doi:10.1017/S1431927611006398
Microsc. Microanal. 17 (Suppl 2), 2011

Development of Rotation Dark-Field Imaging for Mapping Secondary-Phase Distributions in Nano-Scale

M. Watanabe*, J. Cubero**, K. Edalati** and Z. Horita**
* Dept of Materials Science and Engineering, Lehigh University, Bethlehem. PA 18015.
** Dept. of Mater. Sci. & Eng., Faculty of Eng., Kyushu University, Fukuoka 819-0395, Japan.

Many materials properties are significantly influenced by their microstructures, for example, sizes and distributions of nano secondary phases. The secondary phases in nanoscale are normally characterized not only by conventional imaging in (scanning) transmission electron microscopy ((S)TEM) but also by spectrometry-basis analysis such as X-ray energy dispersive spectrometry (XEDS) and electron energy-loss spectrometry (EELS). One of the best approaches to visualize two-dimensional distributions of those secondary phases is elemental mapping, which can be performed by XEDS and/or EELS in STEM and by energy-filtering in TEM. However, these spectrometry-based approaches can be restricted especially when there are least difference in composition between matrix and secondary phases. The secondary phases can also be seen by dark-field (DF) imaging with a selection of appropriate reflections. However, selecting the reflections manually for DF imaging would not show all the distributions of secondary phases if the microstructure consists of multiple nano-grains.

To visualize the complete distributions of secondary phases, a rotation DF imaging (RotationDFI) procedure has been developed, which acquires a series of DF images by tilting the incident beam to a specific Bragg angle of the secondary phase and rotating it circularly. The individual DF images are recorded at certain rotation angles. A schematic diagram of the RotationDFI procedure is shown in Fig. 1(a). This procedure has been developed in the Gatan DigitalMicrograph platform. The main dialog for RotationDFI is also shown in Fig. 1(b). Using a diffraction pattern from polycrystalline NiO, the tilt angle was calibrated and the accurate beam rotation without noticeable hysteresis was confirmed as shown in Fig. 1(c). This proposed approach is essentially equivalent to a DF image acquisition under hollow-cone illumination, which has been used for grain enhancement imaging [1], dislocation characterization [2, 3] and secondary-phase distribution mapping [4, 5]. In contrast, multiple DF image acquisition is synchronized with the beam rotation in the present approach. So, any DF image can be reconstructed by integration at any rotation angle of interest in post processing as spectrum-imaging datasets are treated. Since multiple images are acquired, the total acquisition time is longer and hence spatial drift during acquisition would be an issue. However, the spatial drift can be corrected by bright-field (BF) image series acquired between successive DF images.

An example of the RotationDFI method applied to a severely deformed pure Ti is shown in Fig. 2. In previous study, it has been found that the ω phase is transformed from α-Ti by the high-pressure torsion (HPT) method [6]. A diffraction pattern (b) obtained from a field of view (a: BF image) indicates that there is deformation-induced ω phase in the matrix. Obviously, any chemical analysis is useless since there is no composition difference between α and ω phase in the pure Ti. A RotationDFI series was acquired at the tilt angle of 8.8 mrad, corresponding to the ω 001 reflections. A DF image integrated from the RotationDFI dataset is shown in Fig. 2(c) and the brighter areas represent the ω phase. The RotationDFI approach thus reveals fine secondary-phase distributions efficiently.

References

[1] B. Yao et al, *Micron*, **41** (2010), 177.
[2] Z.L. Wang, *Ultramicrosc*. **53** (1994), 73.
[3] B. Yao et al, *Micron*, **42** (2011) 29.
[4] R.A. Ristau et al, *Micron* **29** (1998), 33.
[5] C.J. Bettles and C.J. Rossouw, *Micron* **31** (2000), 651.
[6] K. Edalati et al, *Metall. Mater. Trans*., **40A** (2009), 2079.
[7] The author (MW) wishes to acknowledge financial support from the NSF through grant DMR-0804528, and authors (ZH, JC and EK) acknowledge the MEXT grant "Bulk Nanometals" and the Kyushu Univ. P&P program.

