Reconstruction of Parent Austenite Grain Structure Based on Crystal Orientation Map of Bainite with and without Ausforming

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By using a reconstruction program recently developed by the present authors, the parent austenite grain structure is reconstructed from the crystal orientation map of bainite structure. Local austenite orientation can be reconstructed from non-ausformed bainite structure with errors of 1.7° and 1.0° at spatial resolutions of 5 and 20 μm respectively. The angle of errors is further reduced to be less than 0.5° when an analysis area is extended to a whole austenite grain. Application of the reconstruction program to the bainite structure transformed from deformed austenite has clarified that the variation in grain shape and deformation texture of parent austenite can be well reconstructed.

KEY WORDS: bainite; texture; orientation relationship; ausforming; EBSD.

1. Introduction

Transformation into martensite from work-hardened austenite (γ) has been known to increase strength without significant deterioration of elongation.¹⁾ This process has been called as ausforming. The inheritance of high density of dislocation in γ to martensite and variations in microstructure size and transformation texture are considered to be responsible for improving mechanical properties.^{1,2)} The effects of this ausforming on bainite transformation have also been investigated from the view points of mechanical properties,^{3–5)} block or packet sizes,⁶⁾ variant selection^{7,8)} and transformation kinetics.^{8,9)}

In order to understand the effects of ausforming on martensite or bainite transformation in detail, the microstructure of the parent γ , such as grain shape, crystal orientation and deformation substructure, are needed to be clarified. If the untransformed γ is retained at room temperature, they can be analyzed directly.^{8,10)} However, the volume fraction of retained γ is very low in ordinary low-alloy low-carbon steels.

In order to investigate the parent grain structure, reconstruction methods have been developed by some researchers in Ti and Zr alloys^{11–15)} or steels.^{16–18)} These methods are based on the crystal orientation map, which is measured by electron back scattering diffraction (EBSD), of the product phase transformed from a non-deformed parent phase. They assume a rational orientation relationship (OR) between the parent and product phases, such as Kurdjumov-Sachs or Nishiyama-Wasserman relationships for fcc to bcc transformation, then calculate the orientation of the parent phase. In contrast, the present authors¹⁹⁾ have recently developed a new program to reconstruct a γ orientation map based on an orientation map of bcc martensite, in which an OR between γ and martensite is assumed to have non-parallel relations in

close-packed planes and directions.²⁰⁾ The same program was applied to ausformed martensite, and a deformed γ structure was successfully visualized. However, the question whether the deformation in γ that has appeared as misorientation in reconstructed γ is due to ausforming or to plastic accommodation of transformation strain in γ ²¹⁾ still remains unresolved.

In this paper, effects of parameters used in the reconstruction procedure on accuracy in determining γ orientation from non-ausformed bainite structure have been examined from the view point of orientation spread within austenite grains. Then γ orientation maps have been reconstructed from ausformed bainite structure in order to discuss the origin of the misorientation in reconstructed γ .

2. Experimental Procedure

Fe–0.15C–3Ni–1.5Mn–0.5Mo (mass%) alloy was used in this study. The alloys were homogenized at 1453 K for 259.2 ks. Cylindrical specimens, 8 mm in diameter and 12 mm in height, were cut from the homogenized specimen and heat-treated using a thermecmaster as follows. The specimens were austenitized at 1473 K for 0.6 ks and subsequently compressed at 973 K by 10, 30 or 50% at a strain rate of 1 s^{–1}. They were then cooled by He gas to 673 K, held for 3.6 ks to isothermally transform into bainite structure and gas-quenched to room temperature. These specimens are referred to as 10, 30 and 50%-ausformed specimens, respectively. A non-ausformed bainite specimen was also made for comparison. In order to confirm validity of reconstructed γ orientation maps, γ -stabilized Fe–25Ni–0.5C (mass%) alloy, which is homogenized at 1423 K for 345.6 ks, was also examined. The alloy was austenitized at 1323 K for 0.6 ks and subsequently deformed at 973 K by 30% at a strain rate of 1 s^{–1} followed by He gas quenching.

The ferrite (α) and γ orientations were measured by EBSD at an accelerating voltage of 25 kV and a step size of 0.5 μm or 2 μm . Only orientation data with confidence index (CI)⁽²²⁾ values higher than 0.1 were used for the OR determination and reconstruction of γ orientation. The specimens for the EBSD measurements were prepared by mechanical polishing using colloidal silica, followed by Ar ion milling at 3 keV.

