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In situ EBSD observation of grain boundary character distribution evolution during thermomechanical process used for grain boundary engineering of 304 austenitic stainless steel

Shun Tokita*, Hiroyuki Kokawa, Yutaka S. Sato, Hiromichi T. Fujii

Department of Materials Processing, Graduate School of Engineering, Tohoku University, 6-6-02 Aramaki-aza-Aoba, Aoba-ku, Sendai 980-8579, Japan

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A B S T R A C T

Electron backscatter diffraction (EBSD) was used to examine the microstructural evolution in a one-step thermomechanically processed 304 austenitic stainless steel specimen during the thermomechanical process of grain boundary engineering. Solution-treated materials were cold-rolled to 3% reduction and subsequently annealed at 1220 K for different annealing times. The EBSD observation of the specimen showed an increase in the frequency of coincident site lattice (CSL) boundaries and a decrease in the percolation probability of random boundaries. Additionally, the specimen exhibited heterogeneous growth of clusters of grains that contained a high frequency of CSL boundaries. These clusters of grains were developed in the entire observed area by strain-induced grain growth according to the results of grain orientation spread analysis. The details of the growth of the clusters and the disconnection of random boundaries were successfully observed in situ using EBSD and a heating stage. The frequency of CSL boundaries increased with the growth of the clusters. Disconnection of random boundaries between the clusters was achieved by the formation of annealing twins through the impingement of the growing clusters during the thermomechanical process. Twin variant selection to introduce CSL boundaries into a random boundary network was observed by the in situ EBSD observation.

1. Introduction

Austenitic stainless steels have been widely used in many industrial applications because they have good mechanical properties and high resistance to general corrosion. However, intergranular corrosion caused by sensitization often occurs at grain boundaries during high-temperature use and the welding process. Grain boundary engineering (GBE) has been attracting attention as an effective method to prevent grain boundary degradations such as intergranular corrosion [1,2]. Fundamental research has shown that grain boundary phenomena strongly depend on the structure and character of grain boundaries [3,4]. Low-Σ CSL boundaries have higher intergranular corrosion resistance than random boundaries [5,6], and the properties of polycrystalline materials can be improved by increasing the frequency of CSL boundaries and disconnecting random boundary networks. Previous studies performed by some of the authors of the present paper reported a drastic improvement of the intergranular corrosion resistance of austenitic stainless steels by the application of the GBE thermomechanical process [7–9]. Additionally, GBE has led to the suppression of various grain boundary degradations, such as segregation-induced embrittlement [10], grain boundary sliding [11], liquation cracking in weld heat-affected zones [12], weld decay [13,14], knife-line attack [15], and corrosion erosion [16].

After decades of GBE research, the optimization of the grain boundary character distribution (GBCD) by one-step and iterative thermomechanical processes has been reported in various low stacking fault energy (SFE) materials, such as nickel and Ni-based alloys [12,17,18], austenitic stainless steels [7,8,19,20], brass [21], and copper [22]. Recrystallization and grain growth are known to occur during the thermomechanical process. Therefore, to develop an efficient thermomechanical process and improve the applicability of GBE to various materials, it is necessary to reveal the relationship between these microstructural changes and GBCD optimization. The optimization mechanism of GBCD for GBE has been actively discussed for many years. Randle et al. proposed that GBCD optimization mainly consists of a "Σ3 regeneration mechanism" and a "new twinning mechanism" [23]. Transmission electron microscopy observations reported by Kumar et al. showed boundary decomposition by the formation of annealing twins [24]. However, no direct observations exist that sufficiently explain how the grain boundary engineered microstructure develops and

* Corresponding author at: Joining and Welding Research Institute, Osaka University, 11-1 Mihogaoka, Ibaraki-shi, Osaka 567-0047, Japan.
E-mail address: tokita@jwri.osaka-u.ac.jp (S. Tokita).

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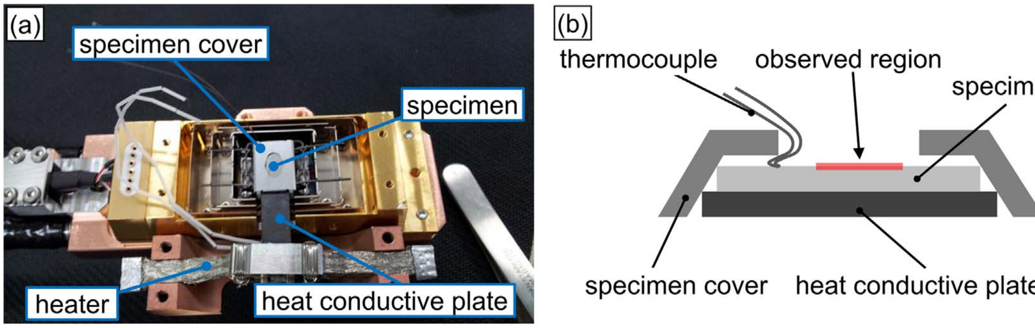

Fig. 1. (a) Photograph and (b) schematic illustration of heating stage used for in situ EBSD observations.

covers the entire material so as to disconnect the random boundary networks. Therefore, the objective of this study is to examine the microstructural evolution in 304 austenitic stainless steel during the GBE thermomechanical process. An in situ EBSD observation was conducted using a heating stage to examine the detailed characteristics of the grain growth and the changes in the GBCD, particularly, the disconnections of random boundary networks during the annealing process. Because the annealing time in the GBE process of 304 austenitic stainless steel is longer than that of other materials, the GBCD changes during the thermomechanical sequence were clearly observed in this material [25].