Fig. 1

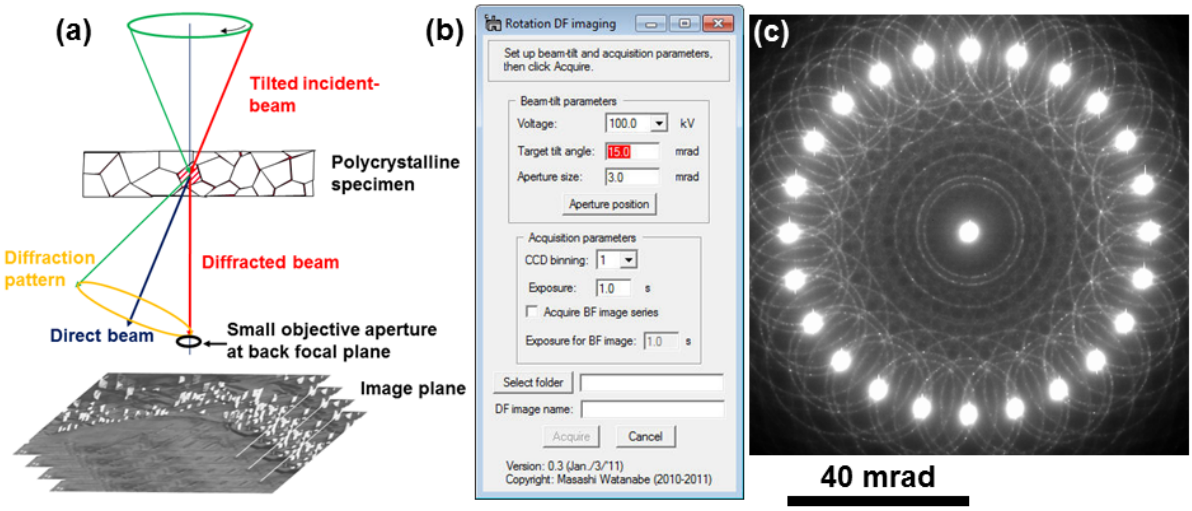

Fig. 2

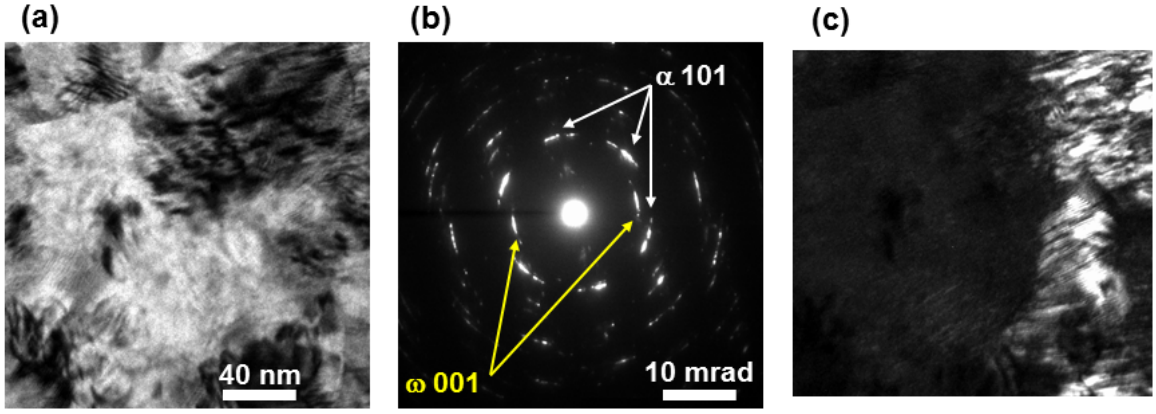

Figure 1: (a) A schematic diagram of the RotationDFI method and (b) a dialog for RotationDFI acquisition in DigitalMicrograph and (c) a calibration of tilt and rotation angle via ring patterns.

Figure 2 Application of RotationDFI to pure Ti severely deformed by the HPT process: (a) a BF image, (b) a diffraction pattern and (c) a DF image integrated from a RotationDFI dataset acquired at the tilt angle of 8.8 mrad, which corresponds to ω phase distribution.

https://doi.org/10.1017/S1431927611006398 Published online by Cambridge University Press