Dislocation tomography made easy: a reconstruction from ADF STEM images obtained using automated image shift correction

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Abstract. After previous work producing a successful 3D tomographic reconstruction of dislocations in GaN from conventional weak–beam dark–field (WBDF) images, we have reconstructed a cascade of dislocations in deformed and annealed silicon to a comparable standard using the more experimentally straightforward technique of STEM annular dark-field imaging (STEM ADF). In this mode, image contrast was much more consistent over the specimen tilt range than in conventional weak–beam dark–field imaging. Automatic acquisition software could thus restore the correct dislocation array to the field of view at each tilt angle, though manual focusing was still required. Reconstruction was carried out by sequential iterative reconstruction technique using FEI’s Inspect3D software. Dislocations were distributed non-uniformly along cascades, with sparse areas between denser clumps in which individual dislocations of in-plane image width $24\,\text{nm}$ could be distinguished in images and reconstruction. Denser areas showed more complicated stacking-fault contrast, hampering tomographic reconstruction. The general three-dimensional form of the denser areas was reproduced well, showing the dislocation array to be planar and not parallel to the foil surfaces.

1. Introduction
Dislocation tomography has been attempted previously using weak–beam dark–field (WBDF) contrast. A successful reconstruction was produced but the process was needlessly difficult. The diffraction condition had to be lined up exactly, requiring the operator to be very skilled. Moreover, the contrast was not consistent enough for acquisition software to correct for specimen drift on tilting, so the area of interest had to be manually recovered at every tilt angle — even this was not always successful.

An alternative approach was tried — using annular dark field STEM to collect intensity from a spread of dark–field beams. This is less sensitive to small specimen misalignments, making it more suited to material that has parts under significant strain, like many complex dislocation arrangements. In addition, ADF STEM images are less dynamical than WBDF images, so other strong features that made WBDF images impractical for reconstruction, such as bend contours, thickness fringes and striped dislocation contrast, are less prevalent and pose less of a problem for the final reconstruction.
Figure 1. (a) ADF-STEM image from tilt series at $-4^\circ$ tilt, 28,500x. (b) WBDF $g(3g)$ image at zero tilt, showing variation of stacking fault contrast (inset). $g = [220]$

2. Sample and experimental details
The sample of p-doped single crystal silicon was made using the Czochralski method [1]. The sample was indented, then deformed in three–point bending at 800°C, resulting in emission of dislocation cascades along $\langle110\rangle$ directions that preceded cracks around the indentation [2]. A TEM sample with foil normal [001] was prepared by Ar ion milling.

Images were taken on a Tecnai F20 operating in STEM mode at 200kV HT. An annular dark field detector of inner diameter 35 mrad and outer diameter of 105 mrad was used, with camera length 200 mm chosen empirically for best dislocation contrast. This angular range corresponds to 5.3–16.0 $\theta_{220}$ (parallel to tilt axis) or 3.8–11.4 $\theta_{200}$ (perpendicular to tilt axis). A probe of maximum width 3.94 Å and illumination angle 5 mrad was used. Images were taken every 2° over the tilt range $-70^\circ$ to $+70^\circ$. FEI’s Xplore3DTM software was used to acquire the images, and automatic correction of specimen drift was possible, though automatic focusing failed.

Image alignment and reconstruction was carried out using FEI’s Inspect3DTM software, using first filtered backprojection and finally the sequential iterative reconstruction technique (SIRT) with 10 iterations. To obtain a clear reconstruction of the stacking fault, a sharpening filter was applied to the images to remove smoothly increased background intensity close to the cascade, by subtracting a smoothed version from the original images.

