NIPPON STEEL & SUMITOMO METAL TECHNICAL REPORT No. 114 MARCH 2017

Technical Report

UDC 621 . 785 . 36

Development of a Reconstruction Method of Prior Austenite Microstructure Using EBSD Data of Martensite

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Abstract

A method for reconstructing a prior austenite microstructure from a martensite microstructure using EBSD data has been developed. Structure of prior austenite with its orientation is obtained through a series of calculation to find a group of neighboring martensite variants that have the common prior austenite orientation. The calculation is based on the Kurdjumov-Sachs (K-S) orientation relationship, which try to choose the actual K-S orientation relationship from among the 24 possible relationships. This new method has enabled us to reconstruct the prior austenite microstructure in a large area, which provide us with not only the average grain size but also the texture of prior austenite at high temperature without in-situ measurements.

1. Introduction

The phase transformation from the austenite phase (γ) to ferrite phase (α) is a very important phenomenon to control the microstructures of steel products with appropriate mechanical properties. The formation of microstructures of many steel products is greatly influenced by the austenite microstructure before phase transformation. Therefore, information about the austenite microstructure is very important in controlling the microstructures of steel products. In recent years, demand for steel products with higher strength has increased and bainite and martensite microstructures being more widely used than ever before. The importance of controlling the austenite microstructure that strongly influences the packet and the block in those microstructures is growing.

To acquire the information about austenite microstructure conveniently, reconstruction methods by analyzing the data of crystal orientations of martensite and or bainite have been researched and developed [1–5]. Several programs for reconstructing austenite have already been provided in commercial use. In those methods, analysis based on the specific crystal orientation relationship between ferrite and austenite of steel such as the Kurdjumov-Sachs (K-S) relationship [6] are considered, and orientations of austenite before phase transformation can be determined by analyzing the relationship among the variants of martensite or bainite.

Nippon Steel & Sumitomo Metal Corporation has developed an analysis method based on the K-S relationship for automatic reconstruction of austenite polycrystalline microstructure. This method is realized by statistic analysis of the crystal orientation data of multivariant microstructures of martensite or bainite obtained through Electron Back-scatter Diffraction (EBSD) measurement. This article reports the details of the analysis method of reconstructing prior austenite. The accuracy of the austenite reconstruction by the method is also shown with some examples of application.

2. Main Discourse

2.1 Analytic method of austenite orientation based on crystal orientation of martensite variants

Humbert et al. proposed a calculation method of analyzing the crystal orientations of the parent phase before transformation based on the crystal orientation relationship. They have shown that the orientations of the parent β phase in Ti alloys can be obtained by analyzing the crystal orientations of α phase variants based on the Burgurs relationship [4, 5]. Various methods to reconstruct the parent phase that are reported later on similarly employed the method of Humbert et al.

In the austenite reconstruction method reported in this article, in accordance with the method proposed by Humbert et al., the crystal orientation relationship between the parent austenite phase and a ferrite phase variant is expressed with 3×3 rotation matrices based

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on the K-S relationship as following [4, 5],

$$\mathbf{R}_j \mathbf{g}^{\alpha} = \mathbf{V}_k \mathbf{R}_i \mathbf{g}^{\gamma} \quad (1)$$

where $\mathbf{g}^{\alpha}$ and $\mathbf{g}^{\gamma}$ are the rotation matrix from the sample coordinate system to the ferrite crystal coordinate system and to the austenite crystal coordinate system, respectively. $\mathbf{V}_k$ (k = 1, 2, ⋯, 24) is the rotation matrix to convert the austenite crystal coordinate system to the ferrite crystal coordinate system based on the K-S relationship. $\mathbf{V}_k$ can be expressed as the rotation by 90 degrees around the <112> axis in the austenite crystal coordinate system [8]. In literatures, it is reported that the precise orientation relationship between martensite and austenite is not the exact K-S relationship but the orientation relationship deviated from the K-S relationship by a few degrees [2]. Therefore, $\mathbf{V}_k$ in the present study is the medial orientation relationship between the K-S relationship and the Nishiyama-Wasserman (N-W) relationship [7] instead of the exact K-S relationship. $\mathbf{R}_i$ (or $\mathbf{R}_j$) (i, j = 1, 2, ⋯, 24) are a group of rotation matrices of 90 degrees around <001> axis in crystal coordinate system, which are multiplied to take account of the cubic lattice symmetry of austenite and ferrite.

