

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 [1-2]. RED has shown to be powerful in phase identification and structure determination. More than 70 structures have been solved from the RED data [3]. 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.

In this work we aim at improving the processing of RED data, in particular the unit cell determination. Since the orientation matrix and unit cell determination are based on the positions of diffraction peaks, accurate determination of peak positions is very important for unit cell determination. In RED, the diffraction peaks are identified and the positions are determined according to the maximum intensities of the spots in 2D and 3D. Dynamic effects due to multiple scattering of electrons are known to distort electron diffraction data and they may change the distribution of diffraction peaks, i.e., peak shapes and profiles. Accuracy of peak positions determined using the maximum intensity may be affected accordingly. Therefore we introduced an alternative method for determining the peak positions using intensity weighted coordinates, i.e. the peak coordinates are calculated as the average of the coordinates of all the pixels of the reflection with intensities above a certain threshold, weighted using the intensities. By using the new peak positions for a RED data set of garnet it was shown that the accuracy of unit cell determination was improved.

References


Keywords: Rotation Electron Diffraction, Peak Searching, Unit Cell Determination

Electron crystallography, Zeolite, Electron microscopy, Cryo-EM, Method development