Strain-induced transformation process of SUS304 stainless steel revealed by *in situ* X-ray diffraction and high-resolution TEM observation

Metastable austenitic (γ) stainless steels such as SUS304 (Japanese industrial standard type 304 stainless steel) are commercially used as structural materials because of their excellent mechanical properties, including well-balanced corrosion resistance and ductility [1]. It is well known that SUS304 exhibits a strain-induced martensitic transformation, which is defined as a phase transformation from austenite to α'-martensite induced by plastic deformation. One of the important aspects of this transformation is that the enhancement of tensile strength and elongation to fracture results from the appearance of α'-martensite.

Some previous studies by *in situ* transmission electron microscopy (TEM) observations reported that no intermediate ε-martensite was present during the transformation processes and that α'-martensite formed from the twin structures on account of the fcc structure [2]. The appearance of intermediate ε-martensite strongly depends on the subtle balance between the stacking-fault energy and the surface energy. Recently Hatano *et al.* reported a substantial reduction of tensile ductility was found in hydrogen-charged SUS304, which should originate from the formation of ε-martensite of high density at room temperature [3]. In order to elucidate the nanoscale microstructures significantly affecting the transformation processes forming the α'-martensite from austenite in SUS304 at room temperature, we have performed X-ray diffraction experiments and high-resolution TEM observations. It is clearly demonstrated that ε-martensite with hexagonal symmetry appears as an intermediate structure during the plastic deformation of SUS304 stainless steel. In addition to stacking faults and dislocations, interfaces between the twin structures presumably play a key role in the formation of ε-martensite [4].

The materials employed in the present study were commercial SUS304 steel sheets with 0.4 mm thickness. Flat specimens, with 60 mm length and 12.5 mm width at the gauge section, were prepared along the rolling direction. X-ray diffraction experiments were performed using a PILATUS 100 K detector at SPring-8 **BL02B1** in the transmission geometry [5]. The experimental setup is shown in Fig. 1. The energy of an incident X-ray beam was 30.05 keV. The beam size was 3.0 × 0.5 mm^2. The distance between the specimen and the detector was 1100 mm and the detector was fixed at a diffraction angle where the typical diffraction peaks of the α'-martensite and γ-phases could be measured. In an *in situ* measurement under applied stress, the loading device was mounted on the diffractometer. Diffraction data were collected under stepwise stress application. The exposure time for each measurement was 60 min. High-resolution lattice images of microstructures associated with the γ-phase and ε-martensite were obtained using a JEM-2100F TEM apparatus at room temperature.

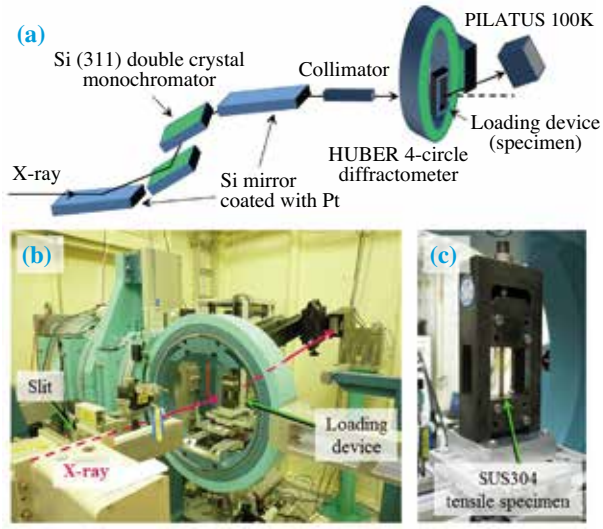

Fig. 1. Instruments for *in situ* measurement of X-ray diffraction data under stress. **(a)** Schematic illustration of experimental layout. Photographs of **(b)** 4-axis diffractometer at BL02B1 and **(c)** SUS304 tensile specimen mounted on a loading device.

Figure 2 shows the *in situ* X-ray diffraction patterns under the application of external stress. In the unstressed specimen, only the diffraction peaks for the γ-phase were observed. As a result of stress application, the diffraction peak for ε-martensite appeared and the intensity increased with increasing stress. However, the intensity was very weak compared with that of the γ-phase, indicating that the volume fraction of the ε-martensite was very small. The diffraction peak for α'-martensite also appeared and the intensity increased in the same manner as for ε-martensite. Our results revealed that ε-martensite was present in some of the specimens at room temperature.

Thus, to clarify the formation process of α'-martensite, we carried out a high-resolution TEM observation of the 20%-elongated specimen. Figure 3 shows a series of high-resolution TEM images obtained around the twin structures in respect of the formation of α'-martensite. As shown in Fig. 3(a), there were many twin structures in the specimen.