3. Reconstruction Procedure

In the reconstruction program developed in the previous study,⁽¹⁹⁾ an α orientation map measured by EBSD at a certain step size is analyzed. The measured data set of α orientations in a small mesh, of which edge length is L_1 , are here represented by the orientation matrix, M_i^{BCC} , of the i -th measurement point in the mesh. In the determination of average γ orientation in the mesh, the deviation matrix (D_i) between the experimental and calculated γ orientations is estimated for each M_i^{BCC} by assuming the γ orientation matrix (M^{FCC}) and the OR matrix between bcc and fcc ($V^{F \rightarrow B}$) as:

$$D_i = V^{F \rightarrow B} (C_n M^{FCC}) (C_m M_i^{BCC})^{-1} \dots\dots\dots (1)$$

where C_n and C_m are the conversion operation matrices of the basic axes of cubic crystals. C_n and C_m are chosen for the misorientation angle of D_i ($\Delta\theta_i$) being minimal for each i -th measurement point. The average misorientation ($\Delta\theta_{average}$) on assuming the γ orientation and the OR in question is given by:

$$\Delta\theta_{average} = \sum_N \Delta\theta_i / N \dots\dots\dots (2)$$

where N is the number of data in a single mesh. Finally, M^{FCC} and $V^{F \rightarrow B}$ are determined by minimizing $\Delta\theta_{average}$ by numerical fitting. By changing the position of mesh centers by a given distance (L_2) from that of a neighboring mesh and repeating the above procedure, the γ orientation map is reconstructed.

However, the quantity of data taken from the small mesh is not sufficient to determine both the orientation of the γ and the OR between bainite and γ . Thus, the OR is first determined by analyzing a large area of non-ausformed bainite, and then reconstruction of the γ orientation is carried out by using the determined OR. The determined OR matrix, which is defined as the orientation matrix of a variant of bainitic ferrite in the coordinate system of parent γ , is expressed by Euler angles in Bunge's form⁽²²⁾ as (120.72°, 8.88°, 195.38°). The angular deviations between γ and bainite in close-packed planes and directions are 1.32° and 2.90°, respectively. This OR is used throughout this study, since OR does not vary significantly by the deformation prior to the bainite transformation.⁽¹⁰⁾

4. Results and Discussions

Since mesh size (L_1) determines the number of α orientation data in each mesh, it would strongly influence the accuracy of γ orientation determination. Therefore, first, the effects of L_1 on the accuracy were investigated by changing L_1 from 5 to 20 μm with keeping a ratio of L_1 to L_2 to be two. Fig. 1(a) shows the α orientation map of non-ausformed bainite measured at a step size of 2 μm and Figs. 1(b) to 1(d) are γ orientation maps reconstructed from Fig. 1(a) by using different mesh size L_1 . In the γ orientation maps, γ grain boundaries and annealing twin boundaries are represented by black and white lines, respectively. As shown in Fig. 1(b), the γ orientation map reconstructed by using 5 μm for L_1 represents γ grains and annealing twin boundaries clearly, although it contains many black dots inside the γ grains. These dots represent the failure in fitting, for which the value of $\Delta\theta_{average}$ obtained using Eq. (2) is larger than 5°. In addition, some points inside the γ grains are misindexed as twin oriented grains to the matrix as already reported in the previous paper.⁽¹⁹⁾ These errors are caused by a small number of bainite variants contained in each mesh. By increasing L_1 to be 7 μm (Fig. 1(c)) and 20

Fig. 1. (a) α orientation map of non-ausformed bainite measured at a step size of 2 μm . (b)–(d) γ orientation maps reconstructed from (a) by using (b) $L_1 = 5 \mu\text{m}$ and $L_2 = 2.5 \mu\text{m}$, (c) $L_1 = 7 \mu\text{m}$ and $L_2 = 3.5 \mu\text{m}$, and (d) $L_1 = 20 \mu\text{m}$ and $L_2 = 10 \mu\text{m}$. In (b), (c) and (d), black and white lines describe high angle and twin boundaries, respectively. Black dot represents failure in fitting, where the value of $\Delta\theta_{average}$ obtained using Eq. (2) is larger than 5°.

μm (Fig. 1(d)), the number of failure and misindexed points decreases significantly due to increasing a number of data in each mesh, whereas spatial resolution of the orientation map becomes poorer, so that black regions corresponding to γ grain boundaries become thicker and zigzag in alignment.

