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In-situ Observation of Microstructure Change in Steel by EBSD

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Abstract

In-situ EBSD analysis is attracting attention as a method for direct observation of microstructural changes of steel under deformation or during heat treatment. New types of microscope stages for tensile deformation and heating have been introduced for EBSD. At analysis of JIS SUS304 stainless steel during deformation using the tensile stage, increase in low-angle boundaries inside crystal grains and a rise in the KAM value (to be explained herein) near grain boundaries have been observed. In addition, through the analysis of transformation behavior of pure Ti and steel during heating in real time, specific orientation relationships such as the Burgers and the K-S relationships have been confirmed to exist between a parent phase and a product phase.

1. Introduction

Crystal grains of steel are aligned, to a greater or lesser extent, in a certain preferred orientation depending on the chemical composition and the working applied during manufacturing processes. Evaluation of such a microstructure, or a texture, is important in studying the anisotropy of material properties such as strength, ductility, toughness and magnetic characteristics. For the examination of steels, X-ray diffractometry has long been employed widely for texture analysis; pole figure measurement by the Schultz reflection method is one of such examples.[1] While the method can evaluate ordinary textures, it is difficult to analyze orientations of individual crystal grains or local configuration of a texture. Precise control of microstructure is essential for developing high-performance and high-functionality steels, and therefore analysis technology for precision observation of microstructure is of great importance. Electron back scattering diffraction (EBSD)[2] can produce fundamental information on the microstructure of polycrystalline materials, or analyzing the orientation of individual crystal grains that compose each material, on micro- to nanometer scales.

In the field of application of EBSD to microstructural analysis, there have been some reports recently on the methods for clarifying material deformation behavior or formation mechanisms of microstructure based on in-situ observation.[3–6] Clarifying the changes in microstructure during deformation or heat treatment is of great importance in the study of steels, but at present, such changes are merely conjectured based on a comparison of the microstructures before and after the deformation or heat treatment in question. If it is possible to observe and evaluate microstructural changes of steel in situ in real time, microstructures ideal for obtaining higher functionality will be identified, and new steel products developed more easily. This paper outlines the tensile and heating stages for scanning electron microscopes (SEM) introduced for the purpose of observing and evaluating metallographic structure in situ, and presents some examples of microstructural changes in steel materials observed during deformation and heating.

2. In-situ EBSD Analysis during Tensile Deformation

2.1 Tensile Stage

Figure 1 shows the appearance of the tensile stage used for the present study. Its maximum tensile load is 2 500 N; using a test piece having a sectional area of 2 mm^2 at the parallel portion, for example, it is possible to apply a tensile load of approximately 1 200 MPa for material evaluation. The standard size of the parallel portion is 10 mm in length and 2 mm in width, but this can be changed flexibly in consideration of the strength and ductility of the material to be examined. The surfaces of the test pieces must be, like those for common EBSD analysis, mirror-polished and then finished by electrolytic or colloidal silica polishing to remove processing strain in the surface layer. Since the maximum tensile load is 2 500 N as stated earlier, the acceptable test piece thickness is, depending on material strength, about 1 mm, which allows for easy handling and processing.

It is necessary to position a test piece for EBSD analysis obliquely at 70° so that it faces squarely to the detector, but in the case of the developed stage, it is set at 20° and then the stage turns

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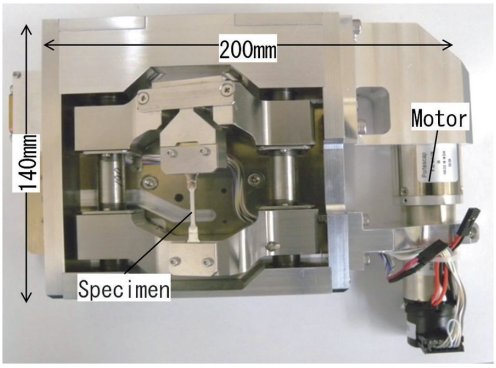

