WORKSHOP

FIVE YEARS OF EXPERIENCE WITH 4D (S)TEM IMAGING USING THE PIXELATED pnCCD (S)TEM CAMERA

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(S)TEM imaging in TEM is an interesting and challenging field in material analysis which became more and more popular within the last years as it allows atomic resolution images, as well as diffraction pattern analysis, ptychography, tomography, electrical and magnetic field mapping, strain analysis as well as spatially resolved elemental analysis by use of highly integrated large solid angle energy dispersive detectors (SDD). So far mainly single or minor segmented (S)TEM detectors have been used for the detection and quantification of the deflected electron beam current within Bright Field (BF), Angular Dark Field (ADF) or HAADF (High Angular Dark Field).

For more precise and advanced measurements a comprehensively pixelated (S)TEM detector is needed. However, this detector should not lose the essential properties of standard BF/ADF/HAADF measurement capabilities i. e. from a detector's point of view

- radiation hardness (beam currents can go up to 1 nA easily)
- speed (it is the target to record a full (S)TEM image with 256 x 256 (S)TEM pixels in less than a minute i.e. 50.000 up to 100.000 (S)TEM diffraction patterns per minute)
- full well capacitance (highly brilliant as well as low intensity peaks e.g. of a diffraction experiment have to be differentiated).

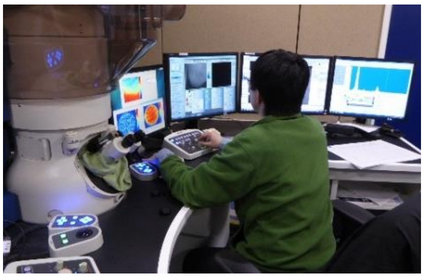
About five years ago for the first time the pnCCD (S)TEM Camera has offered these properties to the market and has since then continuously improved its convenience and capabilities.

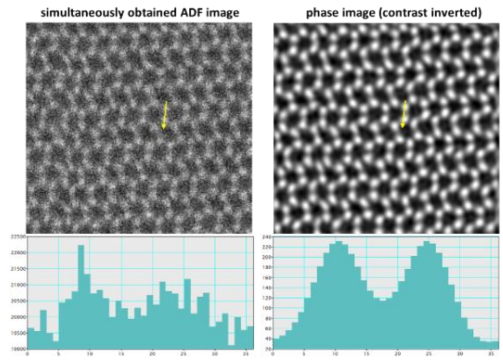
The pnCCD (S)TEM Camera [1] is based on a fully sensitive pnCCD well established as a high performance direct detecting electron or X-ray sensor: with a pixel size of 48 x 48 μm^2 the pnCCD exhibits a spatial accuracy of better than 10 µm and works over an electron energy range from 20 keV to 300 keV. The 264 x 264 format devices can be read out 1.150 times a second with the highest position and intensity resolution. If on-chip binning is used the speed can be increased to 8.000 frames (images) a second. Its radiation hardness allows a constant use of the detectors over more than 10 years without noticeable degradation.

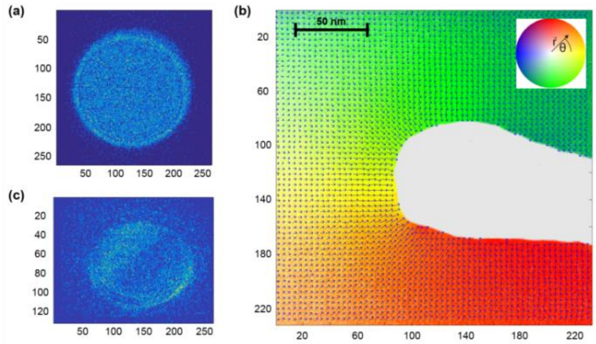
Live View Imaging has been developed to see instantaneous and averaged diffraction pattern in parallel to the well-known and established (S)TEM images which at the same time could be filtered to certain diffraction peaks only. Moreover, as the camera is able to measure the Bright Field it can be used for easy adjustment and position alignment of the sample without the need of detector switching. The newly developed operation modi of the camera allow the detection and quantification of highly brilliant diffraction peaks simultaneously to very weak signals (see e.g. Fig. 4 which has been taken in anti-blooming mode). Due to the very low noise level of only 30 ENC single electrons can be detected and quantified down to 20 keV electron energy.

Several applications have been tested in the past years for the first time – pioneer work has been done by Knut Müller in the field of strain analysis [2], by Paul Kotula [3] in the field of diffraction mapping, by Hao Yang for nano tube analysis [4], by the group of Peter Nellist in ptychography [5], see below for some additional application examples, taken in cooperation with JEOL Japan (Y.Kondo, R.Sagawa) and FZJ Jülich (R. Dunin-Borkowski, V. Migunov) and Cambridge University (P. Midgley, D. Johnstone).

In the talk specifications and advantage of pixelated detectors in modern (S)TEM analysis are described as well as several application examples. An outlook of the pnCCD camera to be used for ultrafast TEM imaging is provided.







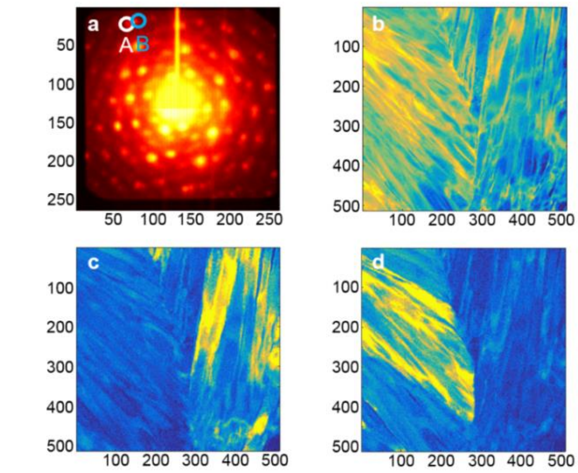


Fig. 1 – (a) The pnCCD Camera and (b) its user interface. **Fig. 2** – A graphene sample measured with a JEOL ARM 200 F with 256 x 256 probe positions at 2000 fps within 35 sec – CCD results in comparison with ADF.
Fig.3 – Electrical field mapping around a tungsten needle by means of analysis of the displacement of the BF disk. The image was recorded with 1000 fps and 256 x 256 probe positions within less than 1 min.
Fig. 4 -- A TiFeMo alloy sample with brilliant and weak diffraction peaks was measured in anti-blooming mode with a FEI Titan 80-300 at 512 x 512 probe positions at 1000 fps within 4 mins: (a) average diffraction pattern (b) full (S)TEM image (c-d) (S)TEM image filtered to diffraction regions A and B respectively.

References:
1. H. Ryll et al, Journal of Instrumentation 11, P04006 (2016).
2. K. Müller et al., Appl. Phys. Lett. 101 (2012), p. 2121101-2121104.
3. P. Kotula et al., Microsc. Microanal. 22 (2016), 466
4. H. Yang et al., Nature Comms. 7 (2016), 12532.
5. P.D. Nellist et al., Microsc. Microanal. 22 (2016), 466.