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Fast electron low dose tomography for beam sensitive materials

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Nanomaterials are important for a wide range of applications because of their unique properties, which are strongly connected to their three-dimensional (3D) structure. Electron tomography has therefore been used in an increasing number of studies. Even atomic resolution electron tomography and *in situ* investigations in 3D have become possible. However, the majority of these studies were performed for samples that are relatively stable under the electron beam since the 3D characterisation process needs a larger amount of electrons compared to 2-dimensional (2D) imaging. Therefore, the investigation of nanomaterials that are more sensitive to the electron beam, requires the development of more advanced 3D characterization techniques.

In a conventional electron tomography experiment, pre-acquisition steps, such as focusing and tracking at every tilt angle needs additional acquisition time and electron dose. Recently, we proposed an acquisition approach where a tilt series of 2D high angle annular dark field scanning transmission electron microscopy (HAADF-STEM) projection images is acquired by continuously tilting the holder and simultaneously acquiring projection images while focusing and tracking the particle [1,2]. For HAADF-STEM, this approach results in an electron dose reduction of almost an order of magnitude and an acquisition time of a few minutes instead of an hour or longer. Furthermore, an incremental methodology was proposed for combining the benefits of the conventional and continuous techniques [3].

Fast HAADF-STEM tomography was successfully used to investigate structural and compositional changes in metal nanoparticles [4,5]. The question remains if samples that are more beam sensitive such as Zeolites and Metal Organic Frameworks (MOFs) will withstand the electron dose required for fast HAADF-STEM tilt series with the new acquisition methods. Hereby, the combination of fast electron tomography with novel low-dose imaging techniques, such as integrated differential phase contrast (iDPC) imaging in STEM mode can be considered as a next step towards 3D imaging of beam sensitive materials. For nonmagnetic and thin samples, iDPC-STEM yields images that can be directly connected to the projected electrostatic potential of the atoms in the sample [6]. The iDPC-STEM intensity is expected to scale linearly with the sample thickness and therefore fulfills the projection requirement for electron tomography.

In this contribution, the different acquisition strategies will be experimentally compared in terms of speed, resolution and electron dose, based on experimental tilt series acquired for SBA-16 and MCM-41 particles. Moreover, a quantitative comparison will be made between HAADF-STEM tilt series and iDPC-STEM tilt series.

Figure 1 illustrates a comparison between 3D reconstructions for an SBA-16 particle, based on HAADF-STEM tilt series acquired using fast incremental tomography and electron doses/pixel of 16.96 $e^-/Å^2$ (a), 5.65 $e^-/Å^2$ (b) and 1.70 $e^-/Å^2$ (c). From the orthoslices through the 3D reconstructions, it can be seen that the morphology of the particle can still be characterized for all electron doses, but the pore structure becomes difficult to interpret for Figure 1.c. Figure 2 presents a comparison between a 3D reconstruction based on HAADF-STEM and an iDPC-STEM reconstruction. Hereby, the series are simultaneously acquired using conventional electron tomography with an electron dose of 3.17 $e^-/Å^2$. By comparing the orthoslices through the 3D reconstructions, it is clear that the signal-to-noise ratio for the iDPC-STEM reconstruction is superior in comparison to the

HAADF-STEM reconstruction. We therefore expect that iDPC-STEM in combination with fast electron tomography will become an important technique for the 3D characterization of beam sensitive materials. Further progress can be obtained by optimization of the 3D reconstruction algorithms for these low dose tilt series.

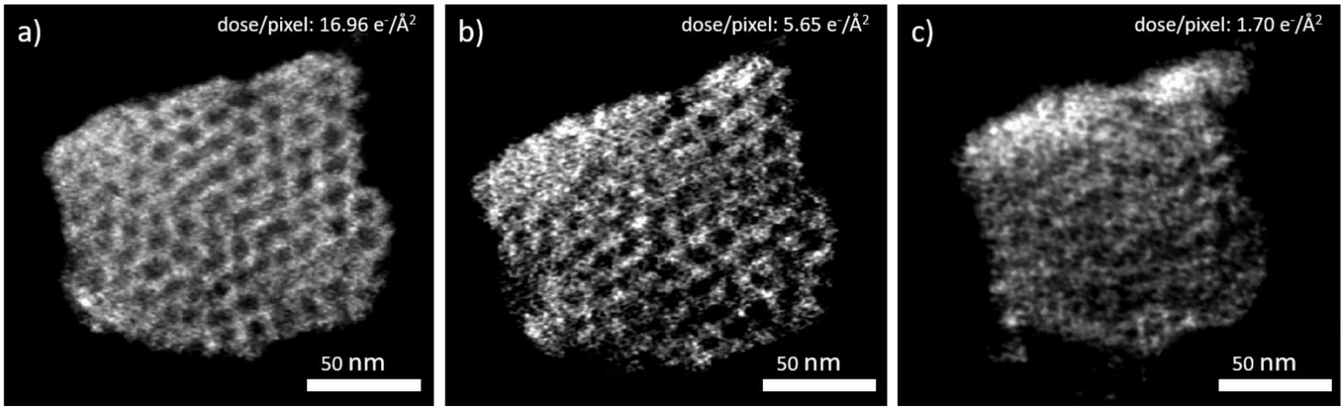

Figure 1. Orthoslices through 3D reconstructions of a SBA-16 particle. The reconstructions are based on fast incremental HAADF-STEM tomography with decreasing electron doses. Electron doses/pixel of 16.96 e-/Å2 (a), 5.65 e-/Å2 (b) and 1.70 e-/Å2 (c).

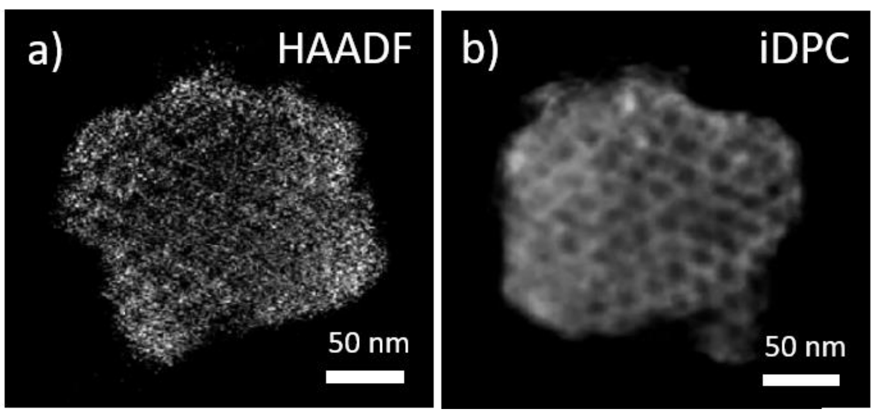

Figure 2. Orthoslices through 3D reconstructions of a SBA-16 particle. The reconstructions are based on simultaneously acquired HAADF-STEM (a) and iDPC-STEM (b) tilt series. (total dose per pixel during the acquisition: 1200 e/Å2).

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