Fig. 1 Tensile stage

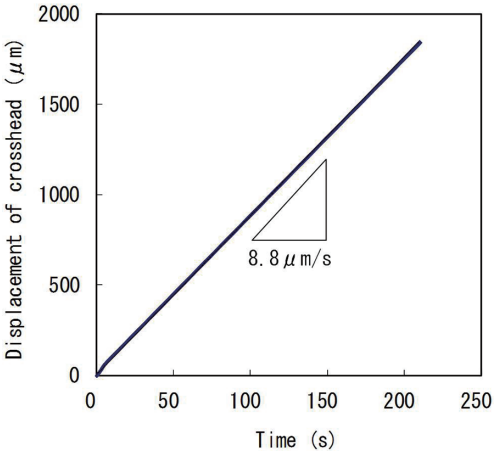

Fig. 2 Crosshead speed of tensile stage

by 50° to make it face the detector squarely at 70°. The mechanism of the developed stage is such that the test piece is pulled from both sides to deform it evenly, and the field of view does not shift as a result of the deformation. In addition, because the tensile load and the displacement can be output to outside systems, it is possible to suspend the loading during analysis while watching the behavior of the actual stress-strain curve. In in-situ tensile EBSD analysis, the region under observation does not necessarily deform as expected. This may not always pose problems with materials that deform homogeneously in the entire parallel portion, but with a material in which deformation bands do not spread easily, such as ultra-fine-grain steel, it is necessary to take some measures to have the entire EBSD object zone deform evenly; these measures include making the parallel portion shorter, cutting a notch there, etc.

Figure 2 shows the speed change of the crosshead of the tensile stage; a constant speed is maintained in the whole stroke from the start of deformation to failure. The average speed of the crosshead for the test is 8.8 μm/s (roughly 0.5 mm/min), and it has been confirmed to follow speed control at 1 μm/s or less.

2.2 In-situ EBSD Analysis of JIS SUS304 during Tensile Deformation

A specimen of JIS SUS304 stainless steel was analyzed in situ by EBSD during tensile deformation by the stage. First, an area 200 × 300 μm in the center of the parallel portion was analyzed by EBSD at intervals of 1.5 μm before tensile deformation, and then during tensile deformation, the same area was analyzed under the same conditions. After the specimen yielded, the displacement was temporarily held for EBSD analysis at intervals of 100 μm, approximately. After that, the analysis was continued further into large deformations (1 700, 3 200 μm). The time of the EBSD analysis was 6 to 7 min. per field of view. As seen with the stress-strain curve in **Fig. 3**, the stress fell by 30 to 50 N during the EBSD analysis after the yield point, but it returned to the original level every time the tensile deformation was resumed. These stress falls presumably resulted from stress relaxation due to coalescence and annihilation of dislocations, but the details are still unclear. Through analysis of the measurement data, maps of inverse pole figure, misorientation and kernel average misorientation (KAM) were produced. KAM is the average of the misorientation between a particular pixel and those adjacent to it; it is widely used as an indicator of plastic strain gradient in a micro region.

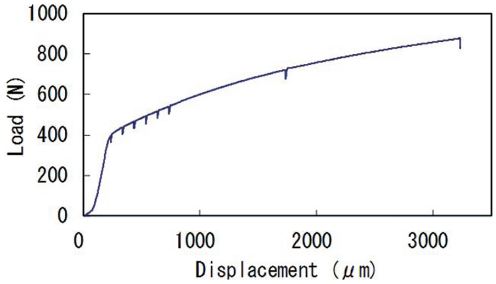

Fig. 3 Load-displacement curve of SUS304

The maps obtained through the EBSD analysis are given in **Fig. 4**; low-angle grain boundaries and KAM were found to increase as the deformation advanced. **Figure 5** shows a KAM map and a Schmid factor map before the deformation and a KAM map after it. The KAM map before the deformation is mostly in blue, which means that there was little local change in crystal orientation. In contrast, the map after it shows local rises of the KAM value. There is no distinct correlation between the KAM distribution after the deformation and the Schmid factor before it. The KAM value is presumed to correlate to crystal grain size, in other words, the KAM value does not increase much in regions of large crystal grains, but it looks to increase at grain boundaries in small-grain regions. This is presumably because, while in large-grain regions local orientation change is mitigated by slip deformation, grain deformation is mutually restricted in small-grain regions, leading to local orientation change at grain boundaries.

3. In-situ EBSD Analysis during Heating

3.1 Heating Stage

Figure 6 shows the appearance of the heating stage; its size is 110 × 40 mm, and a specimen 5 × 5 mm in size and 0.5 to 1.0 mm in thickness, is set at the center. A major concern about in-situ high-temperature EBSD analysis is heat damage to the secondary electron detector of the SEM and the EBSD detector. In the present study, the heating temperature was set at 800°C or higher for the purpose of observing phase transformations of steel in situ, and the heater is fixed in such a manner to heat a specimen at as small a heat input as possible.

