microstructure and orientation analysis at the interface in Fe-0.1C-1Mn alloy quenched at 730°C in the heat treatment ((a) Bright-field image, (b) Dark-field image taken by $01\bar{1}$ reflection from Ferrite grain 1, (c) The diffraction pattern of $01\bar{1}$ reflection from Ferrite grain 1).

Table 2. Orientation relationships and the deviation of grain boundary normal from the close-packed planes of the phases at each interfaces of two martensite regions (martensite region 1 and 2 correspond to those shown in Figs. 5 and 6, respectively).

Austenite grain	Interface	Misorientation from the K-S, $\Delta\theta_{KS}/\text{deg.}$	Misorientation between $\{111\}\gamma$ and $\{011\}\alpha$, $\Delta\theta_{cpp}/\text{deg.}$	Misorientation between $\langle 011 \rangle \gamma$ and $\langle 111 \rangle \alpha$, $\Delta\theta_{cpd}/\text{deg.}$	Deviation of boundary normal from the close-packed planes	
					Deviation/deg.	Location to the c.p.p
1	Interface 1	0.8	0.4	0.8	$\sim 5^\circ$	In the vicinity
1	Interface 2	2.7	0.5	2.6	$\sim 6^\circ$	In the vicinity
1	Interface 3	8.0	7.7	7.0	Large	Not related
1	Interface 4	21.7	19.8	11.6	Large	Not related
2	Interface 1	6.3	0.8	6.2	$\sim 17^\circ$	In the vicinity
2	Interface 2	11.8	2.6	11.6	$\sim 24^\circ$	In the vicinity
2	Interface 3	22.1	16.7	18.4	Large	Not related
2	Interface 4	26.8	23.1	12.1	Large	Not related

Fig. 8. The misorientation of the close-packed planes and close-packed directions at α/γ interfaces measured using *in situ* EBSD of Fe-0.2C alloy.

close-packed-planes, $\Delta\theta_{cpp}$, and the other is that between the close-packed-directions, $\Delta\theta_{cpd}$. Among the symmetric families of directions $\langle 111 \rangle_\gamma$ and $\langle 011 \rangle_\alpha$, and planes $\{111\}_\gamma$ and $\{011\}_\alpha$, the closest pairs are selected and the misorientations $\Delta\theta_{cpp}$ and $\Delta\theta_{cpd}$ are calculated using the following equations:

$$\Delta\theta_{cpp} = \arccos(((\mathbf{g}^\gamma)^{-1} \mathbf{P}_{cpp}^\gamma) \cdot ((\mathbf{g}^\alpha)^{-1} \mathbf{P}_{cpp}^\alpha)) \dots\dots (4)$$

$$\Delta\theta_{cpd} = \arccos(((\mathbf{g}^\gamma)^{-1} \mathbf{P}_{cpd}^\gamma) \cdot ((\mathbf{g}^\alpha)^{-1} \mathbf{P}_{cpd}^\alpha)) \dots\dots (5)$$

Here, $\mathbf{P}_{cpp}$ and $\mathbf{P}_{cpd}$ are the vectors of the normal direction of the close-packed plane and the close-packed direction respectively. The relationship between the two types of misorientations is shown in **Fig. 8**. In the graph, the interface that migrated by more than $1 \mu\text{m}$ between 730°C and 790°C is indicated by open circles and those that migrated by less than $1 \mu\text{m}$ are indicated by solid circles. The mis-

orientations of the orientation relationships are concentrated in the specific range that lies between 0° and 5° in $\Delta\theta_{cpd}$ while maintaining $\Delta\theta_{cpp}$ below 2° , which correspond to the orientation relationships between the K–S and N–W relationship. In addition, the orientation relationships around $(\Delta\theta_{cpd}, \Delta\theta_{cpp}) = (2.5^\circ, 2.0^\circ)$, which correspond to the Greninger–Troiano relationship,⁴⁵⁾ are frequently observed. A clear tendency is found that low mobile interfaces are related to the orientation relationships with a less misorientation between the close-packed planes than that between the close-packed directions. That is, the interface satisfying the relationships close to the K–S relationship shows low mobility.

