Measurement of the internal stress distribution in grains of stainless steel using white X-ray microbeam diffraction

The mechanical strength of a structural metallic material depends on the distribution of internal stress in crystalline grains controlled by their metallographic factors such as the dislocation density. In the development of stainless steel for the structural material of a nuclear reactor, it is important to inhibit the stress corrosion cracking in the presence of high-temperature and high-pressure water. Recently, it has been revealed that the susceptibility to intergranular stress corrosion cracking (IGSCC) of stainless steel of SUS316 tends to depend on its level of cold working. It has been suggested that the cold working affects the internal stress distribution generated by the external stress. To clarify the effect of the cold working on the internal local stress around the grain boundary, we developed the X-ray diffraction measurement technique with white X-ray microbeam for the observation of the internal stress distribution in individual grains of polycrystalline metal [1].

The internal stress can be evaluated from crystal lattice spacing determined by X-ray diffraction. The technique we developed measures crystal lattice spacing on a point-by-point basis by scanning X-ray irradiated position. This development had two technical problems. One was how to detect the diffraction signal from each single crystalline grain whose crystalline orientation is unknown. The other was that a visualizing technique of the grain boundary is needed for identifying the grain irradiated with the microbeam. The former problem can be solved using a white X-ray microbeam. The white X-ray microbeam must contain some X-ray photons with wavelengths fitting the Bragg condition of some crystalline planes of the irradiated grain. As the solution to the latter problem, we developed the technique of visualizing the grain structure by detecting the grain boundary from the change in the X-ray diffraction pattern during the scan of the irradiated position in the sample [2]. When the irradiated position on the sample crosses a grain boundary during the scanning of the sample position, the diffraction patterns must be drastically changed. The grain boundary can be recognized by evaluating this change in the diffraction patterns quantitatively.

This technique was developed at the white beam X-ray diffraction beamline **BL28B2**. The schematic of the experimental setup is shown in Fig. 1. The white X-ray microbeam was formed by a slit set at the upstream side of the sample. The minimum size of the beam we could obtain was about 10 μm. Data on the diffraction angle and wavelength of the X-ray diffraction peak are necessary to determine the crystal lattice spacing of their diffraction plane using the Bragg equation. The fundamental procedure of the measurement is as follows. At first, the two-dimensional (2D) detector was set downstream of the sample to detect the diffraction angle of the X-ray diffraction peak. We used a complementary metal oxide semiconductor (CMOS) flat-panel imager (FPI) made by Hamamatsu Photonics as the 2D detector. The Laue pattern of X-ray diffraction from each grain was detected as an image using the FPI. An example of the image data is shown in Fig. 1. The diffraction angle and azimuthal angle of each diffraction peak were geometrically determined from its position in the image data. Secondly, the solid-state detector (SSD) was set at the position of the diffraction peak determined from the image data to detect its wavelength. A slit is

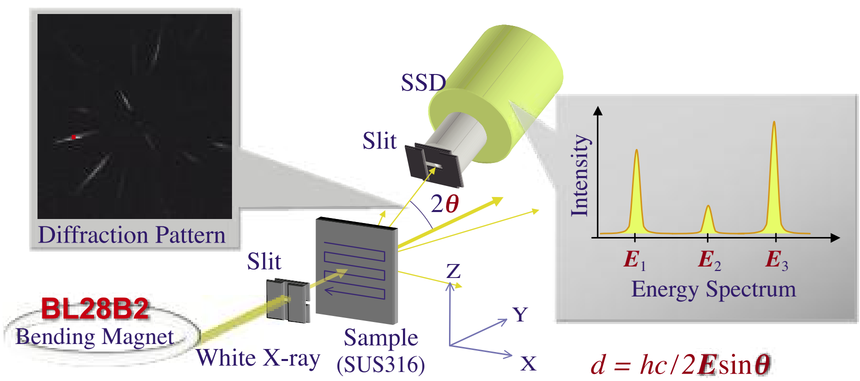

Fig. 1. Schematic of experimental setup. The applied stress is in the x-axis direction.

set at the upstream side of the SSD to keep the angle resolution for the restriction of the diffraction angle of the diffracted X-ray detected by the SSD. The accuracy of the determination of the lattice spacing of the system was estimated at $\delta d/d \cong 4\times10^{-4}$ by the measurement of the crystal lattice spacing of an undistorted silicon single crystal.