To test the performance of the stage, phase transformation was observed in situ using pure Ti specimens.[5] At room temperature, Ti is in the α phase of hexagonal close-packed (hcp) crystals, and transforms into the β phase of body-centered cubic (bcc) crystals at 883°C or higher. In consideration of the above, high-temperature EBSD analysis of the α-Ti and the β-Ti phases was conducted in situ in a field of 300 × 600 μm, at a measurement interval of 4 μm, and at

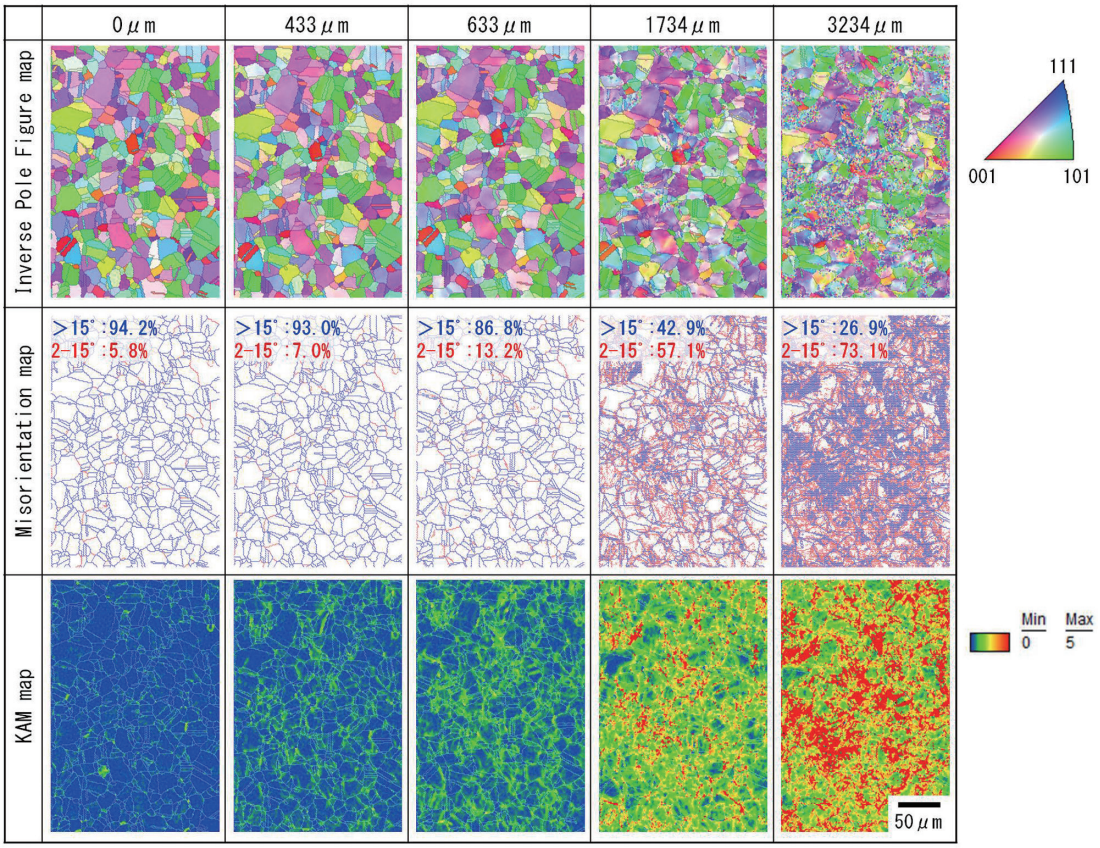

Fig. 4 In-situ EBSD analysis of SUS304 during tensile deformation

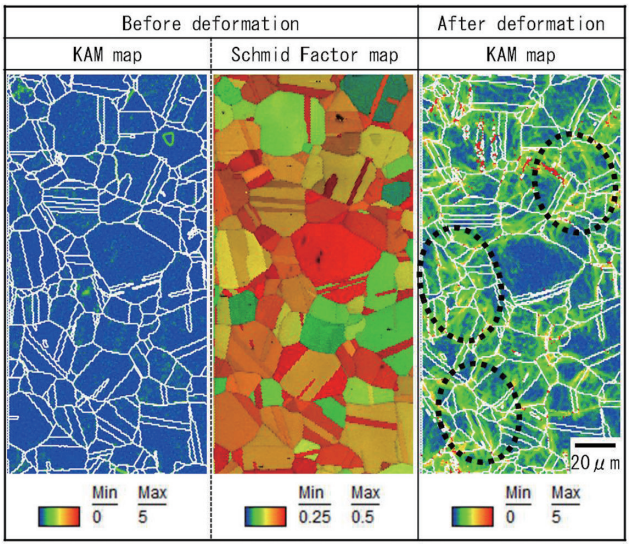

Fig. 5 Changes in the KAM map before and after tensile deformation

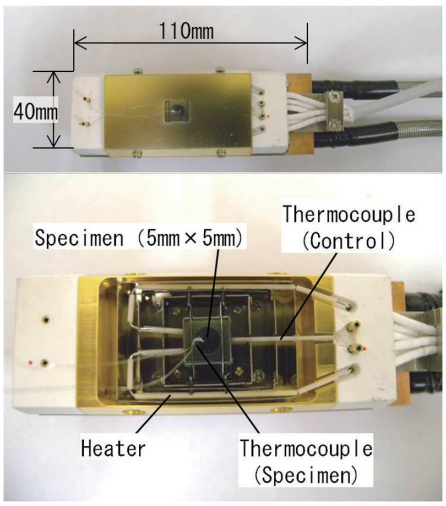

Fig. 6 Heating stage

different temperatures; the measurement time was roughly 3 min. per field. Before the test, there were fears about low definition of the pattern image due to specimen surface contamination or oxidation and the effects of infrared rays due to the heating on the detector, but in fact they posed no problems at the test, and clear diffraction patterns as given in **Fig. 7** were obtained.

Figure 8 shows the results of the in-situ EBSD analysis using the heating stage. In the α-β phase map, a β-Ti grain was found to form through transformation at the right-hand corner during heating to 920°C. The temperature at which the β-Ti phase formed was higher than the transformation point (883°C) by approximately 40°C, but this was presumably due to a measurement error or because the temperature was measured at the surface, not at the inside. From the inverse pole figure maps, of the β-Ti grains that formed through transformation, those of a certain crystal orientation are seen to grow rapidly. When the temperature fell thereafter to 900°C,

