Precession Electron Diffraction - Towards Structure Determination

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Although the best way to solve crystal structures is normally to use x-ray, or neutron, diffraction, for multi-phase samples, disordered structures or polymorphs, the small beam size offered by the electron microscope can be of great advantage. However, in most circumstances the strong interaction between the electron beam and the underlying crystal potential means that conventional electron diffraction patterns recorded at major zone axes are prone to strong dynamical effects. In this situation, diffracted intensities cannot be used directly to solve crystal structures using standard structure determination algorithms (e.g. direct methods or charge-flipping [1]).

Precession electron diffraction [2] is a method in which a focussed electron beam is rocked in a hollow cone both above and below the specimen. This has the effect of allowing 'zone axis' patterns to be recorded whose intensities are less prone to dynamical effects - this is because the beam is never exactly at the zone axis and the intensities are integrated through the Bragg condition. This method is directly analogous to x-ray precession and, in the same way, the precession mode ensures that many more beams are intercepted by the Ewald sphere than would be true in conventional electron diffraction.

In early precession work, the focus was to use reflections in higher order Laue zones as in most circumstances these intensities could be treated as quasi-kinematical. However, in recent years it has become evident that even zero layer reflections can be used for structure determination in some circumstances. We have attempted to understand when and why electron precession diffracted intensities can be used as input for structure determination algorithms. Figure 1 gives an example of the systematic changes in diffracted intensities as the precession angle is increased. The two curves, simulated by a full dynamical calculation, relate to a structure with the same diffracted structure factor moduli but different phases. For true kinematic diffraction the two curves should be the same – and this becomes the case as the precession angle increases. In the lower half of the figure a montage of electron diffraction patterns are shown recorded from the [001] zone axis of $Er_2Ge_2O_7$ patterns comparing a conventional CBED pattern with patterns recorded with precession angles of 20mrad and 45 mrad and a kinematic simulation. Qualitatively, it is apparent that the precessed patterns better match the kinematic simulation.

Figure 2 shows the projected potential (at 300kV) for the [001] zone axis of $Er_2Ge_2O_7$ and structure solutions, one using conventional direct methods [3], one using a charge-flipping algorithm. These results indicate that a faithful solution can be obtained from zero layer electron precession data if a sufficiently high precession angle is chosen and if a charge flipping algorithm is used that takes into account the phase relationships [4] determined by space group symmetry elements (the 2_1 screw axes in this case).

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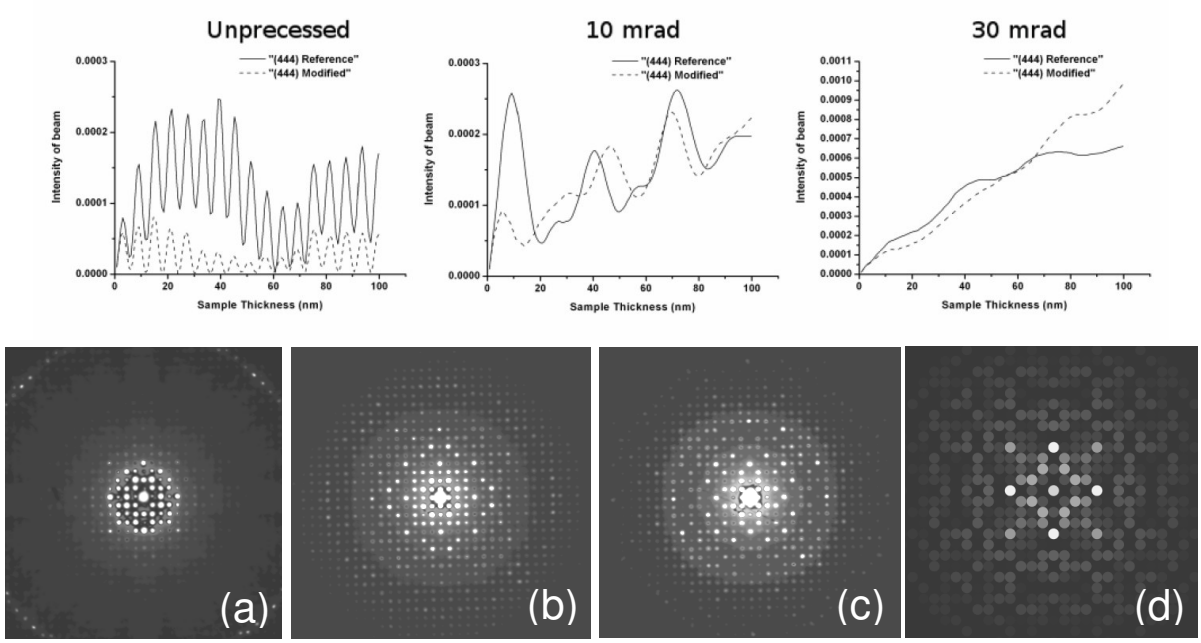

Figure 1. Top: Simulated beam intensity of the 444 beam recorded at the <110> zone axis of silicon for unprecessed and precessed beams of 10 and 30 mrad precession angle. The 'reference' curve is the true silicon structure, the 'modified' curve relates to a structure with the same structure factor moduli as silicon but with scrambled phases. Bottom: Montage of electron diffraction patterns at the [001] zone axis of $Er_2Ge_2O_7$ with (a) zero, (b) 20mrad and (c) 45mrad precession angle; (d) shows a simulated kinematic pattern.

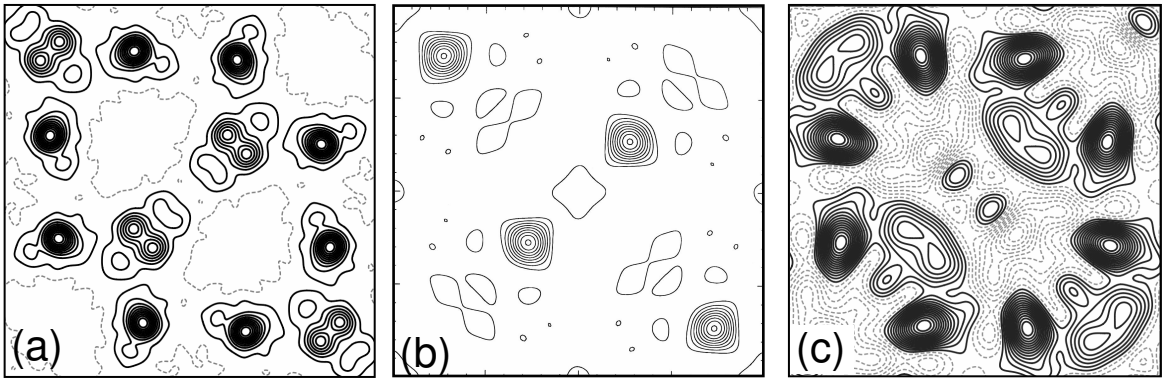

Figure 2. (a) Ideal projected potential for the [001] zone axis of $Er_2Ge_2O_7$, (b) the structure solution obtained by direct methods, (c) a structure solution using a modified charge-flipping algorithm incorporating 'phase symmetry'. Experimental ZOLZ precession data was used for (b) and (c).