The austenite orientation can be expressed as (2).

$$\mathbf{g}^{\gamma} = (\mathbf{V}_k \mathbf{R}_i)^{-1} \mathbf{R}_j \mathbf{g}^{\alpha} \quad (2)$$

Since the K-S relationship includes 24 equivalent crystal orientations that are referred as variants, $\mathbf{V}_k$ consists of 24 different matrices. Therefore, with the expression (2) only, the austenite orientation $\mathbf{g}^{\gamma}$ cannot be determined.

It is reported in the literatures [4] that, in order to identify the orientation of prior austenite, the abovementioned relationship must be considered with at least three variants that were transformed from the same austenite grain [4]. When three austenite orientations derived from different variants agree with each other, the austenite orientation can be identified. Misorientation θ between the austenite orientations obtained from different variants $\mathbf{g}^{\alpha 1}$ and $\mathbf{g}^{\alpha 2}$ is evaluated by using the relations (3) and (4). The set of indices i and k are determined as one set which brings the misorientation θ within a predefined tolerance angle.

$$\mathbf{M}^{\gamma 1 - \gamma 2} = \mathbf{g}^{\gamma 1^{-1}} \mathbf{g}^{\gamma 2} = ((\mathbf{V}_k \mathbf{R}_i)^{-1} \mathbf{g}^{\alpha 1})^{-1} (\mathbf{V}_l \mathbf{R}_j)^{-1} \mathbf{g}^{\alpha 2} \quad (3)$$

$$\theta = \cos^{-1}((M_{11} + M_{22} + M_{33} - 1)/2) \quad (4)$$

As a result, $\mathbf{V}_k$ is determined from the 24 possible matrices, which means that the orientation relationship between the parent phase and the phase transformed is fixed to one, and the austenite orientation $\mathbf{g}^{\gamma}$ can be obtained from relationship (2). When several martensite variants have a common austenite as the parent phase, the austenite orientations calculated from the relationship (2) should agree with each other. Therefore the value of misorientation θ would be theoretically zero. However in reality, θ becomes a finite value due to the influence of the following artifacts: the error that lies between the set crystal orientation relationship and the actual crystalline orientation relationship, errors in measuring orientation by EBSD. Therefore, setting the tolerance angle at a finite value is necessary for the analysis method.

2.2 Reconstruction of an austenite grain from plural martensite variants

By expanding Humbert's analysis similarly to other adjacent martensite variants and assessing the misorientation θ, it is possible to judge whether the martensite variants belong to the common austenite. Furthermore, by continuing this process to all the adjacent variants as long as the common parent austenite orientation is found between them, a group of variants which has the common austenite orientation is obtained, which produces the whole region of a prior austenite grain to which these variants belongs.

However, when analyzing a prior austenite grain with this method, the accuracy of the analysis greatly depends on the magnitude of the tolerance angle which was set artificially since it decides whether or not each variants belongs to the common austenite. If the tolerance angle is set at an excessively large value, variants transformed from other different austenite grains may be erroneously analyzed as one of the variants belonging to the common austenite.

An example of this error processing is demonstrated using EBSD data in the following. 0.2%C-2%Mn steel was used for the analysis. The steel was hot-rolled, cold-rolled and heated up to 900°C and held for 100 seconds and then water-quenched. EBSD measurement was conducted at the position of 3/4 in the sheet thickness direction on the cross section perpendicular to the transverse direction. The image quality mapping and the orientation mapping obtained from the EBSD data is shown in **Figs. 1** (a) and (b), respectively. Colors in Fig. 1 (b) indicate the crystal orientations of the corresponding colors in the attached IPF map. For the display of EBSD map, OIM analysis (ver.7.1) of TSL Solutions K.K. was used.