Figures 2(a) to 2(c) show γ inverse pole figures calculated from Figs. 1(b) to 1(d) respectively. Each group of data points corresponds to orientation data from a single γ grain. By comparing Figs. 2(a) ($L_1 = 5 \mu\text{m}$), 2(b) ($L_1 = 7 \mu\text{m}$) and 2(c) ($L_1 = 20 \mu\text{m}$), it is found that the spread of γ orientation decreases with increasing L_1 . This tendency was quantitatively analyzed by adopting the “grain orientation spread” method and is shown in Figs. 2(d) to 2(f). The grain orientation spread is obtained as follows by using TSL[®] OIM analysis;²³⁾ the average orientation for each γ grain is first calculated. The spread is then the average deviation between the orientation of each point in the grain and the average orientation for the grain. As shown in Fig. 2(d), the grain spread angle is mostly distributed from 1.5° to 2° , and its

mean value is 1.7° when $5 \mu\text{m}$ is used for L_1 . By increasing L_1 (Figs. 2(e) and 2(f)), angles of grain spread decrease and the mean value of them reaches 1.0° at L_1 of $20 \mu\text{m}$ (Fig. 2(f)).

In order to achieve better accuracy for the determination of parent γ orientation, the quantity of orientation data for the analysis was increased by decreasing the step size in EBSD measurement and increasing the area for the analysis. **Figure 3(a)** shows the α orientation map of a non-ausformed bainite specimen measured at the step size of $0.5 \mu\text{m}$. A single γ region without containing annealing twins was selected manually from this map and divided into two regions as indicated by the regions A and B. **Figures 3(b) and 3(c)** show $\{001\}_\alpha$ pole figures measured for the regions A and B in Fig. 3(a), on which the estimated $\langle 001 \rangle_\alpha$ of bainite by fitting for each region are superimposed. The number of orientation data to determine one γ orientation increases from about one hundred (Fig. 1(d)) to tens of thousands. The $\langle 001 \rangle_\alpha$ directions for the regions A and B shown in Figs. 3(b) and 3(c) appear quite similar, thus the $\langle 001 \rangle_\gamma$ directions obtained by the fitting are also nearly the same in the two regions. The misorientation angle of the parent γ calculated between the regions A and B is 0.74° . Such analyses were carried out for more than ten different γ regions and the mean misorientation angle between two regions divided is obtained to be 0.65° . Since the misorientation between two regions includes errors arising in determination of orientations for both regions, it is concluded that the orientation of each region can be determined with an average error of 0.33° . Such accurate determination of the parent γ orientation has been recently applied to analyze OR between reversely formed γ and pearlitic α in high carbon steel²⁴⁾ or between γ and grain boundary α in medium carbon steels.²⁵⁾

Figures 4(a), 4(b) and 4(c) show γ orientation maps reconstructed from α orientation maps of ausformed bainite, of which deformation strains applied to γ are 10%, 30% and 50% respectively. After trial and error, $7 \mu\text{m}$ and $3.5 \mu\text{m}$ were chosen for L_1 and L_2 , respectively, in reconstruction of γ from ausformed bainite because deformation structures in γ reconstructed by using these values were clearer than other conditions investigated. The compression axis (CA) was taken as the reference axis for the color orientation maps according to the stereographic triangle shown in Fig. 4(a). As shown in Fig. 4(a), the deformation by 10% hardly changes γ grain shape although a gradual lattice rotation of γ is partly observed as indicated by black arrows. By

Fig. 2. Inverse pole figure of CA direction in γ corresponding to Fig. 1, (a) $L_1 = 5 \mu\text{m}$ and $L_2 = 2.5 \mu\text{m}$, (b) $L_1 = 7 \mu\text{m}$ and $L_2 = 3.5 \mu\text{m}$, and (c) $L_1 = 20 \mu\text{m}$ and $L_2 = 10 \mu\text{m}$. (d), (e) and (f) are distributions of grain orientation spread in γ corresponding to (a), (b) and (c), respectively.

Fig. 3. (a) α orientation map of non-ausformed bainite structure measured at a step size of $0.5 \mu\text{m}$ and $\{001\}_\alpha$ pole figures taken from the regions (b) A and (c) B, on which $\langle 001 \rangle_\alpha$ directions obtained by fitting are superimposed. Solid and broken lines in (a) indicate prior γ grain boundaries and annealing twin boundaries, respectively.

