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

S. Yamasaki,⇑ M. Mitsuhara, K. Ikeda, S. Hata and H. Nakashima
Faculty of Engineering Sciences, Kyushu University, Kasuga 816-8580, Japan
Received 6 January 2015; revised 28 January 2015; accepted 2 February 2015
Available online 12 February 2015

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 μm³, 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.

© 2015 Acta Materialia Inc. Published by Elsevier Ltd. This is an open access article under the CC BY license (http://creativecommons.org/licenses/by/4.0/).

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 μm³ 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
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 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](image1.png)

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 [14219] and the acceleration voltage of 15 kV.

![Figure 2](image2.png)

Figure 2. Serial slices of SEM-ECC images which focused on the appearance of the dislocation structure in the slice thickness of 10 nm.
penetration depth of the electron probe into the material is approximately \( 5 \xi_e \) when observing the dislocation using the ECC image. Here, \( \xi_e \) is the extinction distance of the diffracted wave obtained by the following formula [16]:

\[
\xi_e = \frac{\pi V_c \cos \theta}{2F(\theta)}
\]

where \( V_c \) is the volume of the unit cell \([\text{m}^3]\), \( \theta \) is the Bragg angle \([\text{rad}]\), \( \lambda \) is the wavelength of an electron beam \([\text{m}]\), and \( F(\theta) \) is the crystal structure factor for a diffraction plane \([\text{m}]\). With regard to diffracted waves of \( g_{111} \) and \( g_{200} \) excited by the observation condition of this study, the extinction distance with the accelerating voltage of electron beam at 15 kV is calculated from the equation [1] as \( \xi_{111} = 11.7 \text{ nm} \) and \( \xi_{200} = 13.3 \text{ nm} \), respectively. Therefore, the penetration depth, \( 5 \xi_e \), of an electron beam probe in this study ranged between 59 nm and 67 nm, which was nearly consistent with 60–70 nm thickness where the dislocation line was observed in the continuous tomogram.

Figure 3 shows the three-dimensional volume obtained by the reconstructive calculation of continuous tomographic images. The portions of dislocation lines are indicated in orange, and one of them is highlighted in green. In addition, matrix and precipitates are erased through contrast adjustment. Figure 3(a) shows the image where the three-dimensional volume is viewed from a direction parallel to the incident beam direction of \([142\,19]\). The highlighted dislocation segment passes through the three-dimensional volume in the \( Z \) direction. And it can be observed that the dislocation lines, observed in fragments in the continuous tomogram, continue without any breaks and curving stERICALLY by displaying three-dimensionally. In Figure 3(b) the three-dimensional volume is viewed from the inclination beam direction \([0\,\,0\,\,-1\,\,1]\). The tensile direction in the creep test shown in the figure is \([5\,5\,3\,\,-4]\) in the coordinate system of the observed crystal grains, and the primary slip system in which the Schmid factor becomes the maximum under this condition is \((-1\,1\,1)\) \([11\,0]\). As shown in Figure 3(b), most dislocations including the highlighted one arrange on the \((-1\,1\,1)\) plane. It consistent with the expectation from the Schmid factor, therefore, the three-dimensional volume properly reflects the three-dimensional dislocation arrangements of the material. In addition, some of the dislocations deviate from the \((-1\,1\,1)\) plane, suggesting that cross-slip or climbing motion of dislocations could occur. Thus, reconstructing the three-dimensional volume and observing it from any direction can verify that several slip systems are active and the cross-slip of dislocations occurs in the materials. It also can estimate a dislocation density from the three-dimensional volume which includes well-defined dislocation segments. A total volume of the identified dislocation segments in the reconstructed three-dimensional volume was evaluated. Then, a dislocation density was calculated as \( 3.4 \times 10^{13} \text{ m}^{-2} \) by dividing the total volume of the dislocations by the cross-sectional area of the dislocation segment. The estimated dislocation density is a reasonable value for a crept nickel-based alloy.

Figure 4 shows an image when viewing the highlighted dislocation from an angle with a tilt of 90° from the incident beam direction \((\text{XZ view})\) and the vertical cross section of the dislocation line at the position of the red dashed line. The slice direction of FIB is also shown in the figure. In the XZ view, a dislocation line has a thickness in the \( Z \)-direction, and its cross section in a three-dimensional volume has a teardrop shape when viewing its vertical cross section. This can be explained from the continuous tomogram of the ECC images shown in Figure 2. In other words, it is believed that a single dislocation line was observed in several continuous tomographic images because the penetration depth of the electron beam was larger than the thickness of the slice, and the dislocation line in the images of lower number of slices was in lower contrast but became clearer as the slicing progressed. However, its contrast disappeared when the volume containing the dislocation line was sliced. Therefore, dislocation lines in the three-dimensional volume had their thicknesses in the \( Z \)-direction, and their cross-section became teardrop-shaped. In the TEM tomography method, an elongation of the reconstructed images is known to occur along the incident beam direction at a tilt of 0° because of a lack of information (missing wedge) caused by the restriction of the tilt angles [17]. It should be noted that the similar elongation described above appears in the three-dimensional volume of dislocations obtained by the SEM serial sectioning method utilized ECC images, although its origin is different from that of the TEM tomography. In view of this, it is necessary to select the appropriate acceleration voltage and slice thickness when performing the three-dimensional visualization of dislocation arrangements using this method.

