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3D visualization of dislocation arrangement using scanning electron microscope serial sectioning method

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We performed the three-dimensional visualization of dislocations through serial sectioning and use of SEM electron channeling contrast (ECC) images for a crept nickel-based alloy. We successfully reconstructed a volume of approximately 7.5 μm^3, including dislocation arrangements, by performing calculations based on the continuous tomograms of ECC images. By incorporating the information on crystal orientation obtained by the electron back-scattered diffraction, we verified that the three-dimensional arrangement of dislocations, such as slip plane, was accurately reflected in the three-dimensional volume.

Keywords: Dislocation; 3D reconstruction; Scanning electron microscopy (SEM); Electron channeling contrast imaging (ECCI); Nickel alloys

A transmission electron microscope (TEM) has usually been used for observing dislocations. However, it has been recently reported that dislocations can be observed using a scanning electron microscope (SEM) through electron channeling contrast (ECC) utilizing the channeling phenomena of electron beam [1–6]. Observation of SEM-ECC images utilizing the channeling phenomena of electron beam is possible by using the contrast caused by the difference in the penetration depth of electron beam, depending on the angles between their incident directions and the crystal orientation, when irradiating crystalline samples. If dislocations exist in crystals, it is possible to observe them in ECC images under appropriate conditions because the disorder in crystal structures causes the scattering conditions of electron beam to vary in their vicinity.

In a TEM observation, it is possible to obtain a three-dimensional volume of the microstructure using the electron tomography method for obtaining several images by intermittently tilting a sample to large angles in a microscope. A three-dimensional visualization of dislocation arrangements has recently been performed using the electron beam tomography method by tilting samples while maintaining proper diffraction condition [7–9]. In contrast, the serial sectioning method [10,11] is a well-known technique for the three-dimensional visualization of the microstructure using a SEM. In the serial sectioning method, three-dimensional volumes are obtained by acquiring continuous tomographic images by cutting samples, mainly using the focused ion beam (FIB) equipment and alternately and continuously observing the SEM images, followed by reconstruction and performing calculations. While the TEM tomography method utilizes thin films or nanopillar-like samples, the SEM serial sectioning method has the advantage that a wider range of information about the microstructures can be derived in a form that is close to the bulk interior because it enables the use of bulk materials. Although it is possible, in principle, to three-dimensionally observe and analyze dislocation arrangements existing within a space of approximately 1000–100,000 μm^3 by combining the ECC dislocation imaging and the SEM serial sectioning method, there is no report on this method. Dislocation arrangements of materials subjected to plastic deformation may be non-uniform on a micrometer scale, and it can be easily imagined that a wide range of three-dimensional dislocation arrangement analysis by SEM is useful for elucidating the origin of material properties. Therefore, this study aims at establishing a technique for the three-dimensional visualization of dislocation arrangements using dislocation image observation via ECC and the serial sectioning method.

We used a nickel-based heat-resistant alloy (Alloy 617) as a sample [12,13]. We conducted a constant-load uniaxial creep test at 973 K and 350 MPa to introduce dislocations into the material. The test was interrupted when 106 h elapsed after the loading. The plastic strain imposed on the sample was 0.016. We picked out a sample from the parallel portion of the creep test piece for microstructural observations.

We used a field-emission type scanning electron microscope (FE-SEM) equipped with an FIB device (Scios, manufactured by FEI Co.) for our observations. We used

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a concentric silicon semiconductor back-scattered electron (CBS) detector to acquire the ECC images. Then, we set the accelerating voltage to 15 kV, the current to 1.6 nA, and the working distance, which corresponds to the distance from the lower edge of the pole piece of the electron gun to the surface of the sample, to 4.8 mm. The convergence angle of the electron beam in this setting is 4.1 mrad. We observed the ECC images by inserting the CBS detector between the sample and the pole piece of the electron gun and set the sample surface perpendicular to the incident direction of the electron beam and parallel to the CBS detector surface. A Ga^+ ion gun on this device was placed at an angle of 52° from the electron gun; we sliced the observation plane from the side by tilting the observation plane of the ECC image such that it was parallel to the direction of Ga^+ ion irradiation. We set the accelerating voltage of Ga^+ ions to 5 kV, the current value to 77 pA, and the working distance to the Ga^+ ion gun to 19.0 mm. Moreover, we measured the crystal orientation of the field of view using an electron back-scattered diffraction (EBSD) device placed in the same chamber as the FIB/SEM by tilting it to 70° from the sample position when observing the ECC images and setting the accelerating voltage to 15 kV, the current value to 1.6 nA, and the working distance to 7 mm. For a crystal grain near the edge of the sample where dislocations were clearly observed in non-tilted ECC images, we measured the crystal orientation of the observation plane by EBSD to determine the incident orientation of electron beam to the crystal grains to be observed. Then, we alternately repeated the FIB slicing and the SEM observation under the abovementioned conditions to obtain continuous tomographic images. We obtained a total of 33 photographs, 330 nm in the thickness direction, by setting the FIB slicing range to 10 μm in width and 50 μm in depth, with a FIB slicing interval of 10 nm. We set the resolution of each image at 3072×2184 pixels and the irradiation time per pixel to 10 μs for acquiring images of sufficient spatial resolution and signal-to-noise ratio. The pixel size on the observation plane was 1.3 nm. The spatial resolution in the thickness direction was the same as the slice interval, i.e., 10 nm. To extract only the contrasts of the dislocation lines from the acquired ECC images, we homogenized contrast of the matrix and the γ' phase particles as backgrounds using the image analysis software ImageJ. Then, the positions of each tomographic image and the three-dimensional drawing were corrected and the continuous tomographic images were analyzed using the software Amira (manufactured by Maxnet, Inc.).