It is suggested that the interfaces with these specific orientation relationships comprise selected interface boundary planes of ferrite and austenite, which renders the interfaces less mobile, which is consistent with the results obtained in the 3D EBSD analysis and the TEM observation. The boundary structure with the selected interfacial planes is expected to be the reason for the low mobility of the interfaces.

5. Conclusions

The crystallographic characterization of the interfaces of the austenite phase transformed from the ferrite phase at just above of A_{c1} temperature has been investigated by 3D EBSD measurements and TEM observations of the ferrite-martensite microstructure obtained at room temperature. The formation of austenite microstructure has been directly measured by *in situ* observation using SEM-EBSD at high temperature. Based on these experimental results, the mobility of the α/γ boundary and its 3D microstructure is discussed with the interface boundary planes and reverse transformation crystallography. The following conclusions are obtained.

(1) When an austenite grain is transformed from the ferrite on heating with satisfying a small deviation from the K–S relationship in their orientation relationship between grains, one or two of the grain boundaries of austenite has the characteristic crystal orientation relationship, in which the grain boundary normal tends to be in the vicinity of the coincident point of common close-packed planes $\{111\}_\gamma //$

$\{011\}_{\alpha}$.

(2) The determination method of the grain boundary normal direction is introduced to discuss the property of the interface plane obtained by 3D EBSD analysis.

(3) In the TEM observation of the ferrite-martensite microstructure, the flat interfaces are often observed between martensite and adjacent ferrite grain and the typical trace of interface is perpendicular to $[011]_{\alpha}$ in a ferrite grain orientation. The result supports the observations obtained by the grain boundary normal analysis using the 3D EBSD reconstruction.

(4) The migration of the α/γ interface with the orientation relationship close to the K–S relationship (within 9°) is significantly suppressed during the growth process of austenite grains at 730°C . The normal directions of the close-packed plane and direction in the ferrite and austenite are close to the condition satisfying the K–S relationship within 5° . The interfaces are expected to form high coherent structure with specific boundary planes by which the interfaces become immobile in the growth of austenite in the reverse transformation.

Acknowledgements

The authors are grateful to Dr. Tomida of Ibaraki prefecture government for many valuable suggestions concerning the calculation method on crystallographic analysis applied in this work. The authors would like to express their gratitude to Dr. Nakagawa of Nippon Steel and Sumitomo Metal Co., Ltd. and to Mr. Nakamura of NS Solutions Co., Ltd. for their valuable discussions regarding this work.