As an example, the crystalline grain A indicated by the arrow in Fig. 1 was selected as the start point of the abovementioned analysis and, the adjacent variants were analyzed seeking a common austenite orientation. In this calculation, tolerance angles were set at various angles within the range of 2–15 degrees and the analysis was continued as long as the misorientation θ were within the tolerance angle. The result is shown in **Fig. 2**.

When the tolerance angle was set at 2 degrees (Fig. 2 (a)), the analysis failed to discover any common austenite orientation with adjacent variants. Therefore, no grain is shown in the figure. This is because the tolerance angle is set at too small to discover a common austenite orientation within. When the tolerance angles are 3 degrees or above, martensite variants having a common austenite orientation were discovered, which is shown with the orientation color of analyzed austenite orientation in Fig. 2 (b)–(f). However, its region expands greatly as the tolerance angle is increased. These results clearly indicate that the analysis of common austenite grains can vary depending on the tolerance angle.

Such an inconsistency in the results of prior austenite analysis at different tolerance angles is due to the high probability of discovering a common austenite orientation coincidentally among variants that have been transformed from different austenite grains. The probability of discovering any common austenite orientation coincidentally between two variants is estimated as the function of the tolerance angle. In this estimation, fifty thousand pairs of crystal orientations that do not have any crystallographic relationship with each

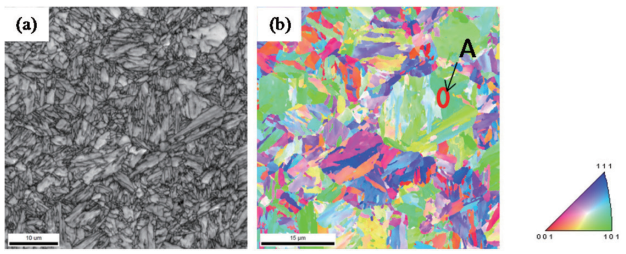

Fig. 1 Image quality mapping of the microstructure (a) and corresponding orientation mapping (b) of the specimen

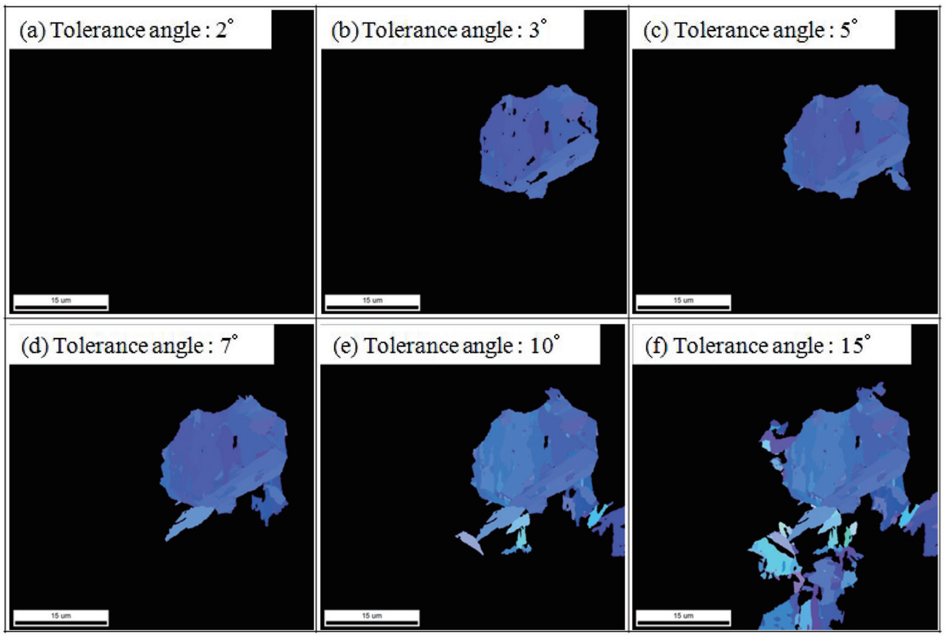

Fig. 2 Group of variants with common prior γ orientation for different tolerance angles (indicated as orientation mapping)

