Scanning three-dimensional X-ray diffraction microscopy for non-destructive observation of plastic deformation in metallic materials

Metals and alloys are formed into functional shapes using a wide variety of plastic-forming operations in automobile industry. The development of advanced metallic materials with high plastic formability and high strength has led to the reduced weight of automobile parts. Because conventional experimental methodologies for the development of such materials are too expensive and time consuming, researchers have attempted to construct models to predict the plastic behaviors of materials. Although many models are now available, there is no adequate way to judge which model is useful owing to a lack of experimental methods to validate such models.

The plastic deformation of metals and alloys is microscopically caused by crystallographic slips or twinning, which are well understood in great detail through observations by transmission electron microscopy. The slips and twinning caused by mechanical loads give rise to macroscopic changes in crystallographic orientations. The macroscopic behaviors of slips or twinning in polycrystals have been investigated using orientation-imaging microscopy (OIM) by the electron backscatter diffraction (EBSD) technique. However, OIM information obtained by the EBSD technique is limited to close to the surface. Although successive serial sectioning of samples allows us to obtain three-dimensional information, changes in orientations cannot be tracked because the observed volumes are destroyed. Recently, a synchrotron radiation (SR) X-ray diffraction technique has enabled new experimental methods. High-energy X-ray diffraction approaches based on 3D X-ray diffraction (3DXRD) microscopy [1] enable the non-destructive 3D mapping of orientations.

In 3DXRD microscopy, an SR beam illuminates a polycrystalline sample and X-ray diffracted beams from multiple grains are detected by near- and far-field area detectors. Orientations of individual grains are analyzed from the positions of diffraction spots on the far-field detector using a polycrystal indexing technique [1]. The grain positions and shapes are analyzed from the positions and shapes of diffraction spots on the near-field detector. The main problem with 3DXRD-type experiments is the overlap of diffraction spots from multiple grains. This implies that there is a limit to the number of grains that can be illuminated by an incoming SR beam. Industrial metallic materials such as steel tend to cause this overlap of polycrystalline diffraction spots due to a large number of grains, mosaicity, strains, and textures. In the case of such samples, the wide sheet-like SR beam illumination often used in 3DXRD-type experiments is applicable to those with relatively small diameters. However, it is often not possible to investigate the mechanical characteristics of bulk materials using narrow samples rather than bulky samples.

In this study, we have proposed a modified 3DXRD technique, named scanning 3DXRD, as a solution to the main problem [2]. The scanning 3DXRD approach is considerably different from 3DXRD. First, the overlap of polycrystalline diffraction spots is reduced as much as possible by SR microbeam illumination. Second, only the far-field detector is employed to provide sufficient space around the specimen for an *in situ* stress rig. The diffraction spots are collected through scans of the sample rotation, ω, and the sample translation, *X*, as shown in Fig. 1. How is a 2D orientation map reconstructed from the ω-*X* scan data? The answer is as follows, (1) Only the diffraction spots obtained when the microbeam illuminates an arbitrary point inside the sample are extracted. (2) The diffraction spots are divided into groups in which the same crystal is the origin of the diffraction spots by using the polycrystalline indexing technique [1]. (3) The selection of the crystal corresponding to a point is performed on the basis of the number of diffractions for each group. The probability of microbeam illumination through a point is the largest among the groups for extracted diffraction sets because it is the imaginal rotation center. The crystal with the largest number of diffraction spots is selected as that corresponding to the given point. A 2D orientation

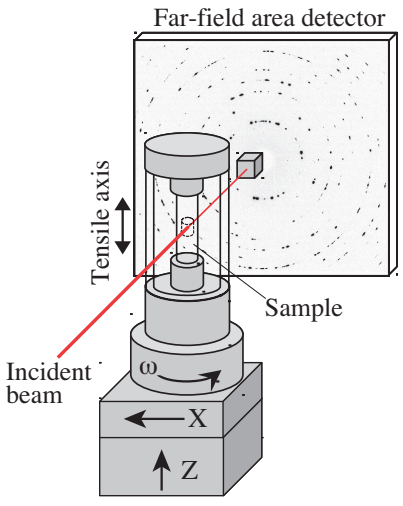

Fig. 1. Schematic of scanning 3DXRD microscopy setup.