REFERENCES

- G. R. Speich, V. A. Demarest and R. L. Miller: *Metall. Trans. A*, **12** (1981), 1419.
- D. Z. Yang, E. L. Brown, D. K. Matlock and G. Krauss: *Metall. Trans. A*, **16** (1985), 1385.
- N. Nakada, T. Tsuchiyama, S. Takaki and S. Hashizume: *ISIJ Int.*, **47** (2007), 1527.
- T. Hara, N. Maruyama, Y. Shinohara, H. Asahi, G. Shigesato, M. Sugiyama and T. Koseki: *ISIJ Int.*, **49** (2009), 1792.
- S. Watanabe and T. Kunitake: *Tetsu-to-Hagané*, **61** (1975), 96.
- K. Ameyama, T. Maki and I. Tamura: *J. Jpn. Inst. Met.*, **50** (1986), 602.
- M. G. Hall, H. I. Aaronson and K. R. Kinsma: *Surf. Sci.*, **31** (1972), 257.
- J. M. Rigsbee and H. I. Aaronson: *Acta Metall.*, **27** (1979), 351.
- J. M. Rigsbee and H. I. Aaronson: *Acta Metall.*, **27** (1979), 365.
- M. G. Hall, J. M. Rigsbee and H. I. Aaronson: *Acta Metall.*, **34** (1986), 1419.
- U. Dahmen: *Scr. Metall.*, **21** (1987), 1029.
- T. Furuhashi, A. M. Dalley and H. I. Aaronson: *Scr. Metall.*, **22** (1988), 1509.
- H. I. Aaronson, T. Furuhashi, J. M. Regisbee, W. T. Reynolds, Jr. and J. M. Howe: *Metall. Trans. A*, **21** (1990), 2369.
- T. Furuhashi and T. Maki: *Mater. Trans. JIM*, **33** (1992), 734.
- Q. Liang and W. T. Reynolds, Jr.: *Metall. Mater. Trans. A*, **29** (1998), 2059.
- T. Furuhashi, K. Oishi and T. Maki: *Metall. Mater. Trans. A*, **33** (2002), 2327.
- T. Nagano and M. Enomoto: *Metall. Mater. Trans. A*, **37** (2006), 929.
- G. Kurdjumov and G. Sachs: *Z. Phys.*, **64** (1930), 325.
- Z. Nishiyama: *Sci. Rep. Tohoku Imp. Univ.*, **23** (1934), 638.
- K. Ameyama, T. Maki and I. Tamura: *J. Jpn. Inst. Met.*, **50** (1986), 602.
- K. Ameyama, G. C. Weatherly and K. T. Aust: *Acta Metall. Mater.*, **40** (1992), 1835.
- T. Furuhashi and T. Maki: *Mater. Sci. Eng. A*, **312** (2001), 145.
- Y. Adachi, K. Hakata and K. Tsuzaki: *Mater. Sci. Eng. A*, **412** (2005), 252.
- T. Takeuchi, Y. Adachi, D. Dorner and M. Enomoto: *Mater. Sci. Forum*, **561-565** (2007), 69.
- G. H. Zhang, T. Takeuchi, M. Enomoto and Y. Adachi: *Metall. Mater. Trans. A*, **42** (2011), 1597.
- T. Tomida, N. Imai, K. Miyata, S. Fukushima, M. Yoshida, M. Wakita, M. Etou, T. Sasaki, Y. Haraguchi and Y. Okada: *ISIJ Int.*, **48** (2008), 1148.
- T. Tomida, M. Wakita, M. Yasuyama, S. Sugaya, Y. Tomota and S. C. Vogel: *Acta Mater.*, **61** (2013), 2828.
- I. Lischewski and G. Gottstein: *Mater. Sci. Forum*, **495-497** (2005), 447.
- I. Lischewski and G. Gottstein: *Acta Mater.*, **59** (2011), 1530.
- K. Hata, M. Wakita, K. Fujiwara, K. Kawano, T. Tomida, M. Sugiyama, T. Fukuda and T. Kakeshita: *Mater. Trans.*, **57** (2016), 1514.
- K. Hata, K. Fujiwara, K. Kawano, M. Sugiyama, T. Fukuda and T. Kakeshita: *ISIJ Int.*, **58** (2018), 323.
- H. I. Aaronson, T. Furuhashi, J. M. Regisbee, W. T. Reynolds, Jr. and J. M. Howe: *Metall. Trans. A*, **21** (1990), 2369.
- M. Enomoto: *Acta Metall.*, **35** (1987), 935.
- M. Enomoto and H. I. Aaronson: *Scr. Metall.*, **23** (1989), 1983.
- T. Fukino and S. Tsurekawa: *Mater. Trans.*, **49** (2008), 2770.
- G. Miyamoto, R. Hori, B. Poorganji and T. Furuhashi: *Metall. Mater. Trans. A*, **44** (2013), 3436.
- S. Zaefferer, S. I. Wright and D. Raabe: *Metall. Mater. Trans. A*, **39** (2008), 374.
- M. Humbert, H. Moustahfid, F. Wagner and M. J. Philippe: *Scr. Metall. Mater.*, **30** (1994), 377.
- M. Humbert and N. Gey: *J. Appl. Crystallogr.*, **35** (2002), 401.
- C. Cayron, B. Artaud and L. Briottet: *Mater. Charact.*, **57** (2006), 386.
- T. Morimoto, F. Yoshida, I. Chikushi, H. Kitahara and N. Tsuji: *Tetsu-to-Hagané*, **93** (2007), 591.
- G. Miyamoto, N. Iwata, N. Takayama and T. Furuhashi: *Acta Mater.*, **58** (2010), 6393.
- K. Hata, M. Wakita, K. Fujiwara and K. Kawano: *Nippon Steel & Sumitomo Met. Tech. Rep.*, **114** (2017), 26.
- Z.-D. Li, G. Miyamoto, Z.-G. Yang and T. Furuhashi: *Scr. Mater.*, **60** (2009), 485.
- A. Greninger and A. Troiano: *Trans. AIME*, **185** (1949), 590.