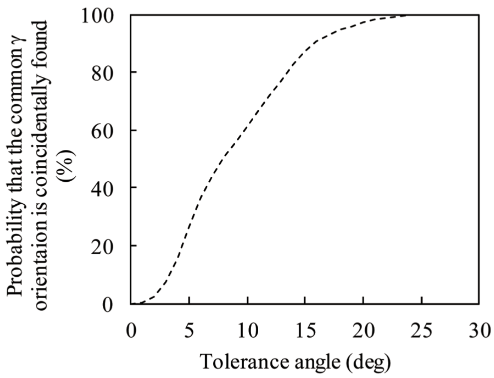

Fig. 3 Probability that a common γ orientation is coincidentally found between two variants for different cases of tolerance angles

other were generated artificially by computer and the probability of discovering a common austenite orientation between the pair was estimated. The result of the estimation is shown in **Fig. 3**. The probability of erroneous analysis is 7% with the tolerance angle of 3 degrees, 26% with the tolerance angle of 5 degrees and 61% with the tolerance angle of 10 degrees. As the result shows, the analysis is liable to give an incorrect result in the variant analysis based on the K-S relationship.

2.3 Reconstruction of austenite polycrystalline microstructure from ferrite microstructure

To solve the problem of the reconstruction method which may yield inconsistent results depending on the tolerance angle, various improvements have been applied in the austenite reconstructing method in the literature. For instance, by setting a mean tolerance angle in the possible tolerance angle range, or by conducting analysis with increasing the tolerance angle stepwise, the analysis gives a stable result of austenite reconstruction.

In the method that has been developed by Nippon Steel & Sumitomo Metal, the problem of inconsistency in the analysis result has been improved in the following method. The improvement of the analysis method is based on an investigation of the probability of the incorrect analysis. We focused on the probability of the variants that may not cause such an inconsistent analysis, and found that a considerable number of variants are sustainable against such the error analysis. For instance, as is shown in Fig. 3, the probability of the discovery of incorrect common austenite is not 100%, which means that there exist variants that are not influenced by such incorrect judgement. Using those variants, just one common austenite orientation with high reliability can be identified. In the calculation process, such reliable austenite orientations are first identified, and then, by making such grains the start point of further analysis, the surrounding martensite variants are analyzed to obtain the entire austenite microstructure with high accuracy.

The whole process of the analysis is started from choosing each variants in the microstructure as the starting grain of the abovementioned method. After seeking common austenite from each starting grain, monitoring all the reconstructed austenite grains obtained by the analysis, all the possible austenite orientations can be taken into account in the analysis. By evaluating the result statistically, variants that have only one candidate of austenite orientation and those that have plural candidates of austenite orientations are identified. The former variants are recognized to have a highly reliable prior austenite orientation.

As an example, the number of variants that have just one candidate prior austenite orientation is investigated in the conventional martensite microstructure in Fig. 1. Variants that have only one candidate of prior austenite orientation are extracted in the EBSD data and displayed with its orientation color. The tolerance angle for finding a common austenite orientation in this analysis was set at 5 degrees. The result is shown in **Fig. 4**. The region with color corresponds to the variants that have only one prior austenite orientation and the black region corresponds to variants that have plural candidates of prior austenite orientations. In most parts of the microstructure, austenite orientations can be identified.

