

Low Dose Structural Analysis of Fragile Materials by Three-Dimensional Electron Diffraction

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Crystalline solids, such as metal, metal oxides, zeolites, and metal-organic frameworks (MOFs), etc., have been intensively investigated both as traditional and state-of-the-art materials. They have been implemented in a wide range of applications from industrial use to laboratorial research. The applications are closely associated with the physical and chemical properties, which are determined by the atomic crystal structures. Therefore, a crucial part in the study of crystalline materials is to determine the structures. The novel properties of a material can be revealed by knowing the structure, and then specific applications can be designed.

High-resolution transmission electron microscopy (HRTEM) is one of the most widely used tool for obtaining structural details at an atomic level. However, it is challenging to perform structural analysis of fragile materials such as zeolites, and metal-organic frameworks (MOFs), which are sensitive to radiation damage from electron beam. Combined with cryo-TEM, the development of direct-detection cameras provides an effective solution to this challenge. Nevertheless, it still requires atoms to be separated at the viewing projection, and a large sample thickness could also hamper the observation.

These challenges have been met by the rapid development of three-dimensional electron diffraction (3DED). Using the intensities of diffraction spots as the basis, the structures of nano-sized crystals can be determined at the atomic resolution. Nowadays, a complete continuous rotation electron diffraction (cRED) data can be acquired in 15-150 seconds using continuous goniometer tilt, and the electron dose rate can be limited to below $0.01 \text{ e } \text{Å}^{-2} \text{ s}^{-1}$. Thus, one major problem in electron microscopy, the beam radiation damage, could be minimized. This is crucial for the study of beam sensitive materials. As a consequence, more structural details, such as guest molecules, hydrogen bonding in the crystals can be revealed. In addition, the fast data collection and large number of data makes it possible for quantitative phase analysis, and for detection of minor phases that may not be detectable by other characterization techniques. I will first present low dose 3DED on investigating heteroatom distribution in a photoactive MOF¹. Further example will be given by applying 3DED on the study of the electrocatalyst, where the spacing between active-sites in the MOF are found crucial for its activity². Last, I will demonstrate 3DED as a high-throughput approach that can accelerate the discovery of new electrocatalysts.

References

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