Electron channelling contrast imaging of individual dislocations in geological materials using a field-emission scanning electron microscope equipped with an EBSD system

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Abstract: Dislocation-related defects in minerals govern globally important rheological processes such as mantle convection in the deep Earth where dislocation creep is a common deformation mechanism of the constituent minerals. Understanding such processes requires the direct observation of individual dislocations in minerals and across their interfaces. Using electron channelling contrast imaging (ECCI) in a conventional field-emission scanning electron microscope (FE-SEM), we successfully observed individual dislocations in experimentally deformed polycrystalline ferropericlase as well as in natural olivine without special surface treatments to highlight dislocations such as oxidation decoration or chemical etching. Combining backscattered electron (BSE) imaging with electron backscatter diffraction (EBSD) techniques, we refined the two-beam condition of a Kikuchi band at the Bragg orientation to maximize the visibility of individual dislocations with a particular Burgers vector. The dislocation microstructures of progressive subgrain rotation as dominant recrystallization mechanism and the co-activation of two non-coplanar slip systems are demonstrated in ferropericlase and olivine, respectively. Inclined dislocations in olivine are also visualized in end-on view in ECCI. Orientation optimized ECCI in a FE-SEM may serve as an alternative to diffraction contrast imaging in transmission electron microscopy. ECCI shows promise as a non-destructive imaging of individual dislocations of rock-forming minerals.

Key-words: backscattered electron; electron channelling contrast image (ECCI); subgrain boundary; dislocation; Kikuchi band; two-beam condition; electron backscatter diffraction; subgrain rotation; inclined dislocation.

1. Introduction

Observation of individual dislocations in minerals is indispensable for the understanding of the deformation mechanisms in rocks at sub-micrometre scale. Transmission electron microscopy (TEM) has been so far the main tool to observe individual dislocations in rock-forming minerals. On the other hand, the chemical etching and oxidation decoration method are often used for dislocation density measurements especially in Fe-bearing olivine, by using optical microscopy (Wegner & Christie, 1974; Kohlstedt et al., 1976) or scanning electron microscopy (Karato, 1987; Farla et al., 2011). With those methods, the Burgers vector and line direction of a dislocation can be determined with certainty only if it can be traced into a small-angle tilt boundary, where the Burgers vector of a pure edge dislocation is normal to the plane of the boundary or into a twist boundary, where the Burgers vector of a pure screw dislocation is parallel to the dislocation line direction, as noted by Kohlstedt et al. (1976). Otherwise, high-angular resolution electron backscatter diffraction (HR-EBSD), based on diffraction pattern cross-correlation, offers a powerful new approach that has been utilised to analyse geometrically necessary dislocation (GND) density in experimentally deformed olivine (Wallis et al., 2016). However, this is also not a direct observation of dislocations. The measured dislocation density depends on the step size in the EBSD map and HR-EBSD only informs on the GND densities and does not reveal the statistically stored dislocations (SSDs).

Currently, electron channelling contrast imaging (ECCI) in a conventional field-emission scanning electron microscope (FE-SEM) is developing into an alternative tool to observe crystal defects in metallic alloys, semiconductors and ceramics containing relatively heavier elements in the material science fields (e.g., Zaefferer & Elhami, 2014). The channelling contrast from differently oriented grains having the same chemical compositions is well-known in backscattered electron (BSE) images and fore-scattered electron (FSE) images (“Orientation contrast”), in comparison to the averaged atomic number contrast (“Z-number contrast”) in conventional SEM imaging techniques (Reimer, 1998). The rapid change in BSE intensity occurring as the beam is being scanned through the Bragg angle arises as a result of the significant changes in the Bloch wave excitations (e.g., Fig. 2 in Joy et al., 1982; Figs. 1 and 2 in Kaboli et al., 2015).