An Investigation on 3D Electron Diffraction and 4-Dimensional Scanning Diffraction Tomography Using a Scanning Electron Microscope

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During the last decade, 3D-Electron Diffraction (3D-ED) has emerged as a powerful technique for studying the structure of sub-micron particles. It is used in a variety of applications, from unit cell and space group determination to the complete solution of the structure similar to X-ray diffraction techniques. So far, this technique has been only implemented in Transmission Electron Microscopes (TEMs). Performing such an experiment in a Scanning Electron Microscope (SEM) can be challenging, mainly due to the lower acceleration voltage. This lower beam energy raises concerns about severe multiple scattering and limited transmissivity of the sample for the weakly accelerated electrons.

In this work, we show the possibilities of operating similar studies in a modified SEM. For this aim, we equipped our SEM with a custom stage and holder, a direct electron detector and a custom high-angle annular dark-field detector (HAADF). A range of samples was studied in the form of lamellas and sub-micron particles, and the quality of the diffraction data was evaluated for different purposes, such as unit cell determination and space group determination. Moreover, the ability to integrate the diffraction data for structure solution and refinement has been assessed and compared to similar data acquired in a TEM.

Finally, we also demonstrate the potential for combining diffraction tomography and 4-dimensional scanning transmission electron microscopy (4D-STEM) in our setup. This method opens an avenue to obtain multiple 3DED datasets out of 5D-STEM data. These 3DED datasets can be created using object tracking methods from several regions of a multi-domain particle or from multiple single crystals within the scanning region. This provides an attractive route to high-throughput and statistically relevant characterization of polycrystalline materials or powders of nanoparticles [1].

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