The width and depth of slices were determined to be 10 μm and 50 μm, respectively, and in this study, the observation was terminated after 35 slices for the convenience of spatial resolution in the observation magnification and the time required for serial sectioning. However, this method enables the three-dimensional visualization of dislocation arrangements throughout a single crystal grain in a polycrystalline material as long as the ECC of the observed crystal grain remain unchanged, which could be an effective tool for visualizing bulk dislocation arrangements. In future, optimization of accelerating voltage and slice thickness would be required, considering the penetration depth.
of electron beam, along with an automated operating procedure to obtain continuous tomographic images on a micrometer scale. Furthermore, magnetic influences, such as the effects of magnetic fields on samples, are alleviated by constructing an objective lens of a suitable structure in the current SEM. Therefore, this method is also considered to be effective for the three-dimensional visualization of dislocation arrangements, such as those in steel, that are difficult to observe with the TEM tomography method.

In summary, we were successful in the three-dimensional visualization of dislocation arrangements of a nickel-based heat-resistant alloy subjected to creep deformation through the serial sectioning of SEM-ECC images. By tilting the reconstructed three-dimensional volume in any direction based on the information of crystal orientation obtained by EBSD, we verified that the dislocation lines were arranged on the (−1 1 1) planes as the main slip plane and that some of the dislocations were in motion while deviating from the (−1 1 1) plane. In addition, we elucidated the reason why dislocation lines had a thickness along the slice direction in the three-dimensional volume by considering the penetration depth of an electron probe in the observed ECC images. It may also be possible to use this method to visualize bulk dislocation arrangements on the micrometer scale.

This work was partly supported by the Grants-in-Aid for Scientific Research from the Japan Society for the Promotion of Science (JSPS) and the Ministry of Education, Culture, Sports, Science and Technology (MEXT), Japan.

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.scriptamat.2015.02.001.

[1] A.J. Wilkinson, P.B. Hirsch, Micron 28 (1997) 279.
[2] B.C. Ng, B.A. Simkin, M.A. Crimp, Ultramicroscopy 75 (1998) 137.
[3] M.A. Crimp, Microsc. Res. Tech. 69 (2006) 374.
[4] N. Kuwano, M. Itakura, Y. Nagatomo, S. Tachibana, J. Electron Microsc. 59 (2010) S175.
[5] I. Gutierrez-Urrutia, D. Raabe, Scripta Mater. 66 (2012) 343.
[6] S. Zaefferer, Nahid-Nora Elhami, Acta Mater. 75 (2014) 20.
[7] S. Hata, H. Miyazaki, S. Miyazaki, M. Mitsuhara, M. Tanaka, K. Kaneko, K. Higashida, K. Ikeda, H. Nakashima, S. Matsumura, J.S. Barnard, J.H. Sharp, P.A. Midgley, Ultramicroscopy 111 (2011) 1168.
[8] M. Tanaka, M. Honda, M. Mitsuhara, S. Hata, K. Kaneko, K. Higashida, Mater. Trans. 49 (2008) 1953.
[9] G.S. Liu, I.M. Robertson, J. Mater. Res. 26 (2011) 514.
[10] S. Zaefferer, S.I. Wright, D. Raabe, Metall. Mater. Trans. 39A (2008) 374.
[11] M.D. Uchic, M.A. Groeber, D.M. Dimiduka, J.P. Simmons, Scripta Mater. 55 (2006) 23.
[12] W.L. Mankins, J.C. Hosier, T.H. Bassford, Metall. Trans. 5 (1974) 2579.
[13] J.K. Benz, L.J. Carroll, J.K. Wright, R.N. Wright, T.M. Lillo, Metall. Mater. Trans. 45A (2014) 3010.
[14] A.J. Wilkinson, Scripta Mater. 44 (2001) 2379.
[15] A.J. Wilkinson, G.R. Anstis, J.T. Czernuszka, N.J. Long, P.B. Hirsch, Philos. Mag. A 68 (1993) 59.
[16] L. Reimer, H. Kohl, Transmission Electron Microscopy, fifth ed., Springer, New York, 2008.
[17] I. Arslan, J.R. Tong, P.A. Midgley, Ultramicroscopy 106 (2006) 994.