Electron nanocrystallography: Advancements toward automated structure solution

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The synthesis of new nanocrystalline structures demands new rapid methods of solving their crystal structures. Our goal is real-time structure solution at the electron microscope, based on automated acquisition of three-dimensional electron diffraction data with subsequent phasing of the data set and presentation of a unit-cell potential map that displays atomic positions and even species. To achieve this we must consider: 1) translation of the specimen during automated tilting; 2) automated recognition of zone-axis orientations; 3) multiple-scattering artifacts; 4) indexing methods; 5) absolute intensity scaling of the data; 6) scaling of data collected at different orientations; and 7) the phase problem. Initially, we have focused on issues 3) through 7) following manual acquisition of three-dimensional diffraction data from a known test crystal (the MgAl2O4 spinel structure). Data was collected by two techniques, both of which minimize multiple-scattering artifacts: precession electron diffraction (PED) and kinematic convergent beam electron diffraction (CBED) using an in-column Omega energy filter. After indexing and scaling, experimental structure-factor magnitudes were obtained from the patterns. These provide input to the charge-flipping algorithm [1], which works well with relatively poor-quality electron diffraction data or powder diffraction data [2], to solve the phase problem and obtain the correct crystal structure. Solutions for PED and kinematic CBED data are presented for comparison with each other and with simulations. Further development requires automated, scripted control of specimen tilt and data acquisition.

Keywords: electron diffraction, crystal structure analysis, electron microscopy and diffraction

A study of structure properties of ZnS nano-crystals using electron crystallography

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We have characterized the structure properties of two types of ZnS nano-crystals by electron crystallography. For determination of their initial structures, we have performed XRD analysis for ZnS crystals of 5 nm and 10 nm which were synthesized by same route. Their real crystallite sizes were about 5.9 nm and 8.1 nm and their crystal systems were hexagonal and cubic, respectively. Their quantitative structures, however, could not be determined because of their weak diffraction intensities. To overcome the intensity problem, the structure of ZnS nano-crystals was resolved by using EF-PED (Energy-Filtered Precession Electron Diffraction) and HREM (High Resolution Electron Microscopy) utilizing a HVEM (High

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