An example of the dislocation arrangement observed in the ECC image is shown in Figure 1(a). The vertical direction of the paper surface is parallel to the tensile direction in the creep test. Dislocations can be observed as bright lines in the right side crystal grain which shows dark matrix. The spherical γ' phase was present, which was densely precipitated in a matrix, and the dislocations moved while curving between the precipitates. From an EBSD measurement of the crystal orientation for the right side crystal grain, shown in Figure 1(a), the incident direction of the electron beam to this crystal grain was [14 2 19]. Figure 1 (b) shows the result of simulating the diffraction pattern excited under the condition of acceleration voltage in the ECC observation (15 kV) and the incident orientation [14 2 19] with the angle scale. This simulation was carried out using an electron beam diffraction simulation software ReciPro. White spots in Figure 1(b) represent diffraction spots from the crystal plane indicated by the index, and the white line represents the Kikuchi line. It can be seen from Figure 1(b) that 1 1 1 and 2 0 0 systematic row excitation under this observation condition. It should be noted here that, there are some errors for estimation of the incident direction using an EBSD method, that is, the determination of a crystal orientation includes an error of 8.7 mrad (0.5 deg.) [14] and the accuracy of the SEM stage tilting is 1.8 mrad (0.1 deg.).

Figure 2 shows eight consecutive images of continuous tomographic observation. The SEM back-scattered electron images at a high magnification revealed that approximately 7% of the γ' phase particles existed in an area fraction with a particle diameter of approximately 14 nm in this sample, however, they cannot be observed in Figure 2 because their contrasts are homogenized by image processing. On the other hand, bright dislocation lines can be observed within the dark matrix in Figure 2. In addition, it is possible to observe how the positions and the shapes of dislocation lines change gradually as the slicing progresses. Focusing on the dislocation line within the red circle in Figure 2, it began to be observed in the image of the 21st slice and it was observed most clearly in the 26th slice. The dislocation line disappeared suddenly in the 27th slice, and almost whole of the dislocation line of interest disappeared in the 28th slice. Because the thickness at a single slicing was 10 nm, the thickness at which the contrast of the dislocation line was visible was 60–70 nm, and it was observed more clearly when the positions of the dislocation line were closer to the sample surface. According to Wilkinson et al. [15], the

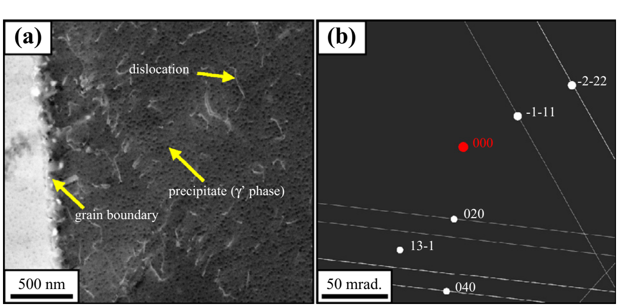

Figure 1. (a) SEM-ECC image in the grain which visualizes a 3D dislocation structure, (b) a simulated diffraction pattern corresponding to the observed grain with the incident beam direction of [14 2 19] and the acceleration voltage of 15 kV.

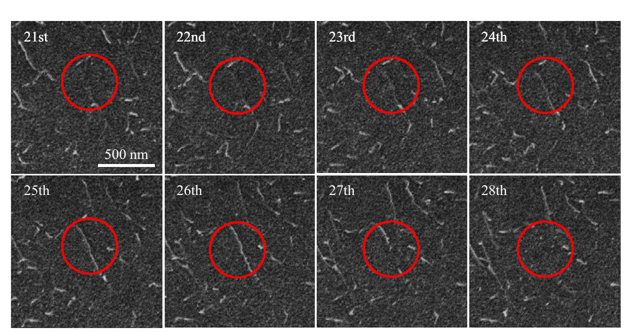

Figure 2. Serial slices of SEM-ECC images which focused on the appearance of the dislocation structure in the slice thickness of 10 nm.