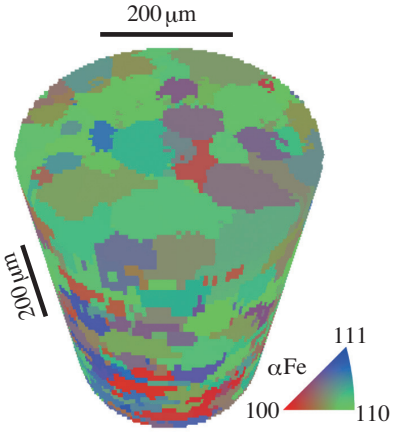

Fig. 2. 3D orientation map of coarse-grained polycrystalline iron. The colors correspond to the inverse pole figure in the tensile direction.

map is thus reconstructed by sweeping the given point. The stack of 2D orientation maps results in a 3D orientation map as shown in Fig. 2, where orientations are represented by colors, and single domains with the same color correspond to single grains.

As a first demonstration, a scanning 3DXRD experiment was performed at the Toyota beamline **BL33XU** using an SR beam through 20 μm slits and a coarse-grained pure iron sample with an average grain size of 60 μm. Actually, the result is the 3D orientation map in Fig. 2. Then, using an *in situ* stress rig, the 3D orientation map in a deformed state is obtained in the same way. Thus, the changes in orientations, i.e., crystal lattice rotations, are observed [3].

By adopting the observed 3D orientation map before deformation as the initial orientation map in the model, crystal lattice rotations caused by plastic deformation can be simulated. Figure 3 shows the observed and simulated orientations for some typical coarse grains at macroscopic tensile strains of ε = 0.2%, 4.0%, 8.0%, and 10.7% [4]. The simulation reproduced significant observed rotation behaviors: (i) mean rotations toward the preferential <110> orientation of the body-centered-cubic tensile texture, (ii) an increase in intragranular misorientation, which is seen as the spread of orientations in single grains, and (iii) intragranular multidirectional rotations of grains near the <100> corner toward orientations between <110> and <111>.

Thus, the scanning 3DXRD microscopy method was demonstrated using a coarse-grained pure iron sample. The next step is the observation of industrial materials such as steel with a grain size of 10–20 μm. Toward this challenge, a scanning 3DXRD microscope apparatus using a high-energy microbeam with a beam size of 1–2 μm has been installed at the Toyota beamline BL33XU. In terms of the reconstruction algorithm, improvements are needed because the above observed spread of orientations (ii) in single grains is smaller than the simulated spread, which implies that the experimental reconstruction method underestimates intragranular misorientations. Finally, we are planning to apply the improved scanning 3DXRD microscopy method to the validation of a crystal plasticity finite element model that predicts slip deformation in carbon steel [5]. Then, we will work on a current hot topic of 3DXRD, the investigation of twin deformations in magnesium alloy, toward improving the formability of next-generation light alloys in automobile industry.

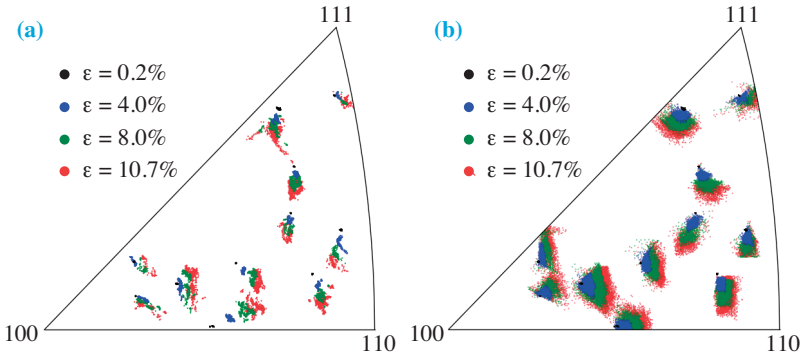

Fig. 3. **(a)** Observed and **(b)** simulated grain rotations represented by the inverse pole figures in the tensile direction for some coarse grains at ε = 0.2%, 4.0%, 8.0%, and 10.7%.

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