Here, we will introduce the result of the experiments performed on the specimen of 20% cold-rolled stainless steel of SUS316 applied with external tensile stress. Stainless steel of SUS316 is used for the nuclear reactor because of its high corrosion resistance. The thickness of the specimen was 0.3 mm. The average size of the grains in the specimen was estimated to be about 0.1 mm from the electron backscatter diffraction (EBSD) image. This specimen was mounted on the tensile testing machine set on the sample stage of the diffractometer. In this experiment, we applied the tensile stresses of 300 and 380 MPa to the specimen, where the former was the yield stress and the latter was the 0.1% proof stress. The direction of the external tensile stress is in an x-axial direction, as shown in Fig. 1.

Firstly, the grain boundary image of the specimen is shown in Fig. 2(a). The dark contrast in the image indicates the grain boundary. It was confirmed by comparison with the EBSP image (Fig. 2(b)) that this image was reasonable. Secondly, we evaluated the internal stress that occurred in 7 grains selected among the grains indicated in this image. The grains are indicated by the numbered-filled circles in Fig. 2(a). We measured more than three crystal lattice spacings to fully determine the component of the stress tensor.

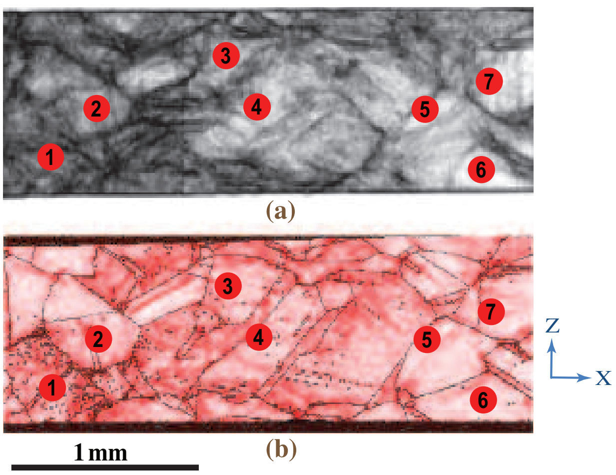

Fig. 2. Image of grain distribution obtained by the developed technique **(a)** and kernel average misorientation map of EBSP **(b)**.

Figure 3 shows the dependence of the internal stress measured at the selected grains on the external tensile stress. This data indicates that normal stress occurred on the plane perpendicular to the direction of the applied stress. The average value of the measured stress of seven grains is indicated by the red broken line. As shown in Fig. 3, the values of the internal stress that occurred in the grains were dispersed in a wide range and the increments of internal stress were almost proportional to the external stress. It suggested that the distribution of the internal stress in the grains of the cold rolled stainless steel was affected by the residual stress due to the cold rolling, which was dispersed in a wide range from tensile to compressive.

The measurement technique of internal stress in individual grains was successfully developed as the first step to clarify the mechanism of IGSCC. We will apply this technique to the measurement of the stress distribution around the grain boundary as the next step.

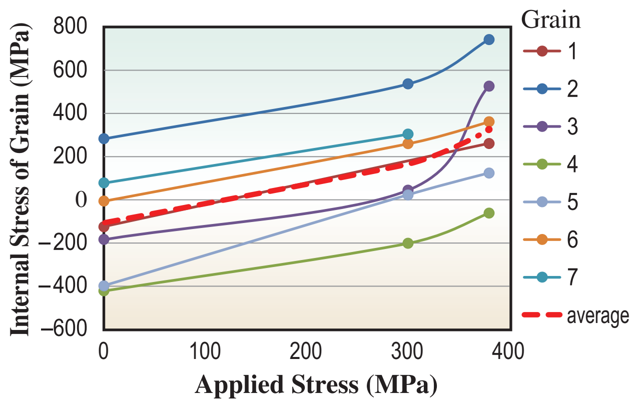

Fig. 3. Relation between applied stress and measured stress of individual grains. The grain number corresponds to that in Fig. 2.

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References

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