an α-Ti grain was found to form inside a β-Ti grain. The newly formed α-Ti grain grew as the temperature fell further. The validity of the analysis result was examined based on the relationship of the measured orientations of the α- and β-Ti grains. When Ti transforms from the high-temperature β phase to the low-temperature α, the Burgers relationship below applies to the two phases:

$$\{110\}_{\beta}//(0001)_{\alpha}, \text{ and } <111>_{\beta}//[11\bar{2}0]_{\alpha}.$$

Accordingly, regarding the analysis result at 870°C, we examined the orientation relationship between the β phase (the β-1 grain in **Fig. 9**) and the surrounding α-phase grains by turning the measured data of the former so that its orientation became {110}<111> (see Fig. 9). The crystal orientation of the α-1 grain, which formed through the transformation, was different from $(0001)[11\bar{2}0]$ by 1.6° only; this indicates that the Burgers relationship applies to the α-1 and the β-1 grains. Likewise, the crystal orientation of the α-2 grain was different from $(0001)[11\bar{2}0]$ by 10.1°, which means that it is highly probable that the α-2 grain also formed through transformation of the β-1 grain satisfying the Burgers relationship. The α-1 grain looked as if it formed as a result of the growth of a portion of the α-3 grain that remained unchanged through the transformation, but because the orientation difference between the two is as large as 21.8°, the former does not seem to originate from the latter.

What must be noted is that the information obtained through EBSD is that on the crystal orientation at the surface, which may not represent exactly the material behavior at the bulk interior. Nevertheless, the present study has demonstrated that in-situ EBSD analysis using the heating stage can follow the transformation behavior of metal materials considerably accurately. Because the stage can heat a specimen to around 900°C, it is expected to be highly instrumental in analyzing the α-γ transformation of steel.