2. Experimental

2.1. Specimen Preparation

The material used in this study was 304 austenitic stainless steel with the chemical composition (wt%) 18.36 Cr, 8.15 Ni, 0.58 Si, 1.27 Mn, 0.040 C, 0.032 P, and 0.001 S. The as-received material was cut into specimen blocks, 10 W × 40 L × 9 T mm^3 in size, solution-treated at 1323 K for 0.5 h, and quenched in cold water. The solution-treated material will be hereafter denoted as 304 base material (BM). The BMs were deformed by cold rolling, resulting in a 3% reduction of thickness. The cold-rolled specimens were cut into small chips, 5 W × 4 L × 2 T mm^3 in size, because of the specimen size limitation of the heating stage that was used for in situ EBSD observation by scanning electron microscopy. The chip specimens were annealed in an electric furnace at 1220 K for 2–24 h and quenched in cold water. These cold-rolling and annealing conditions were selected according to our previous study on 304 austenitic stainless steel with the same chemical composition [26]. The specimens were ground with water-abrasive paper and subsequently polished with 1 μm Al_2O_3 particles. To obtain the suitable surface conditions for EBSD observation, electropolishing was conducted in a solution of 10% perchloric acid in ethanol at 273 K at a potential of 25 V.

2.2. EBSD Analysis of Thermomechanically Processed Specimen

The GBCD of the thermomechanically processed specimens was analyzed with a HITACH SE-4300SE field-emission-gun scanning electron microscope (SEM) equipped with the orientation imaging microscopy (OIM) system. EBSD analysis with a step size of 5 μm was conducted in the 4 × 4 mm^2 analyzed area, which was perpendicular to the rolling direction. The SEM was operated at an accelerating voltage of 25 kV. The material data of the 304 austenitic stainless steel were set for indexing diffraction patterns. The cleanup procedures were performed with OIM analysis software using the neighbor confidence index (CI) correlation method with a minimum CI value of 0.1 and grain dilation with a minimum grain size of 5 analysis pixels.

The relationships between grain boundary characters and intergranular corrosion resistance have been discussed in previous GBE studies [7,27,28]. It has been reported that the Σ3 boundary has higher resistance to intergranular corrosion than other CSL boundaries, while some CSL boundaries do not show significant resistance to intergranular corrosion [27]. On the other hand, in our previous studies, we observed non-corroded Σ9, 13b, 17a, 29a CSL boundary segments connecting to corroded random boundaries [7,28]. Therefore, in this study, the grain boundaries were approximately categorized into CSL boundaries with Σ ≤ 29 and random boundaries to discuss the general tendency of the GBCD. Additionally, low-Σ CSL boundaries with Σ ≤ 29 were denoted as CSL boundaries and Brandon's criterion [29] was adopted to define the critical deviation from the exact CSL orientation relationship.

2.3. In Situ EBSD Observation

The heating applied for the in situ EBSD observation had limited duration because the growing surface oxidation of the specimen decreased the quality of the EBSD pattern image during heating. Therefore, the specimen was 3% cold-rolled and annealed in an electric furnace at 1220 K for 6 h prior to the in situ EBSD observation. The specimen preparation and polishing procedure were the same as those described in Section 2.1.

The in situ EBSD observation was conducted with a HSEA-1000 heating stage equipped with a Philips XL-30FEG SEM; a photograph and a schematic illustration of the heating stage are shown in Fig. 1. The specimen contacted a heat-conductive plate that was connected to an electric heater. The specimen was partially covered by a titanium plate to delay the oxidation of the specimen during heating. The specimen temperature was measured using a chromel-alumel (K-type) thermocouple attached to the specimen surface near the analyzed area by resistance spot welding.

The temperature history during the in situ EBSD observation is shown in Fig. 2. Although measurements at 1220 K were possible, long exposure to 1220 K could cause visible GBCD changes of the specimen during the data collection period for one area. To eliminate these changes, the specimen was heated at 1220 ± 3 K for 80, 3, and 30 min

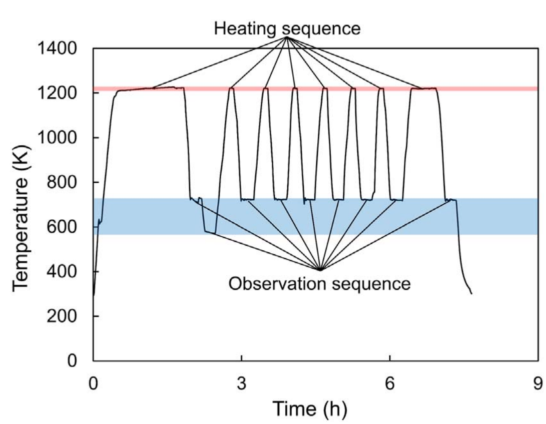

Fig. 2. Temperature history during in situ EBSD observations.