3. Results
A typical image from the ADF STEM tilt series is given in Figure 1a.

The reconstruction obtained was of good quality (Figure 2). Individual dislocations could be seen in parts of the cascade where they were relatively sparse, with the narrowest distinguishable feature in the out-of-plane direction having width of 24 nm — this can be considered one kind of resolution of the reconstruction. The slip plane could be measured on the reconstruction to be (111) as previously reported [2].
Figure 2. (a) View of the reconstruction in the plane of the cascade, showing that not all sections are on the same slip plane; they are parallel, but there are steps between them. The thick volumes on these steps are an artifact of diffraction contrast. (b) Individual dislocations were reconstructed in sparse areas.

4. Discussion
Compared with weak–beam dark–field images, the thinnest ADF STEM images of dislocations are of the same width when images of comparable resolution are considered. The width of ADF STEM dislocation images is much more variable with tilt angle, however; at some angles the images are twice as wide in ADF STEM as in WBDF. Conversely, ADF STEM images have fewer thickness fringes and bend contours than WBDF images, if any, and dynamical contrast features such as striped contrast of threading dislocations are reduced or eliminated. Features such as those tend to result in artifacts in reconstructions that dominate the reconstruction and sometimes obscure the dislocations. It could be considered that deciding between the two is a compromise between resolution and reduction of artifacts.

This range of annular dark field collection corresponds to dark field spots from the zero order Laue zone, allowing diffraction contrast to form images of the defects here [3]. The background is formed from elastic and inelastic scattering. At this intermediate collection angle, the image is not wholly incoherent [4], nor can it be treated as a conventional TEM image, because the narrow probe interacts with less dispersive states compared to when wide illumination is used [5]. Our dislocations appear as bright lines with no contrast oscillations or other dynamical features apparent in these images, unlike in some previously reported studies in other orientations of silicon in which it is considered that more states are excited for oscillations to occur between [3].

Development of ADF STEM dislocation tomography has been variable; we have tried this technique on a sample of gallium nitride with much less success. The dislocation contrast from that sample was much weaker and automation could not be used in data acquisition or in aligning the images before reconstruction; the reconstruction was uninformative. Calculation from a number of line profiles on each image found the GaN tilt series had average contrast of 11% whereas the average contrast from the silicon tilt series was 26%.

There are many reasons for the contrast to be better in the silicon sample. The silicon sample was 270 nm thick, alongside the dislocation cascade, measured using the zero–loss peak log–ratio method. The GaN sample was 300 nm thick, from parallax measurements on threading dislocations. This is not a large difference in thickness and does not wholly explain
the difference in contrast. In addition, the two materials have different average atomic numbers. The variation with atomic number of intensity of elastic and inelastic scattering in ADF STEM images (from thermal diffuse scattering and other phenomena) depends on the collection angle; at low angles, total elastic scattering is proportional to $Z^{3/2}$ and total inelastic scattering is proportional to $Z^{1/2}$, but as the collection angle increases toward those used for HAADF, where scattering is incoherent, the total scattering approaches proportional to $Z^2$ as for Rutherford scattering [4]. At this intermediate collection angle the dependence on $Z$ is somewhere between these extremes [6]. The variation of the contrast with tilt angle is also interesting; intensity of defect and background follow similar patterns of variation with tilt angle, so there is interrelation between them, suggesting that even a simple rule of atomic number exponent is not sufficient to explain the contrast.

Channeling contrast may be present at some tilt angles and not others, affecting the background intensity [7] and the defect contrast, as channeled intensity is transferred out to dark field spots when meeting the defect [8]. The anisotropy of gallium nitride affects WBDF reconstructions by changing the relative position of the dislocation line from the core with tilt angle [9]; there is likely to be a similar effect, both within the ADF STEM image composed of many dark field images, and between tilt angles. Further analysis of ADF STEM contrast, to enable optimisation of contrast conditions, is key.

5. Conclusions

STEM ADF imaging of dislocations is a promising technique for use in electron tomography. In some cases they give contrast that is good enough for standard electron tomography software to correct for specimen drift during tilting, and this represents a significant step in making dislocation tomography into a technique suitable for use by the wider scientific community.

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6. References

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