For the rest of the variants that have plural candidates, the misorientation to the adjacent variants having only one reliable austen-

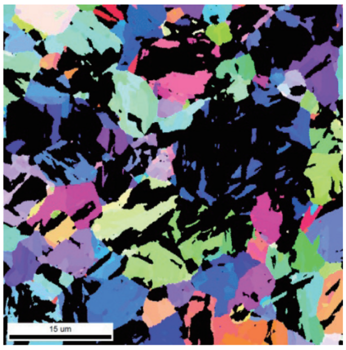

Fig. 4 Reconstructed γ microstructure using the variants in Fig. 1 which has just one candidating γ orientation

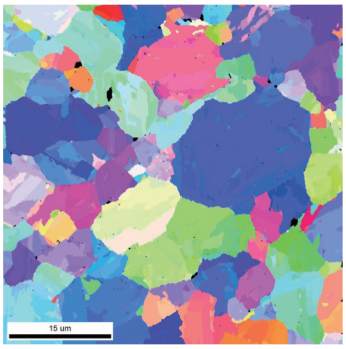

Fig. 5 Orientation mapping of γ microstructure reconstructed from the martensite microstructure shown in Fig. 1

ite orientation is examined, and the orientation that gives the smallest misorientation is selected as the final result. Through the above-mentioned process, the variants with plural candidates are incorporated into austenite grains that provide the smallest misorientation with the surrounding highly reliable austenite grains.

Using the above method, the entire austenite microstructure is reconstructed. **Figure 5** shows the result of the reconstruction of the whole austenite microstructure from the EBSD data in Fig. 1. It is confirmed that austenite grains having a clear equiaxed shape have been reconstructed from martensite microstructure. The regions shown in black are the grains that could not discover any common austenite with all of the surrounding variants.

2.4 Verification of accuracy of austenite reconstruction method

In this section, validity of the austenite reconstruction method is evaluated. The grain boundaries and crystal orientations of the reconstructed austenite are respectively compared to the microstructure observed by other experimental techniques in the following two experiments.

2.4.1 Verification of accuracy of reconstructed austenite grain boundary

To verify the accuracy of the reconstructed austenite grain boundary, the grain boundary in the reconstructed microstructure was compared to that by the conventional observation technique using picric acid etching. The area of microstructure that was observed in Fig. 1 was marked by Vickers indentation and then the area was etched by picric acid and the austenite grain boundaries were observed by a scanning electron microscope (SEM). The result is shown in **Figs. 6**(a)(b). In Fig. 6(a), the contours considered as austenite grain boundaries are marked with red lines. In Fig. 6(b), the grain boundaries where the misorientation is larger than 15 degrees are indicated by a black line on the reconstructed austenite microstructure so that grains are easily identifiable.

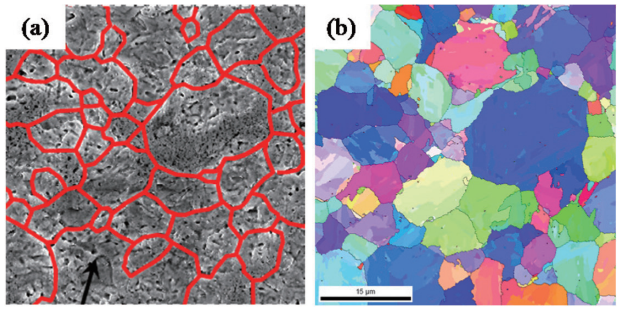

Fig. 6 Appropriateness of the grain boundaries of reconstructed austenite
(a) Austenite grain boundaries emerged by picric acid, (b) Grain boundaries in the reconstructed austenite (indicated by black line)

It is confirmed that most of the grain boundaries in Fig. 6(a) and (b) are in a good agreement with each other. Although there are some grain boundaries not appropriately analyzed and therefore, the reconstruction of prior austenite microstructure is not perfect, sufficient information to grasp the state of austenite microstructure is obtained.

2.4.2 Verification of accuracy of austenite orientation

To verify the correctness of the crystal orientations of the reconstructed austenite grains, orientations of reconstructed austenite grains are compared to orientations of retained austenite grains that remains in the same microstructure. For an experiment, S55C steel was quenched from a temperature in the austenite region to room temperature. The surface of the specimen was analyzed by EBSD. The specimen includes retained austenite grains within its quenched microstructure. Since the orientations of the retained austenite are considered to maintain the same orientations of prior austenite before quenching, the accuracy of crystalline orientation can be examined by comparing the orientation of retained austenite with that of reconstructed austenite.

