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IT-9-P-3080 Aberration-compensated large-angle rocking convergent-beam electron diffraction (LARCBED)

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Convergent beam electron diffraction (CBED) is a very efficient technique for acquiring two-dimensional rocking curve information in a single exposure. This is possible, because, for crystal structures having very small unit cells, the space between the Bragg spots is large enough to provide space for many non-overlapping diffraction patterns. If the sample is thick enough (typically > 100 nm), the dynamical diffraction conditions between these diffraction patterns differ enough to produce strong variations in the diffraction intensities. For materials with larger unit cells, such rocking curves must be acquired sequentially, because the distance between reflections is much smaller. Also thin crystals (e.g. nanocrystals) require a much larger tilt range than thick crystals, in order for intensity variations to be significant [1]. For thin crystals with small unit cells one may therefore acquire many CBED patterns, each with a different beam tilt, and combine them to large angle CBED (LACBED) patterns [2]. However, at large beam tilts, care has to be taken to compensate for movement of the probe on the sample due to aberrations of the objective pre-field lens. Also, imperfections in the separation between beam tilt and shift will become significant at large beam tilts. Aberrations of the imaging system add to the complexity of precisely localizing the probe on the specimen. However, this problem has been solved by the commercially available QED plug-in for DigitalMicrograph (Gatan Inc.) [1,3] which allows for automated calibration and compensation of all aberrations up to 7th order.

Here we present results of acquiring large-angle rocking-beam electron diffraction (LARBED) patterns using the QED plug-in with a convergent probe. Fig. 1 shows two example CBED patterns from the data stack (Fig. 1a and c), as well as the sum of all CBED patterns in the stack (Fig. 1b). Note that the beam tilt range (radius of ‘beam tilt disc’) was 60 mrad in each direction. At such large beam tilts the central spot of the pattern would be outside the detector area if no de-scan (compensation of beam tilt by diffraction shift) would be have been applied.

Fig. 2 shows bright-field and dark-field LACBED discs with a diameter of 120 mrad (6.9°) that have been extracted from the data stack shown in Fig. 1 by simply placing each CBED disc at the position of the pattern where it would have been recorded without applying any diffraction shift.

[1] C.T. Koch, Ultramicroscopy **111** (2011) 828 – 840

[2] R. Beanland, et al., Acta Cryst. A**69** (2013) 427–434

[3] http://www.hremresearch.com

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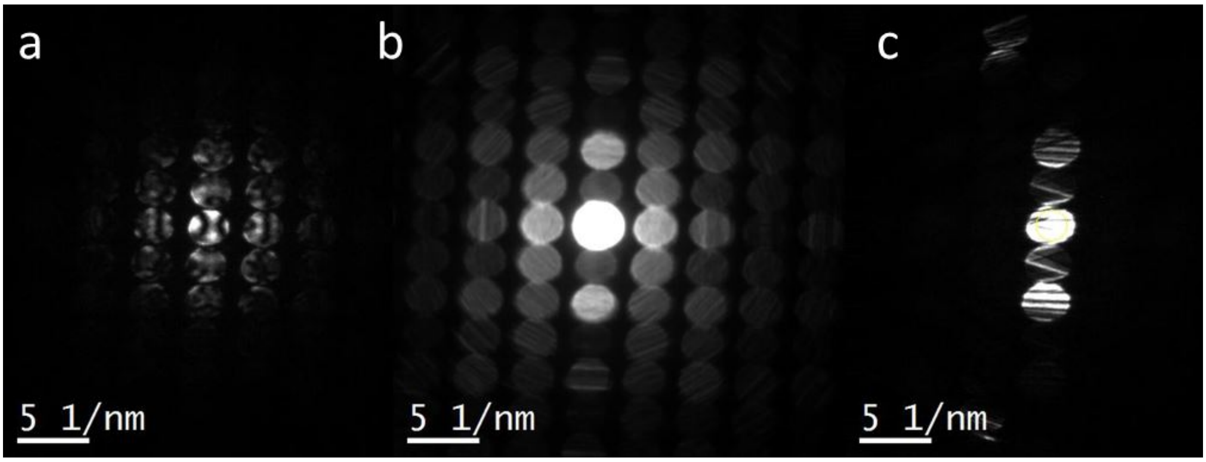

Fig. 1: CBED patterns of $SrTiO_3$ acquired using QED. a) [-110] zone axis pattern, b) sum of all patterns acquired in this experiment (data stack), and c) one of the CBED patterns acquired at high beam tilt. Aberrations of the illumination system have been compensated up to 5^{th} order.