Figure 3(b) is a high-resolution TEM image of the square region in Fig. 3(a) showing the presence of ε-martensite in the γ-phase. In the region indicated by an arrow ε-martensite appeared at the interface of the twin structure generated by the strain of the fcc austenite phase. Figure 3(c) is a high-resolution TEM image obtained from the region indicated by the dotted square in Fig. 3(b). A stacking pattern characterizing the hexagonal close-packed structure was observed in the region indicated by "ε" in Fig. 3(c). These results imply that ε-martensite appeared at the nanoscale level near the interface of the twin structure in the strained fcc austenite phase.

We proposed the formation process of α'-martensite from the γ-phase induced by plastic deformation, which is schematically summarized in Fig. 3(d). As the first step, microtwin structures of approximately 10 nm widths are formed in the γ-phase by plastic deformation at about 10% elongation, as evident in Fig. 3(a). The formation of the microtwin structures increases the interface energy. Intermediate ε-martensite appears around the interface of the microtwin structures, as shown in Figs. 3(b) and 3(c). This implies that the formation of ε-martensite from the γ-phase accompanies the reduction of the total volume and reduces the increase in the interface energy. By applying further plastic deformation, the strain energy increases and ε-martensite transforms into α'-martensite.

In situ synchrotron diffraction measurements revealed the formation of a very small amount of ε-martensite as an intermediate phase during the strain-induced transformation from the γ-phase to the α'-phase. High-resolution TEM observations clearly demonstrated that the intermediate nanosized ε-martensite appeared near the twin boundaries in the strained γ-phase. ε-martensite grew at the twin boundaries in the γ-phase and served as the intermediate structure in the transformation process from the γ-phase to α'-martensite. The presence of ε-martensite in the plastic deformation of SUS304 stainless steel was thus demonstrated for the first time.

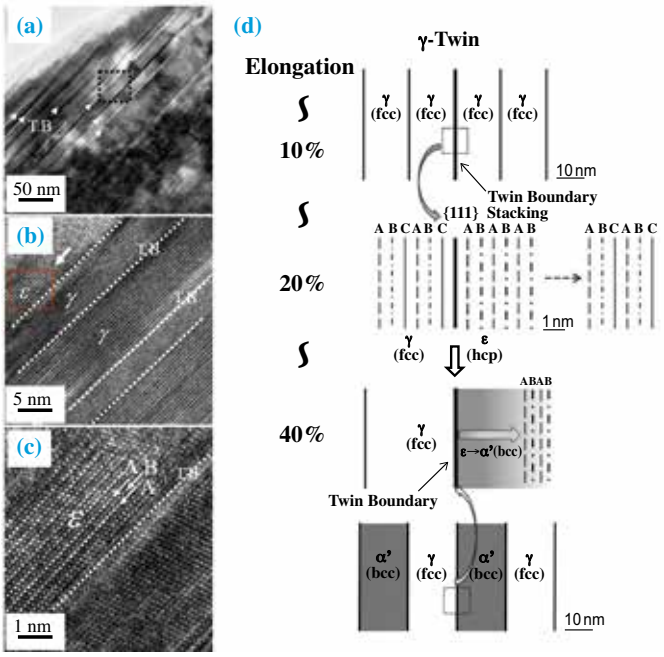

Fig. 3. Microstructures obtained in the 20%-elongated specimen. **(a)** Bright-field images showing twin structures in the fcc austenite phase. Arrows indicate twin boundaries (T.B). **(b)** High-resolution TEM image showing the twin structures, indicated by white dotted lines. **(c)** High-resolution TEM image obtained from the region enclosed by the dotted square in (b). The hexagonal structure is characterized as alternately stacked A and B layers, as indicated in (c). **(d)** Schematic illustrations of formation process of the strain-induced α'-martensite phase of tensile strained SUS304 at room temperature.

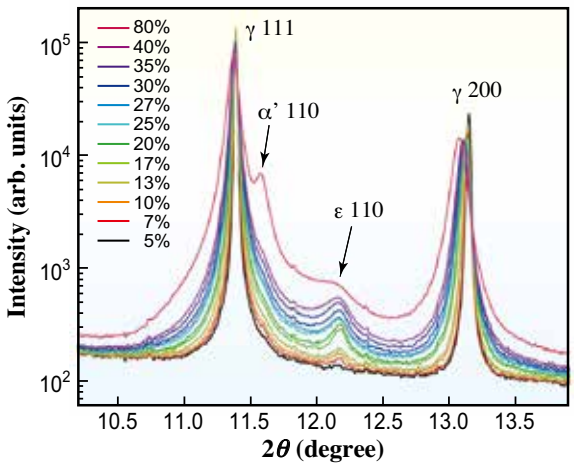

Fig. 2. Evolution of X-ray diffraction patterns in *in situ* measurement under stress. The percentages indicate the elongation applied to the specimen.

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