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Nanocrystal Structure Determination by Kinematic Convergent-Beam Electron Diffraction

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The recent revival of inorganic electron crystallography [1] has been due to technological advances such as detectors that facilitate quantitative analysis, more powerful computers, the development of the electron precession technique [2], and tomographic specimen holders under computer control, along with the need to solve the structures of inorganic nanocrystals. We propose a new approach aimed at presenting a three-dimensional unit-cell potential map of a nanocrystal following collection of three-dimensional diffraction data. Our approach is based on the use of kinematic convergent-beam electron diffraction (KCBED) patterns, combined with the charge-flipping algorithm [3] for solution of the phase problem.

KCBED has the great advantage that data can be collected from the thinnest possible regions to minimize multiple-scattering artifacts and from sample regions that are entirely free from strain and defects. In addition, the method allows recording of absolute intensities since, by spreading out the diffraction disks, the intensity of the central diffraction disk is likely to lie within the dynamic range of the detector along with the other disks. Finally, the appearance of the disks can provide a rough guide to the importance of any multiple-scattering artifacts: for a sufficiently thin sample the CBED disks are filled uniformly without intensity oscillations (as in Figure 1). This test for single scattering is important, as it is one of the very few that does not depend on a prior knowledge of crystal structure. We use the charge-flipping method for phasing the data because of its proven ability to deal well with "poor quality" data (by comparison with X-ray data) for ab-initio phasing (rather than refinement), its speed and simplicity, and it does not require knowledge of the crystal space group [3,4].

We will show the challenges and opportunities associated with this technique and how it can be implemented to solve a wide variety of nanocrystalline structures. The potential for automating this process as a user-friendly tool to solve structures at the microscope make this a powerful tool for future crystallographic research [5].

References

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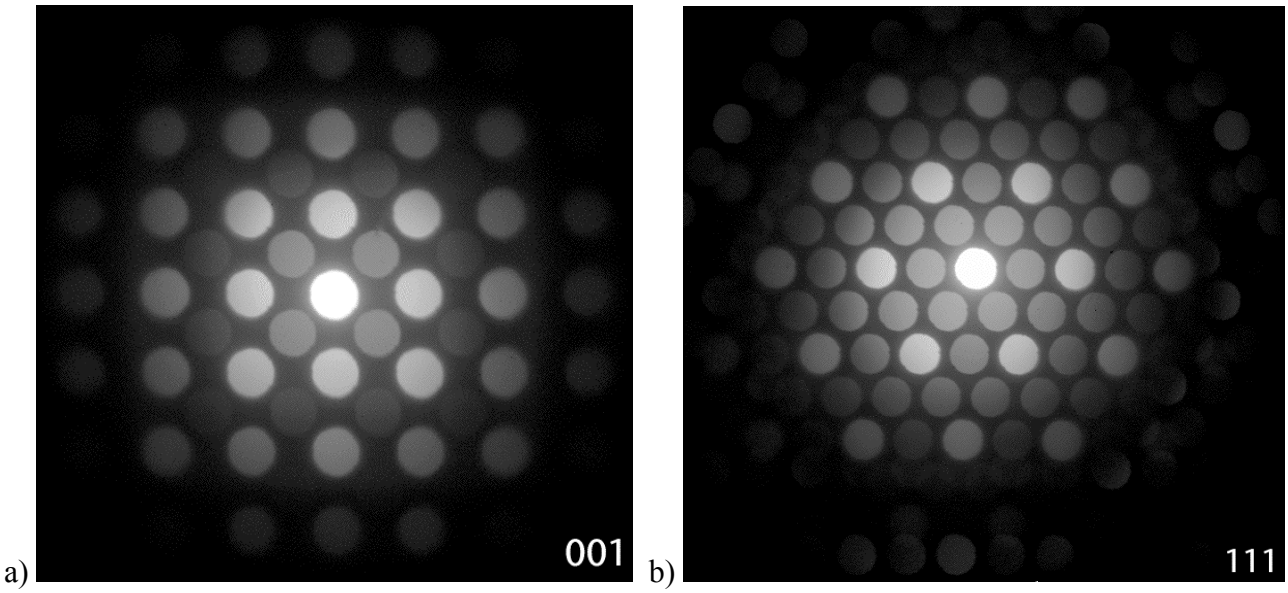

Figure 1: Experimental KCBED patterns from cubic spinel ($MgAl_2O_4$) in the a) 001 and b) 111 zone axes.

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