**Oral presentation**

# 4D-STEM diffraction tomography for solving the structure of nanoparticles

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*3D-electron diffraction* (*3DED*) is a powerful technique for solving the structure of nanoparticles using Transmission electron microscopes (TEM). Typically, 3DED is performed in TEM mode on particles with the size of several hundreds of nanometers. However, as the size of the particle or the region of interest decreases further, some complications appear in acquiring ideal 3DED data. First, the particles might have minimal contrast in TEM mode making it difficult to see and track them. Second, to have a good signal-to-noise ratio, the electron dose should increase which can cause beam damage. Third, the illuminated area should usually be decreased to avoid the illumination of the surrounding particles and to minimize the background signal from the carbon support of the TEM grid. However, there are limitations to how small the illuminated area can be made in TEM mode by decreasing the size of the selected area or condenser apertures. Additionally, a robust and accurate particle tracking method becomes increasingly vital as the size of the beam decreases. Finally, in many cases, the particles might agglomerate or overlap making it even more challenging to acquire the data.

To overcome these complications, we hereby propose an alternative method based *on 4D-Scanning Transmission Electron Microscopy* (*4D-STEM*) tomography in microprobe mode. In this method, the high-angle annular dark-field (HAADF) detector is initially used for finding a region of interest. Later, a probe of typically 1 to 5 nm scans the whole field of view at each desired angle, resulting in a 5-dimensional dataset. Then, 3DED datasets can be extracted from this *5D-electron diffraction* (*5DED*) dataset using object-tracking algorithms on the navigation images. These navigation images can be either the HAADF images collected during data acquisition or the virtual bright or dark field images created from 4D datasets after the data acquisition. Due to the fast scans using direct electron detectors, the beam damage is also minimized. Moreover, in the case of agglomerated samples, the spatial resolution of the technique allows the extraction of 3DED datasets from small, single-crystalline regions, where several 3DED datasets can be extracted from 1 5DED dataset.

For easier, faster, and more accurate experiments, we synchronized the direct electron detector, scanning engine and the TEM, and automated the whole data acquisition process. This allowed the acquisition of several hundred scans with smaller angular steps for the tomography experiment. This, consequently, improves the integration of the intensities in 3DED. Alternatively, we also investigated the combination of this technique with precession-assisted electron diffraction to decrease the required scanning steps and suppress multiple diffraction effects. To demonstrate different applications of this technique, several challenging samples were investigated and their structures were successfully solved, including well-dispersed, beam-sensitive, 20-nm $CsPbBr_3$ and agglomerated, 50-nm $BaTiO_3$ and 30-nm $LiNiMnCoO_2$ (NMC) nanoparticles.

*The authors acknowledge the financial support of the Research Foundation Flanders (FWO, Belgium) project SBO S000121N and SBO 1SHA024N.*