The orientation map of the microstructure of the quenched specimen is shown in **Fig. 7**(a) and the orientation map of the residual austenite in the quenched microstructure is shown in Fig. 7(b). Furthermore, the result of the austenite reconstruction using the EBSD data in Fig. 7(a) is shown in Fig. 7(c). By comparing the orientation of each austenite in Fig. 7(b) with each of the orientations of reconstructed austenite at the corresponding position in Fig. 7(c), it is confirmed that the orientation of the retained austenite agrees well with the orientation of the adjacent reconstructed austenite. Based on this result, it is confirmed that the orientation of the reconstructed austenite is accurately obtained.

2.5 Example of application

Since the analysis method uses the average orientation of variants as input data, the computational load is much reduced. The analysis of microstructure in a wide range can be performed within a short period of time. Therefore, the method is suitable for acquiring average crystal grain size and texture of austenite statistically through a large quantity of data of reconstructed austenite.

As an example of such analysis, the time-dependent evolution of austenite grain size and texture of steel during heat treatment at above A_{c3} temperature is shown in the following experimental results.

The compositions of the sample used are 0.2%C-2%Mn and after hot-rolling and cold-rolling, the steel sample was heat-treated by

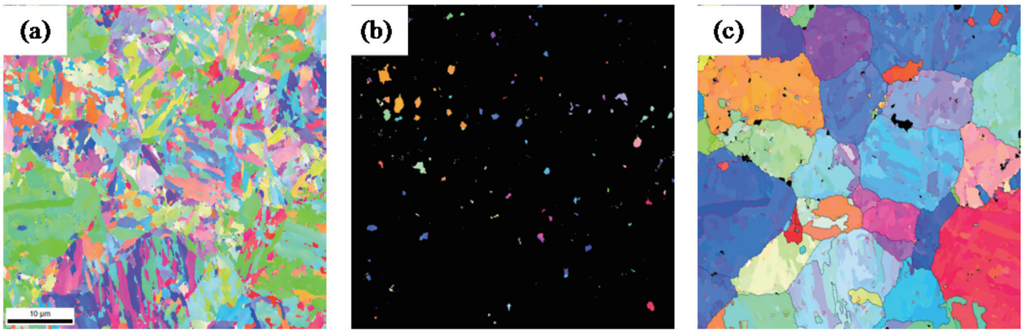

Fig. 7 Appropriateness of the crystal orientation of reconstructed austenite
(a) Orientation mapping of the initial (martensite) microstructure, (b) Orientation mapping of the retained austenite grains in the initial microstructure, (c) Reconstructed austenite microstructure with the retained austenite shown in (b)

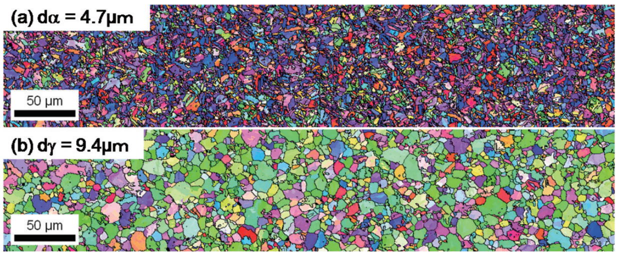

Fig. 8 Orientation mapping by EBSD analysis
(a) Microstructure of martensite after 870×30s annealing and water quenching, (b) Reconstructed austenite microstructure

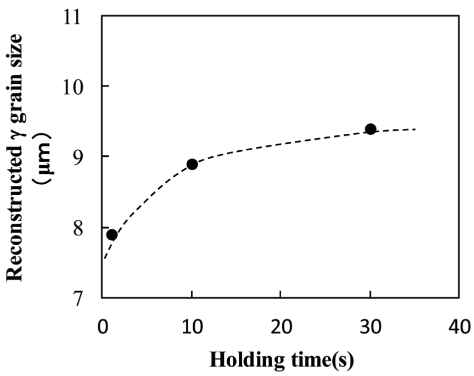

Fig. 9 Changes of grain size of reconstructed austenite microstructure to holding time at 870°C