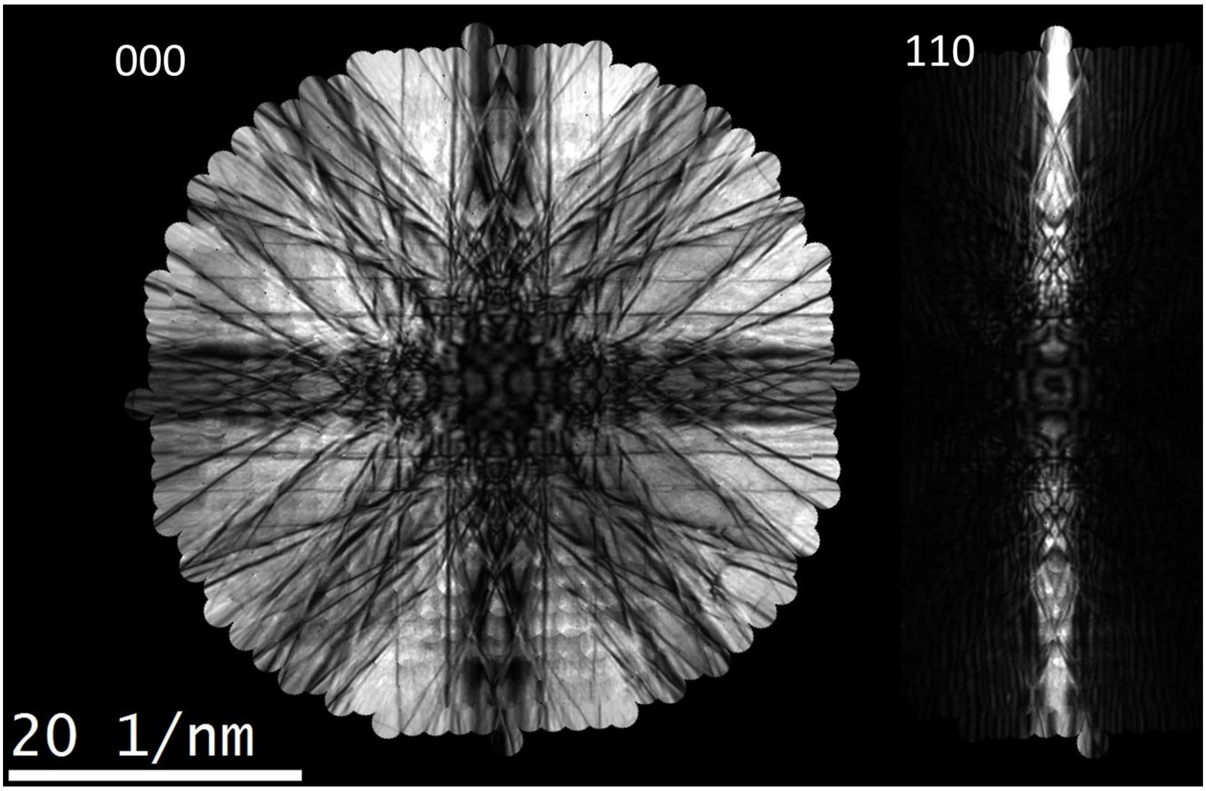

Fig. 2: Bright-field (BF) and dark-field (DF) LACBED discs extracted from the data stack shown in Fig. 1. Similar data can be extracted from any of the more than 70 reflections shown in Fig. 1b.