MS28-P2 Improved Accuracy of Unit Cell Determination for Rotation Electron Diffraction Data

Bin Wang[1], Sven Hovmöller[1], Xiaodong Zou[1], Wei Wan[1]

1. Department of Materials and Environmental Chemistry, Stockholm University, Stockholm 106 91, SWEDEN

email: bin.wang@mmk.su.se

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

[1] D.L. Zhang, S. Hovmöller, P. Oleynikov, X. Zou, Z. Kristallogr, 2010, 225, 94-102.

[2] W. Wan, J.L. Sun, J. Su, S. Hovmöller, X. Zou. J. Appl. Cryst., 2013, 46, 1863-1873.

[3] Y.F. Yun, X. Zou, S. Hovmöller, W. Wan, IUCrJ, 2015, 2, 267–282.

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

MS28-P3 Structural characterisation of complex zeolite structures using electron crystallography

Hongyi Xu[1], Jiho Shin[1,2], Peng Guo[1], Xiaodong Zou[1]

1. Inorganic and Structural Chemistry and Berzelii Center EXSELENT on Porous Materials, Department of Materials and Environmental Chemistry, Stockholm University, SE-106 91 Stockholm, Sweden
2. Center for Ordered Nanoporous Materials Synthesis, School of Environmental Science and Engineering, POSTECH, Pohang 790-784, Korea

email: hongyi.xu@mmk.su.se

The structures of two new zeolites, PST-26 and PST-28 with super-complex structures were predicted, synthesized and confirmed.[1] The prediction of these two structures was achieved by the strong reflections approach[2] and model building, which provided essential information for targeted syntheses of these materials.[3] Due to small energy differences, PST-26 and PST-28 could only be obtained as mixtures, together with a small amount of gismondine which makes structure confirmation by powder X-ray diffraction more difficult. By taking the advantage of electron crystallography, individual particles of PST-26 and PST-28 could be characterized in a transmission electron microscope (TEM) in order to verify their structures. Due to the large unit cell and the structure complexity of the materials, we cooled down the sample to liquid nitrogen temperature (-176°C) during TEM studies to (1) prevent the crystals from being distorted/damaged by the high vacuum inside the TEM column and (2) reduce electron beam damage. For both materials, electron diffraction patterns were collected along both [111] and [001] zone axes and compared against simulated electron diffraction patterns based on predicted models. By analysing the strong reflection distributions and the weak diffraction spots between the experimental and simulated electron diffraction patterns, the structures of as-synthesized PST-26 (*Im*-3*m*, a = 75 Å) and PST-28 (*Im*-3*m*, a = 85 Å) were successfully verified.[1] This work not only demonstrated the power of electron crystallography, but also shows the possibility to combine cryo-EM and electron diffraction techniques to study electron beam sensitive materials with complex structures such as micro-sized protein crystals.

Reference:

1. Shin, J. and Xu, H. *et al.* Targeted Synthesis of Two Super-Complex Zeolites with Embedded Isoreticular Structures. *Angewandte Chemie International Edition* (2016).

2. Zhang, H. *et al.* Structure relations in real and reciprocal space of hexagonal phases related to i-ZnMgRE quasicrystals. *Philos. Mag.* **86**, 543-548, doi:10.1080/14786430500279658 (2006).

3. Guo, P. *et al.* A zeolite family with expanding structural complexity and embedded isoreticular structures. *Nature* **524**, 74-U143, doi:10.1038/nature14575 (2015).

Keywords: Electron crystallography, Zeolite, Electorn microscopy, Cryo-EM, Method development