MS27-P4 Optimization of rotation electron diffraction data collection and data processing

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Electron crystallography is a very powerful technique for structural analysis of nano- and micron-sized crystals [1]. We have developed rotation electron diffraction (RED) for automated collection and processing of 3D ED data [2]. More than 1000 ED frames can be collected from an arbitrarily oriented crystal in less than an hour by RED (Fig. 1). The unit cell, possible space groups and ED intensities can be obtained. The 3D ED methods have shown to be very powerful and efficient for phase identification and structure determination [3,4]. Now an unknown structure can be solved in less than 8 h, from the data collection to structure solution. Although it is possible to solve the structures from the RED data, the R-values are still very high (20-50%). The ED intensities are affected by absorption and multiple scattering. Radiation damage is also a limiting factor [5], which may lead to low resolution and incomplete data, as well as inaccurate intensities.

Here we show how different data collection parameters of the RED method can affect the determination of unit cell, space group, and the accuracy of the atomic positions. The calcined zeolite silicalite-1, which is a pure silica form of the MFI framework family, was chosen as an example to optimize the parameters of RED method on structure analysis of zeolites [6].

Accurate unit cell parameters could be obtained with a large tilt range. For normal unit cell determination, a large tilt step can be applied. On the other hand, for structure determination a small tilt step is preferred, as the intensities would be more accurate due to the fine sampling. The tilt step can affect intensities of the reflections, which cause inaccurate atomic positions and high R-values in the refinement. The resolution of RED data can also affect the refinement result and atomic positions. We will present how to optimize the data processing to obtain better ED intensities.

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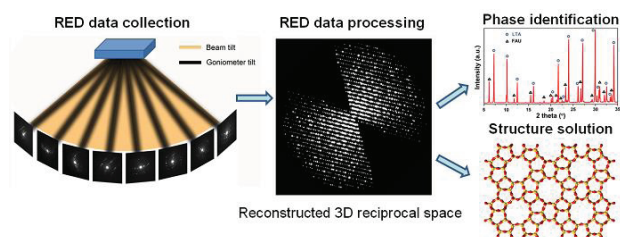[6] C. Nave, *Radiat. Phys. Chem.* 1995, 45, 483.

Figure 1. Illustration of a complete structure solution from Rotation Electron Diffraction (RED) data.