Atomic-resolution Imaging of Sensitive Materials Using Ultralow-dose Transmission Electron Microscopy

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High-resolution imaging of electron beam-sensitive crystalline materials, such as zeolites and metal-organic frameworks, is one of the most difficult applications of transmission electron microscopy (TEM). The challenges are manifold, including the acquisition of images with an extremely low beam dose, the time-constrained search for crystal zone axes, the precise alignment of successive images, and the accurate determination of the defocus value.

We reported that using a direct-detection electron-counting camera, it is possible to acquire useful high-resolution TEM images with electron dose as low as a few electrons per square angstrom to ensure that the intact structure was captured before damage occurred [1]. Later, we reported a suite of new methods that we recently developed to address the rest challenges mentioned above [2]. Our methods advance the HRTEM of extremely beam-sensitive materials from “occasionally possible” to “routine”. We demonstrate the effectiveness of our methodology by capturing atomic-resolution TEM images of several metal organic frameworks (MOFs) that are generally recognized as highly sensitive to electron beams. In the case of MOF UiO-66, individual metal atomic columns, various types of surface termination, and benzene rings in the organic linkers, are clearly identified. We also successfully apply our methods to other electron beam-sensitive materials, and achieve atomic-resolution TEM imaging of the organic-inorganic hybrid perovskite $CH_3NH_3PbBr_3$ for the first time [2]. More recently, we applied this new technology to prove the successful encapsulation of single molecule magnets in MOF NU-1000 [3], and to investigate the evolution and transformation of various defects in MOF UiO-66 [4], and to probe the subtle differences in the surface structure between various MOF MIL-101 samples [5].

In this presentation, I will also discuss iDPC-STEM as a powerful tool to probe guest molecules in sensitive porous material matrix, taking atomically dispersed Mo in zeolite ZSM-5 as an example [6]. Finally, I will introduce a new TEM specimen preparation technique, cryo-FIB, which is particularly useful for beam-sensitive materials [7].

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