heating up to 870°C, being held for various periods of time between 1–30 seconds and then water-quenched. EBSD measurement was conducted at intervals of 0.1 micrometer in the area of 500 micrometers in length and 100 micrometers in thickness at the position of 3/4 of the sheet thickness on the cross section perpendicular to the direction of rolling. The EBSD data of the microstructure was analyzed by the developed method and prior austenite was reconstructed. In the reconstructed results, the average austenite grain sizes and the textures after different periods of heat treatment were analyzed. For the evaluation of austenite grain size, a grain is defined as one surrounded by crystal boundaries with misorientation larger than 15 degrees. The texture of reconstructed austenite was analyzed by a method based on spherical harmonics expansion using the orientation distribution function (ODF).

A representative result of the austenite reconstruction is shown in **Figs. 8**(a) and (b), which are orientation mappings of the quenched steel after holding at 870°C for 30 seconds and that of the reconstructed austenite, respectively. In these figures, average grain sizes of the ferrite and austenite are also shown. Changes in average grain sizes and texture (ϕ_2=45 degree section of ODF) to the holding time are shown in **Fig. 9** and **Fig. 10**. In Fig. 9, it is confirmed that along with the increase in holding time from 1 to 30 seconds, the average grain size grows from 7.9 micrometers to 9.3 micrometers. In Fig. 10, the textures of reconstructed austenite are confirmed to be the typical texture of austenite with high intensity at the brass orientation ((110)[1$\bar{1}$2]) and at the copper orientation ((112)[$\bar{1}\bar{1}$1]). During the heat treatment period, there is no noticeable change in the intensities.

As the result shown above indicates, the changes of austenite microstructure during the heat treatment process are successfully evaluated with this method.

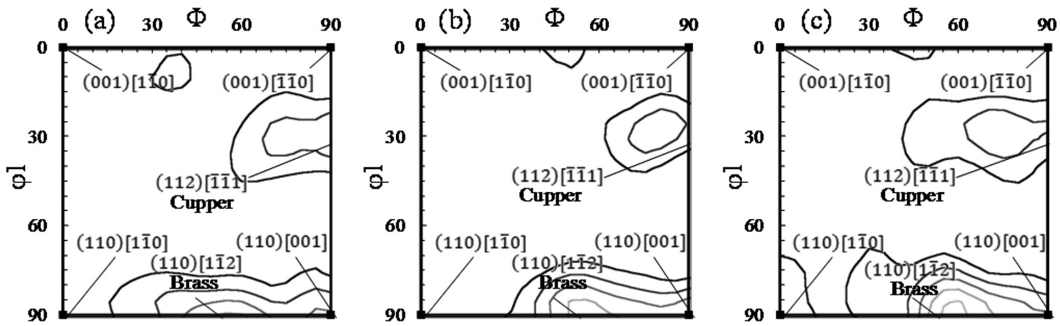

Fig. 10 Changes of the texture of reconstructed austenite microstructure to holding time (ϕ_2=45° section in ODF, (a) after 870°C×1s hold, (b) after 870°C×10s hold, (c) after 870°C×30s hold)

3. Conclusion

An analysis method of reconstructing prior austenite by using the EBSD analysis data of martensite or bainite microstructure of steel has been developed. The method has improved the accuracy of reconstructing by solving the problem of the inconsistent result of analysis due to the tolerance angle, and has been developed as an analysis program to determine the austenite microstructure. As a result of the assessment of the analysis results, it is confirmed that austenite microstructure is reproduced with high accuracy. Furthermore, since the method is able to reconstruct austenite in a wide range of microstructures within a short period of time, the method is suitable for the statistical measurement of microstructure by obtaining a large amount of orientation data of prior austenite.

Acknowledgements

We wish to express our utmost sincere gratitude to Dr. Toshiro Tomida of the Ibaraki Prefectural Government (formerly of Nippon Steel & Sumikin Technology Co., Ltd.) for the great assistance rendered in establishing the program of the analysis method and for the valuable advice given in discussions for writing this article.

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