3.2 In-situ EBSD Analysis of Low-carbon Steel during Heating

Next, we attempted to observe the α-γ transformation of steel in situ using the heating stage. A low-carbon steel specimen 5×5 mm in size was cut out, and its surface was mirror polished and then electrolytically polished to remove processing strain in the surface layer. The specimen was heated and then cooled on the stage, and it

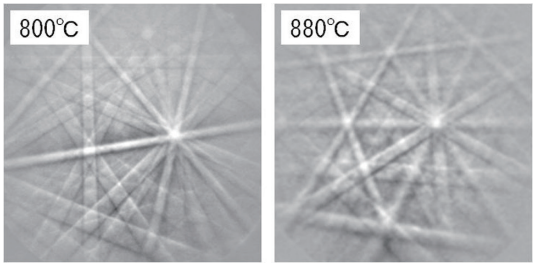

Fig. 7 EBSD patterns obtained at 800°C and 880°C

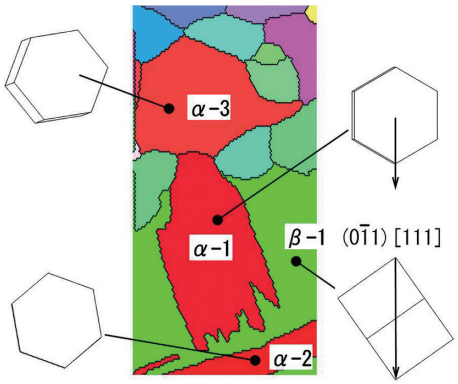

Fig. 9 Crystal orientation relationship between α-Ti and β-Ti at 870°C

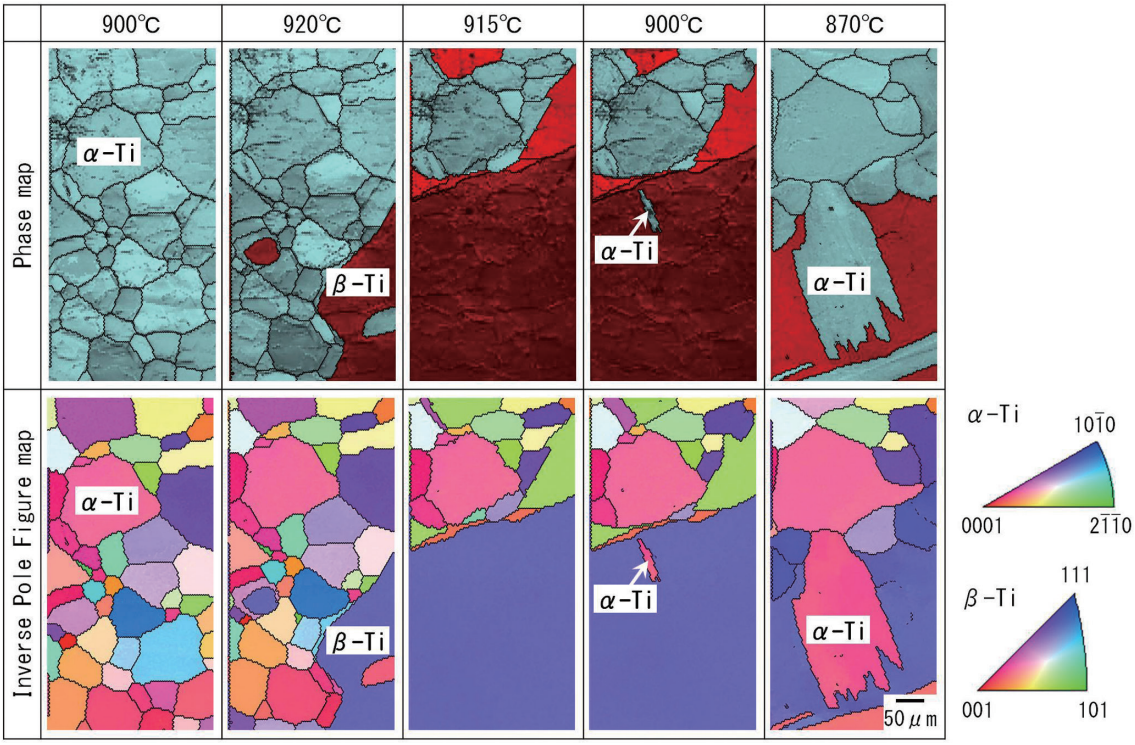

Fig. 8 In-situ observation of the α-β phase transformation of Ti during heating and cooling

was held at 300, 500, 700 and 800°C during the heating and at 300°C during the cooling, for approximately 5 min each, to have the structure stabilize, and analyzed by EBSD in an area 100×200 μm in size at intervals 1 μm. The measurement time was roughly 5 min. per field of analysis.

