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

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Both transmission electron microscopy (TEM) and scanning transmission electron microscopy (STEM) experiments profit from recording two-dimensional camera images at very high readout speeds. This includes, but is not limited to, tomography in TEM and ptychography in STEM. The pnCCD (S)TEM camera uses a direct detecting, radiation hard pnCCD with a minimum readout speed of 1 000 full frames per second (fps) with a size of 264x264 pixels [1]. It 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 4 000 fps. Up to 20 000 fps are possible in windowing modes. Further applications that benefit from the high readout speed range from imaging on the micro- and millisecond timescale to strain analysis or electric and magnetic field mapping.

Typical tomographic reconstructions use tilt series of fewer than 100 images which are recorded in 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. We have recorded a tilt series containing 3 487 images of an inorganic nanotube in only 3.5 s with the pnCCD camera [2]. Due to the high readout speed it was possible to rotate the goniometer continuously over a tilt range of -70 ° to +30 ° in an FEI Titan 60-300, 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 Å^2, i.e. 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 not limited 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 was 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 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.

References:

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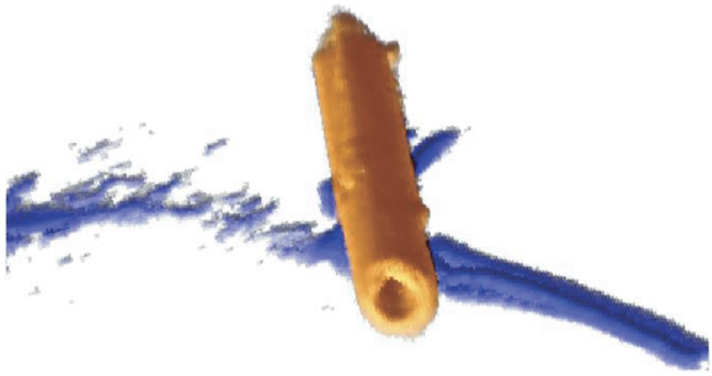

Figure 1: A three-dimensional tomographic reconstruction rendering of an inorganic nanotube (orange) and the underlying amorphous carbon support (blue). The tilt series containing 3 487 images was recorded in only 3.5 s, limited by the rotation speed of the goniometer. Reconstruction was generated with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

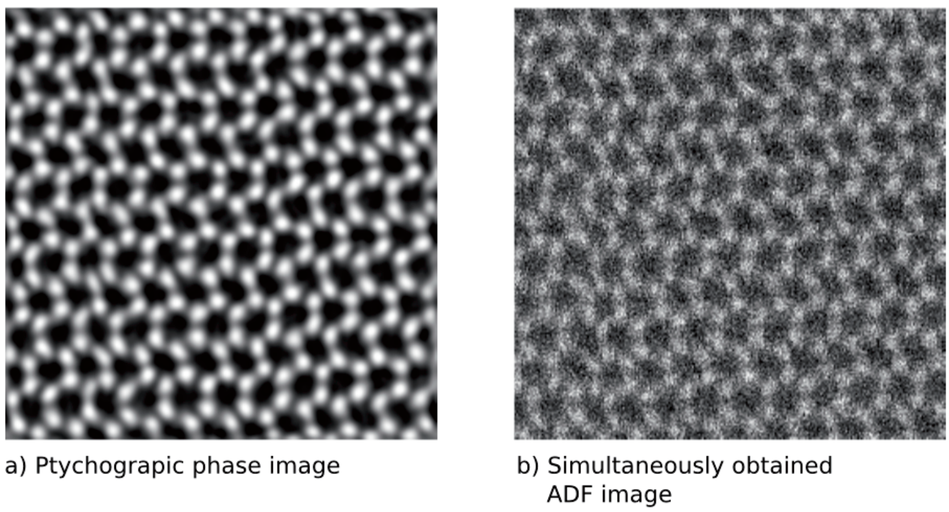

Figure 2: Comparison of the reconstructed phase and the conventional annular dark field (ADF) image of a graphene sample using a ptychography method. Distortions in the images are due to scan artefacts. Images contain 256x256 probe positions recorded at a dwell time of 500 µs, corresponding to a total acquisition time of 35 s.