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Accurate determination of crystal orientation from rotation electron diffraction data

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Electron crystallography can overcome the limitation of X-ray crystallography on crystal size, which is a few microns for modern synchrotron sources, and allow the structures of crystals down to nanometer sizes to be studied. Rotation electron diffraction (RED) was thus developed with the aim of accurate determination of 3D atomic structures of crystals of sub-micrometer sizes [1]. RED has been shown to be powerful in phase identification and structure determination [2]. In RED data collection, electron beam tilt and goniometer tilt are combined to rotate the crystal in a large range (typically 100 – 140 degrees) with a small step (0.1 ~ 0.2 degrees) and electron diffraction (ED) frames are collected for each tilt. In RED data processing, 3D reciprocal lattice of the crystal is reconstructed from the 2D ED patterns. The unit cell parameters are determined, and after indexing the diffraction intensities are output for structure determination and refinement.
Accurate orientation of the crystal is important for diffraction intensity integration and structure refinement from 3D electron diffraction data using dynamical theories [3]. Although the orientation matrix is determined after reciprocal lattice is reconstructed in RED data processing, possible errors in the goniometer angles lead to inaccurate determination of the crystal orientation. In this work we developed a method for determining the accurate orientations of the crystal from RED data. As the crystal remains still during electron beam tilting, its orientation only needs to be determined for individual beam tilt series. By using a fast numerical localized random search method, the orientation of each frame within one tilt series is obtained by least squares minimization of the distances from the reflections to the Ewald sphere, with the indices of the observed reflections in the frames and the unit cell dimensions as input. The accuracy of the fitted orientations was up to 0.01° versus true orientations as shown using simulated tilt series diffraction data with a tilt step of 0.1 degree. The algorithm was tested on an experimental garnet dataset and large errors in the goniometer angle readouts were identified according to the fitted results. The least squares objective function was also analyzed and discussed in connection to the accuracy of the fitting results. The orientation of the tilt axis and the unit cell dimensions can be refined by including them in the least squares objective function. The algorithm was proven to be effective for diffraction tilt series with relatively fine (~0.2°) tilt steps and good number of diffraction spots (> 25) on the frames.
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