**Tomographic 3D observation of crystalline microstructures using a STEM dark-field method**

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A conventional dark-field (DF) observation in TEM uses an objective aperture at the back focal plane to select a diffracted wave. For some cases of crystal structures with large unit cells, it may be difficult to select only the desired diffracted wave, because the distance between the adjacent diffraction spots is short. Also depending on the apparatus, it may be impossible to tilt the specimen to high angles because the sample stage and the objective aperture touch each other. These issues make tomography observation difficult. In this study, we attempted a STEM-DF observation method that enables the similar diffraction contrast observation as TEM-DF without using objective aperture, and also enables its application to tomographic observation. This STEM-DF method does not use an objective aperture and it is easy to avoid the influence of adjacent diffracted waves.

In this study, we used the proposed STEM-DF method for the 3D morphological observation of $D1_a$ (I4/m) type $Ni_4Mo$ ordered phase precipitated in Ni-Mo alloy (18 at.% Mo) [1]. Using the TEM/STEM FEI Titan G2 microscope at an accelerating voltage of 300 kV, we adjusted the illumination-lens system so that the convergence semi-angle was 0.6 mrad, took only the desired diffracted wave on the STEM-BF detector and acquired STEM-DF tilt-series data sets, as shown in Fig. 1 (a) and (b). The electron beam transmittance decreases with the increase of the sample tilt angle and thickness contours appeared in the $Ni_4Mo$ precipitates, as denoted with the circle in Fig. 1 (b).

Furthermore, we applied the STEM-DF method to the observation of dislocations in a Mo single crystal. We adjusted the illumination-lens system so that the convergence semi-angle was 1.5 mrad, selected the diffracted wave of **g** on the STEM-BF detector under the Bragg condition for 3**g**, and acquired STEM-DF weak-beam (WB) images [2]. Fig.1 (c) and (d) show respectively STEM-DFWB and STEM-BF images of dislocations in a Mo single crystal. The STEM-DFWB image (c) gives contrast near the dislocation cores, and the dislocations line widths were about a half that in the STEM-BF image under the two-beam excitation condition (d). Such an effectiveness of STEM-DFWB image contrast was kept up to high specimen-tilt angles, which in promising for high-resolution dislocation tomography.

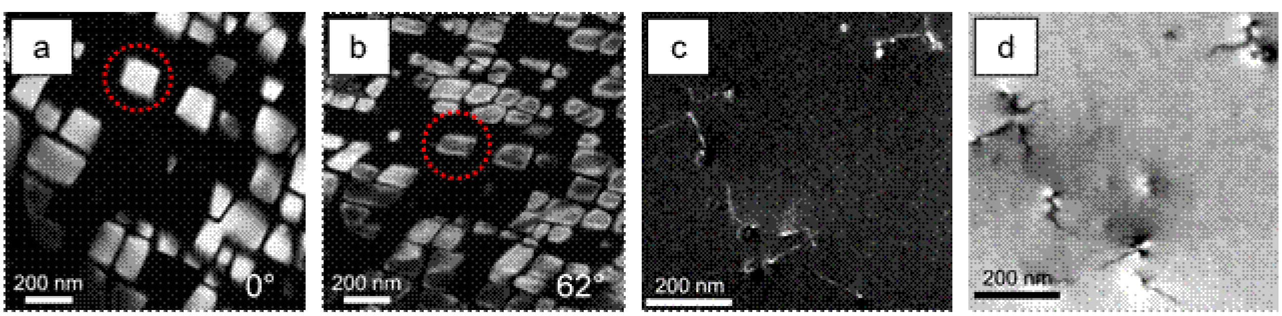


Fig.1 (a) and (b) Part of STEM-DF tilt-series images of $Ni_4Mo$ precipitates, and (c) STEM-DFWB and (d) STEM-BF images of dislocations in a Mo single crystal.

[1] K. Kimura et al., J. Electron Microsc. 54, 373 - 377 (2005).

[2] K. Yoshida et al., Microsc. 66, 120 - 130 (2017).