Fig. 4. Orientation maps of γ reconstructed from (a) 10%-, (b) 30%-, (c) 50%-ausformed bainite specimens, and (d), (e), (f) inverse pole figures of CA direction in γ corresponding to (a), (b), (c), respectively. L_1 and L_2 used are $7\mu\text{m}$ and $3.5\mu\text{m}$, respectively. Details of γ orientation map are described in the caption of Fig. 1.

Fig. 5. (a) Measured orientation map of γ in the Fe-25Ni-0.5C alloy deformed at 973 K by 30% (step size; $2\mu\text{m}$) and (b) inverse pole figure of CA direction in γ CA was taken as the reference axis for the color orientation maps according to a stereo triangle shown in Fig. 4(a). Details of γ orientation map are described in the caption of Fig. 1.

increasing the deformation strain to 30% (Fig. 4(b)) and 50% (Fig. 4(c)), γ grains become elongated perpendicularly to CA, and orientation change inside the γ grains becomes substantial. As indicated by open arrows, banded structures inclined by about 45° to the CA direction are also seen. The frequency of failure in reconstruction represented by black points increases with strain because a large and local misorientation in γ introduced by the deformation results in increase in $\Delta\theta_{\text{average}}$ in Eq. (2). Figure 4(d) represents the inverse pole figure for the CA direction in γ reconstructed for the 10% ausformed bainite specimen, which corresponds to Fig. 4(a). By comparing it with Fig. 2(b), it is clearly seen that the orientation spread of each γ grain becomes much larger by the deformation of 10%. The crystal lattice of γ tends to rotate into a specific direction by further deformation, causing a development of deformation texture. The γ inverse pole figures calculated from 30%- (Fig. 4(e)) and 50%- (Fig. 4(f)) ausformed specimens clearly indicate that the $\langle 011 \rangle_\gamma$ component becomes much stronger than the $\langle 111 \rangle_\gamma$ and $\langle 001 \rangle_\gamma$ components.

In order to confirm the validity of γ orientation map reconstruction, deformation structure in the γ -stabilized alloy was examined. **Figures 5(a)** and **5(b)** show the γ orientation map of the Fe-25Ni-0.5C alloy deformed at 973 K by 30% and the inverse pole figure of CA direction in γ , respectively. Elongated γ grains and banded structures inclined by about 45° to CA are observed, which is similar to those in the reconstructed γ orientation map in Fig. 4(b). It was found by trace analyses that these bands tended to be introduced along $\{111\}_\gamma$ planes. Such deformation band structures were similarly observed in a hot-rolled austenitic stainless steel.²⁶⁾ Furthermore, the deformation texture of γ shown in Fig. 5(b) agrees quite well with that reconstructed (Fig. 4(e)), although the intensity of $\langle 011 \rangle_\gamma$ peak in Fig. 5(b) is higher than that in Fig. 4(e). Such deformation texture of γ coincides also well with the deformation texture reported in fcc metal after compression rolling²⁷⁾ and in γ of the Fe-30%Ni specimen compressed at 573 K.²⁸⁾

Displacive transformation such as martensite and bainite transformations causes a lattice rotation in surrounding γ

due to accommodation of transformation strain.²¹⁾ However, the present reconstruction method reproduces the spreading of γ orientation in each γ grain and the development of deformation texture by ausforming successfully. This fact clearly supports that the lattice rotation in γ due to the ausforming can be reconstructed by the present method. Therefore, whether the misorientation in γ reconstructed from ausformed specimens is due to ausforming or to accommodation of transformation strain should be the subject under discussion only at a small applied strain less than 10%. By comparing the orientation spread in γ reconstructed from the 10%-ausformed specimen (Fig. 4(d)) with the orientation spread in the non-ausformed specimen (Fig. 2(b)), it is further concluded that the contribution of plastic accommodation of transformation strain to the misorientation in reconstructed γ is smaller than that by ausforming even at the strain of 10%.

5. Summary

In the present study, the reconstruction method of parent γ orientation, which has been developed by the present authors,¹⁹⁾ was applied to analyze bainite structure with and without ausforming. In application to the non-ausformed specimen, it was found that the local orientation of γ could be obtained with errors 1° by using a large square analysis area of $20\text{ }\mu\text{m}$ in side length. When the analysis area is extended to a whole γ grain, such angle of errors decreases less than 0.5° . In the application to the ausformed bainite structure, γ grains elongated perpendicularly to the compression axis and deformation texture in γ were reconstructed. These features in reconstructed γ agree well with those observed in a γ -stabilized alloy deformed at the same condition.

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