MS24-1-4 3D ED on isolated nanoparticles: exploring the size limit of crystals
#MS24-1-4

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Abstract
3D ED [1] brings together different approaches to collect diffraction data on single crystals of much smaller size than those usable for single crystal X-ray diffraction. But what is the minimum crystal size at which it is possible to collect 3D ED data suitable for structure solution and refinement?
In order to address this issue, it is necessary to explore some instrumental constraints. Having a transmission electron microscope (JEOL F200) with a condenser system capable to preserve parallel beam illumination condition for a wide range of beam size and brightness is definitely a plus. Smaller particles mean weaker diffraction signals and the use of a hybrid-pixel detector (M3 Cheetah ASI) is certainly interesting here. Having a goniometer with low movement upon tilting is also nice.
In this contribution, we will show our first results obtained on TiO2 brookite nanorods (about 20 nm x 80 nm in size) where it has been possible to collect PEDT (Precession Electron Diffraction Tomography) data and successfully solve the structure. Taking both precession and dynamical diffraction effects into account [1], the structural model was then refined with very good Robs values (about 8%). From our experimental setup, the accurate structure of nanoparticles above 20 nm is obtained quite straightforward. However, the situation becomes more difficult as the particle size decreases as illustrated here (Fig. 1) in the case of 10 nm ITO nanoparticles. Particle tracking, beam sensitivity and weak diffraction signal are the issues I would like to present and discuss with you.
These results are obtained within the framework of the European project NanED (Electron Nanocrystallography – H2020-MSCA-ITN GA956099).

References
[1] L. Palatinus et al., “Structure refinement using precession electron diffraction tomography and dynamical diffraction: tests on experimental data”, Acta Cryst. B71 (2015) 740–751.

Figure 1