Figure 10 shows the results of the above analysis. As was the case in the analysis of the pure Ti specimen explained in Sub-section 3.1, there was no surface contamination this time either up to 800°C, and there were no problems for the analysis. Although the specimen drifted slightly in the vertical direction, the field of view did not shift significantly during the 5-min. measurement. In the images obtained at 300 and 500°C, there was no significant change in microstructure, but at 700°C, grain growth due to boundary migration was confirmed as seen with the dotted ellipses in the misorientation map of Fig. 10. At 800°C, the γ phase was confirmed to have formed, albeit in a small area ratio of 2%. This γ phase was found to have disappeared during the cooling from 800 to 300°C, and thus the heating stage has proved effective at directly observing the $\alpha \rightarrow \gamma \rightarrow \alpha$ phase transformation. Certain orientation relationships typically such as the Kurdjumov-Sachs (K-S) relationship hold in the α-γ transformation of steel as well as the phase transformations of pure Ti explained earlier. In relation to this, we examined the orientation data as follows in order to clarify to what extent a γ grain measured at 800°C satisfied the K-S relationship with the α grains surrounding it:

(1) The orientation data (Euler angle) of the α grains adjacent to the γ grain were extracted from the analysis result at 800°C.
(2) The above orientation data of the α grains were transformed into the orientation of the γ grain in question using a rotation matrix (24 variants) equivalent to the K-S relationship.
(3) The misorientation angle between the measured orientation and the calculated (24) orientations was calculated for each of the γ grains, and the smallest value was chosen as the deviation from the K-S relationship.
(4) The α grains were classified into "K-S", those of orientation deviating from the K-S relationship by 5° or less, and "near K-S", those of orientation deviating from the same by 5 to 10°.

Figure 11 shows some examples of the crystal orientation relationships between the measured γ grains (except those at the ends of the field of analysis) and adjacent α grains, and **Fig. 12** the classification according to the deviation from the K-S relationship. Nearly 70% of the γ grains were found to have the K-S relationship with one or more adjacent α grains, and of these, more than 40% were in the K-S relationship with two or more (2-KS). What EBSD analysis yields is the crystal orientation relationships at a free surface in a two-dimensional plane, and those of a grain with others not showing at the surface are unknown. Considering the probability of the K-S relationship with such inside grains, there are probably more γ

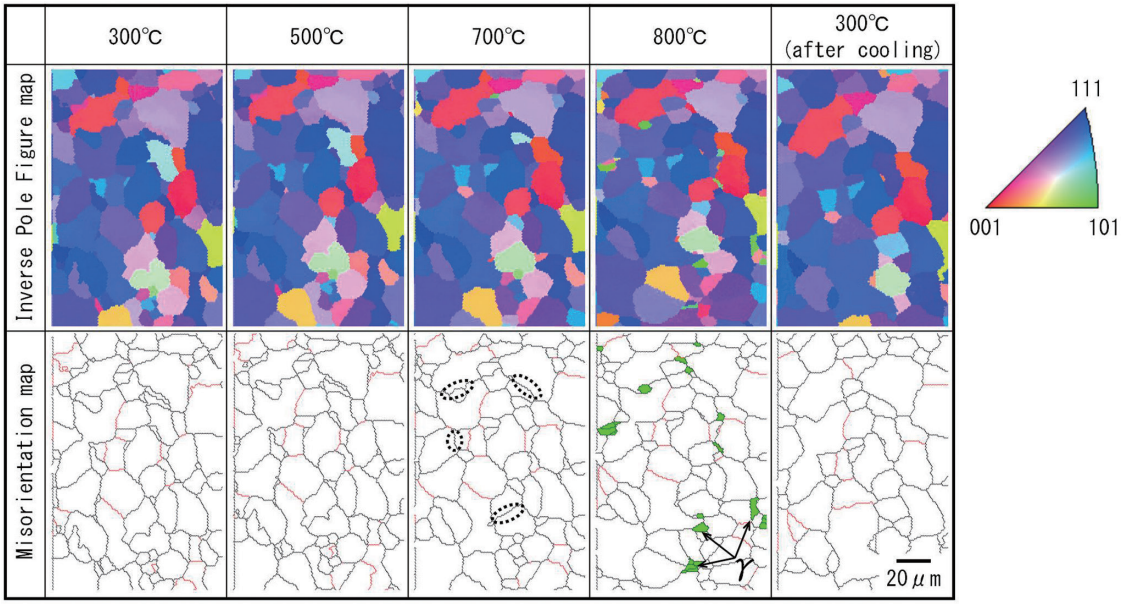

Fig. 10 In-situ observation of the α-γ phase transformation of steel during heating and cooling

