Analytical 4D STEM with the pnCCD camera

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The availability of the pnCCD (S)TEM camera, combining ultra-fast direct detection at single electron sensitivity with a radiation hard diode like technology, inspired the development of various four-dimensional STEM imaging techniques (4D STEM). Here, a focused beam of electrons is applied to a sample in a two-dimensional (2D) raster pattern. At each point, a 2D image is captured which intrinsically contains bright field (BF), dark field (DF), and high angle annular dark field (HAADF) signals. In total, a 4D dataset is recorded allowing a comprehensive analysis and enabling a number of techniques such as strain analysis, magnetic domain mapping, scanning electron diffraction, TEM tomography, and electron ptychography. The key parameters of the pnCCD (S)TEM camera, which are particularly beneficial for 4D STEM techniques are a fast acquisition of data, a large number of detector pixels and a sufficiently radiation hard detector.

With a spatial resolution of 264x264 pixels, the pnCCD (S)TEM camera provides ultra-fast acquisition of 2D camera images [1]. Routinely, the readout speed is 1000 frames per second (fps) in full frame mode and can be further increased up to 8000 fps by binning and windowing. The pnCCD (S)TEM camera can be operated with electron energies ranging from 20 keV to 300 keV. By operating the camera in one of three predefined operation modes [2], optimum results are guaranteed for a wide range of experimental conditions.

The pnCCD (S)TEM camera is a complete system including a data acquisition computer and software allowing a versatile live visualization and full raw data access. The camera head is available as fixed bottom mount or retractable design. The outstanding capabilities of the pnCCD (S)TEM camera were demonstrated in numerous experiments at various TEMs.

As first example, a platinum wedge was imaged on a JEOL-ARM 200F operated at 200 kV using 1000 fps full frame readout speed. The microscope was adjusted so that the BF disk has a diameter of about 40 pixels, allowing efficient phase reconstruction by ptychography. At the same time the DF is recorded up to 65 mrad on the camera. This allows to select specific areas of the detected signal to create synthetic ADF images, as shown in Figure 1.

The second example shows a battery material; a charged Li1.2Mn0.6Ni0.2O2 particle, a Li-rich layered oxide cathode, which has improved capacity and avoids expensive Co, compared to current cathode materials. Both, their advantages and the processes that affect their utilization are inherently local. Measurements of charged samples require special care as phase transformations may be induced by the electron beam. With the pnCCD (S)TEM camera it is possible to obtain high contrast images at a very low dose using sub pA beam currents, enabling imaging of low Z materials in a low dose setting. Figure 2 shows an example of a Li-rich layered oxide cathode, where the phase contrast method visualizes the atomic arrangement.

References:

[1] Ryll, H., et al, Journal of Instrumentation 11 (2016)

[2] Schmidt J. et al, Journal of Instrumentation 11 (2016)

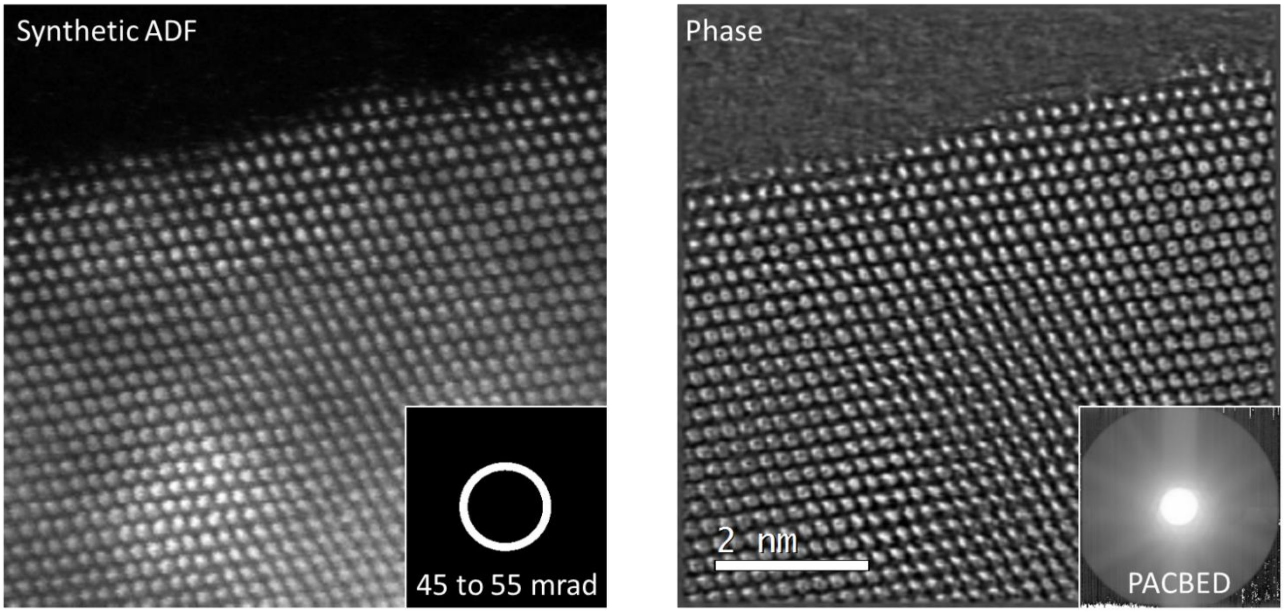

Figure 1. Platinum wedge recorded with 1000 fps on a JEOL ARM200F at 200 kV. Convergence angle was 22.4 mrad and the magnification 30M. Comparison of synthetic ADF image (left) and the reconstructed phase (right). The inset on the bottom right shows the Kikuchi lines in the recorded PACBED. Data recorded and analyzed by University of Oxford, Department of Materials.

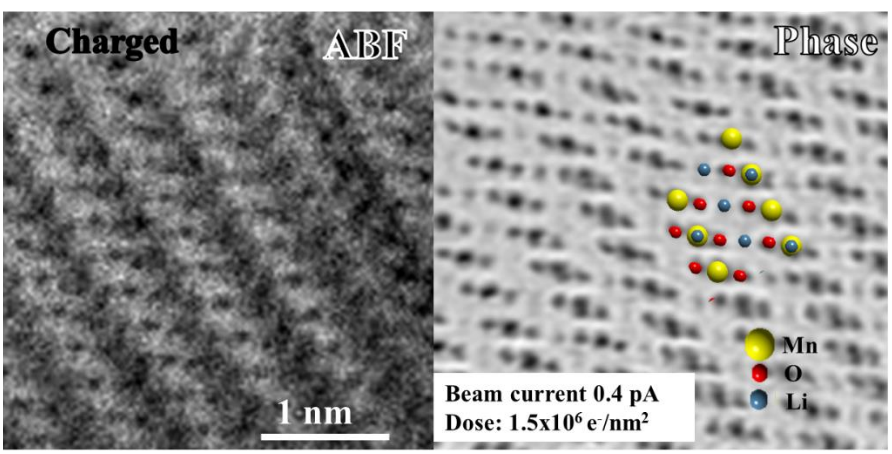

Figure 2. Sample of charged LiMnO recorded with 4000 fps on a JEOL ARM200F at 200kV. Although visible in the ABF image, the phase contrast reveals the positions of Mn, O and Li. The schematic overlay shows the structure and elemental arrangement. Data recorded and analyzed by University of Oxford, Department of Materials.