

## Pushing the Limits of Fast Acquisition in TEM Tomography and 4D-STEM

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In both TEM and STEM many experiments profit from recording two-dimensional camera images at very high readout speeds. This is especially true for tomography in TEM and ptychography in STEM. The pnCCD (S)TEM camera is routinely running at 1 000 frames per second (fps) in full frame mode [1]. This camera uses a direct detecting, radiation hard pnCCD with 264x264 pixels and features binning and windowing modes, which allow to further increase the frame rate substantially. For example, 4-fold binning in one direction, i.e. 66x264 pixels, yields a readout speed of 4000 fps. In windowing modes, up to 20 000 fps are possible. Benefitting applications range from imaging on the micro- and millisecond timescale to strain analysis or electric field mapping.

Tilt series of fewer than 100 images for tomographic reconstructions typically take 15 to 60 minutes with conventional cameras running at speeds below 40 fps. The series are recorded by stepwise rotation of the goniometer and taking a camera image after each rotation step. These long acquisition times restrict the acquisition of tomographic series for beam sensitive samples. With the pnCCD camera we have recorded a tilt series containing 3 487 images of an inorganic nanotube in only 3.5 s [2]. Due to the high readout speed it was possible to rotate the goniometer continuously over a tilt range of  $-70^\circ$  to  $+30^\circ$  in an FEI Titan 60-300 TEM, operated at 60 keV beam energy. The short acquisition time and the high sensitivity of the camera allowed to reduce the cumulative electron dose to about 8 electrons per  $\text{\AA}^2$ , which is about an order of magnitude lower than conventionally used for low dose tomography. A 3D reconstruction of the nanowire is shown in Figure 1. The acquisition time was limited not by the readout of the camera, but rather by the rotation speed of the goniometer.

Combining the high readout speed with the scanning mode makes 4D-STEM imaging feasible, a powerful imaging technique where a two-dimensional image is recorded for each probe position of a two-dimensional STEM diffraction pattern. With the pnCCD (S)TEM camera, a 4D data cube consisting of 256x256 (i.e. 65 536) probe positions with a 132x264 pixel detector image (using 2-fold binning) for each probe position can be recorded in about 35 s. Several measurements have been performed to prove the capability of the camera for 4D-STEM imaging, including strain analysis, magnetic domain mapping and electron ptychography. The latter is a 4D-STEM technique that has been described theoretically already in 1993 [3] but was so far limited experimentally by the low readout speed of existing cameras. In electron ptychography, the intensity distribution in the bright field disk is recorded in 2D for each STEM probe position. In an electron wave-optical approach the phase and amplitude information is extracted from the recorded intensity images. The reconstructed phase image (Figure 2a) shows enhanced image contrast compared to the simultaneously acquired conventional annular dark field image (Figure 2b). Measurements with the pnCCD (S)TEM camera were carried out using a

JEOL ARM200-CF in order to investigate different samples with the ptychographic phase reconstruction technique.

In conclusion, the pnCCD camera enables new techniques in TEM and STEM. Various fields of application benefit from recording two-dimensional detector images at high speeds. With its direct detection, high readout speed and radiation hardness the pnCCD (S)TEM camera permits the recording of tomographic tilt series and large 4D-STEM data cubes in short times and thus paves the way for new science.

[1] H Ryll *et al*, *Microscopy and Microanalysis* **19** (2013), p.1160-1161.

[2] V Migunov *et al*, *Scientific Reports* **5** (2015), 14516.

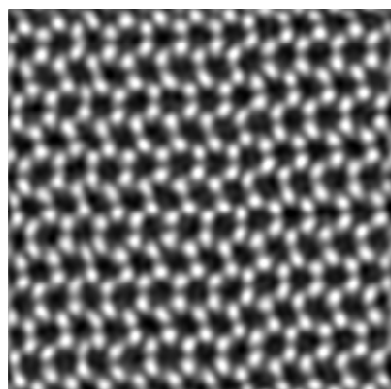
[3] JM Rodenburg, BC McCallum and PD Nellist, *Ultramicroscopy* **48** (1993), p.304–314.

[4] KJ Batenburg and J Sijbers, *IEEE Trans. Image Process.* **20** (2011), p.2542–2553.

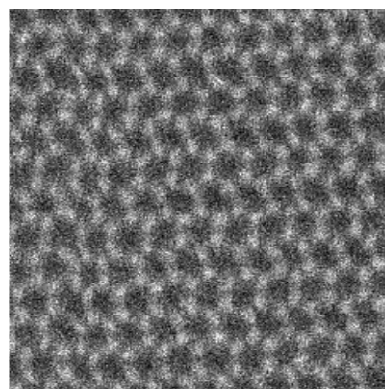
[5] The authors acknowledge Xiaodong Zhuge, K. Joost Batenburg and Lothar Houben for their contributions to the tomography measurement.



**Figure 1:** A three-dimensional DART [4] tomographic reconstruction rendering of an inorganic nanotube (orange) and the underlying amorphous carbon support (blue). The tilt series containing 3 487 images was recorded in only 3.5 s, limited by the rotation speed of the goniometer. Picture taken from [2].



a) Ptychographic image



b) Simultaneously obtained ADF image

**Figure 2:** Comparison of the reconstructed phase and the conventional annular dark field (ADF) image of a graphene sample using a ptychography method. Images contain 256x256 probe positions recorded at a dwell time of 500  $\mu$ s, corresponding to a total acquisition time of 35 s.