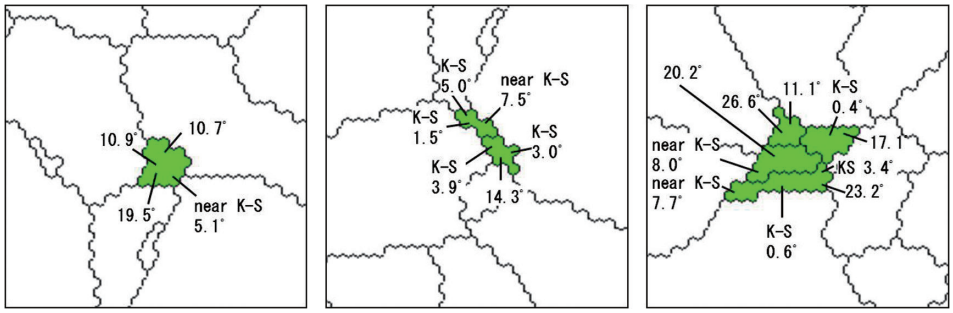

Fig. 11 Crystal orientation relationship between α and γ grains at 800°C

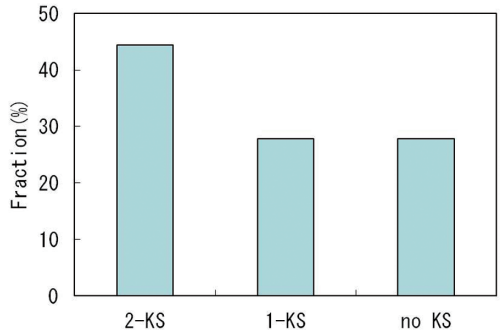

Fig. 12 Classification result of crystal orientation relationship

grains in the K-S relationship with two or more adjacent α grains.

Because of the symmetry of crystals, there are several equivalent crystal orientation relationships (variants) between the α and the γ phases of steel, and when two grains are in the K-S relationship, the number of variants is 24. Various models have been proposed as to which of the 24 variants works in preference (the rule of variant selection),[7] and of those, the transformation texture calculation assuming that the preference variant is selected based on double K-S relationships is reported to agree well with measured textures quantitatively.[8, 9] The results obtained through the in-situ EBSD analysis during heating corroborate the validity of the variant selection rule based on the double K-S relationship. Orientation analysis of more γ grains will strengthen the statistical validity of the rule.

4. Conclusion

Two types of stages for SEM, one for tensile deformation and the other for heating, were introduced, and the following findings obtained through in-situ EBSD analysis using the stages:

(1) The newly introduced stages have proved instrumental for in-situ EBSD analysis of the metallographic behavior of steel during deformation and heating. The tensile stage has been confirmed to enable analysis of 1 200-MPa class high-strength steels during deformation, and the heating stage structural measurement at temperatures up to 900°C, approximately.

(2) Through in-situ EBSD analysis of JIS SUS304 specimens during tensile deformation, low-angle grain boundaries that form inside grains have been found to increase and so has the KAM value near grain boundaries. The increase in the KAM value in regions of small crystal grains is presumably due to that, while in large grain portions local orientation change is easily mitigated by slip deformation, grains are mutually restricted in their deformation in small grain portions, leading to local orientation change at grain boundaries.

(3) Evaluation of the transformation behavior of pure Ti and steel by in-situ EBSD analysis during heating has been enabled. There are specific crystal orientation relationships such as the Burgers relationship and K-S relationship between a parent phase and a product phase resulting from transformation under heating, and the finding of the analysis using the heating stage has proved valid for confirming such relationship. The analysis has also shown that the double K-S relationship applies to the $\alpha \rightarrow \gamma$ transformation of steel with a high probability.

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