Merlin

MerlinEM

Application Note

4D-STEM and virtual STEM detection

Key methods:

- **4D-STEM**
- **ABF**
- **HAADF**
- **BF**
- **ADF**
- **CBED**

MerlinEM key specifications: noiseless readout, zero dark counts; no dead time with 1-bit (18800 fps), 6-bit (3200 fps) or 12-bit (1800 fps) imaging; 30 keV - 300 keV operation; pixel size 55 x 55 μm; active area: 14 x 14 mm (256 x 256 pixels), 28 x 28 mm (512 x 512 pixels), 56 x 14 mm (1024 x 256 pixels); DQE at 60 keV: 1 at Zero frequency, 0.45 at Nyquist; MTF at 60 keV: >0.62 at Nyquist.

Using a pixelated detector in scanning transmission electron microscopy (STEM) opens a plethora of opportunities. Techniques like long range and atomic resolution electromagnetic field imaging, ptychography, fluctuation electron microscopy and others would not be as efficient or outright impossible without resolving a full diffraction pattern for each point of the scan. Using a pixelated detector in STEM results in a 4D dataset (2D scan and 2D diffraction dimensions) - hence 4D-STEM. It will eventually become a goto technique in STEM[1].

In this note, we will show examples of using a MerlinEM detector to generate established signals in STEM. We will use LiberTEM[2] software to reconstruct the data and specifically its GUI web interface. We will show examples of virtual STEM images and convergent beam electron diffraction (CBED) from a few different samples. The figure below shows some examples of data which will be shown in this application note.
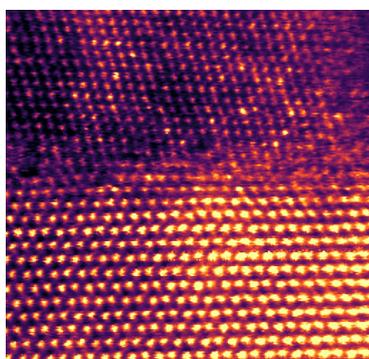
Atomically resolved TiN/HfO2 interface
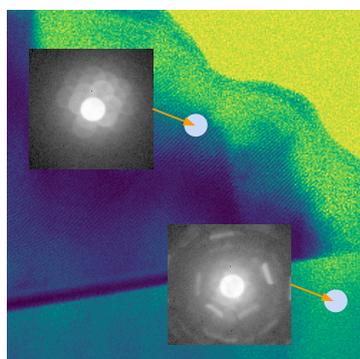
CBED details in conventionally prepared lead free antiferroelectric

Quantum Detectors would like to acknowledge the AEM group at TU Darmstadt for collaboration on this application note, namely Alexander Zintler and Leopoldo Molina-Luna.

1   Ophus, Colin. Microscopy and Microanalysis 25.3 (2019): 563-582. https://doi.org/10.1017/S1431927619000497
2   https://libertem.github.io/LiberTEM/

Virtual detection in STEM

By acquiring a full diffraction pattern for each point of a scan, we can recreate standard STEM signals after the experiment by summation of the information within the respectful area of diffraction patterns. We essentially replace multiple STEM detectors (this could be annular detectors, a bright field detector and/or DPC segmented detector) with a single pixelated detector [3]. Apart from the versatility of this approach, an additional advantage is that all the virtual detectors have a calibrated response and therefore data can be readily used in techniques like atom counting or others.

Fig. 1: Comparison of standard STEM configuration and STEM with a pixelated detector.
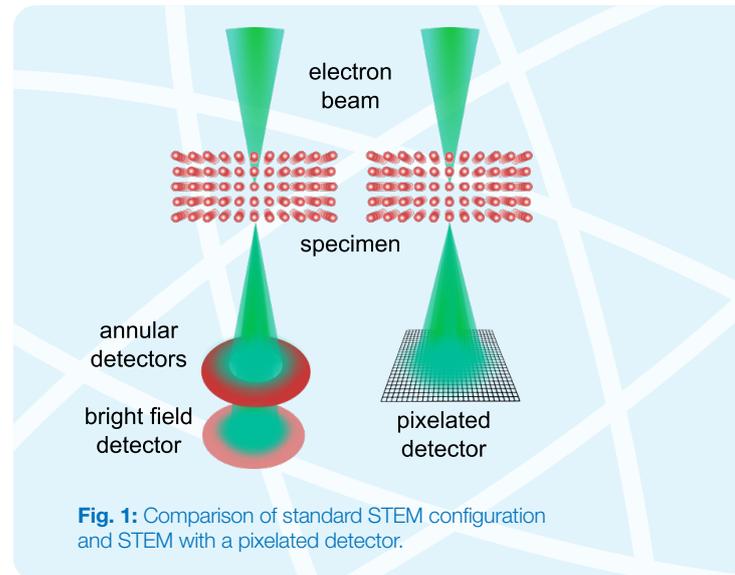
MerlinEM with its high dynamic range and versatile read-out system is ideally suited for 4D-STEM data acquisition. Hardware based electron counting offers exceptionally low noise by removal of thermal background. After the electron is detected, every process is digitised therefore no additional noise is added by the read-out system. **Fig. 2** shows an example of a single probe position CBED diffraction pattern from a 4D-STEM dataset. In the example the contrast within the central probe can be clearly seen together with single electrons further away from the optical axis. The frame time was 0.5 ms and dynamic range was set to 6-bit. With these settings, a 256x256 probe position dataset can be taken in 32 seconds, which will minimise sample drift issues. The maximum speed of MerlinEM can be achieved with 1-bit dynamic range (18.8k fps) and it allows it to image the same area within ~3.5 seconds.
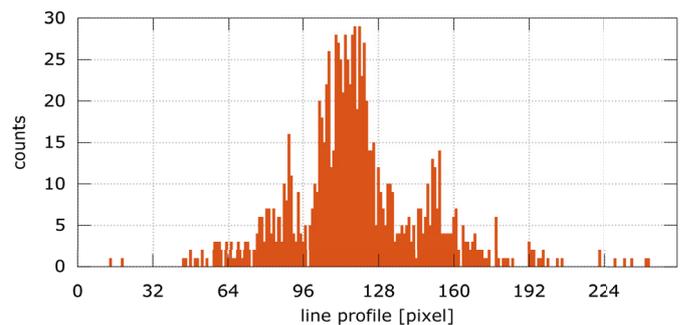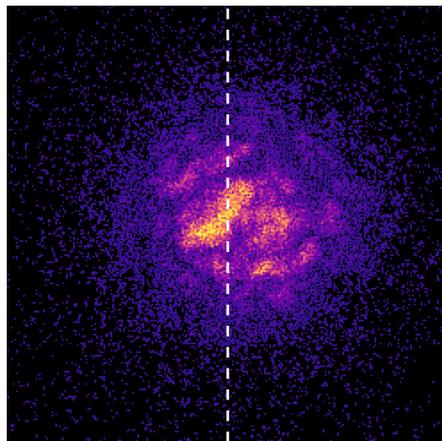
Fig. 2: A single probe position from a 4D-STEM dataset together with a line profile demonstrating single electron sensitivity and zero thermal background noise. The frame was taken with 0.5 ms frame time and 6-bit detector dynamic range.

3   If an annular detector is available above the position of the pixelated detector, the annular dark field images can be acquired together with the pixelated detector to collect additional information from larger scattering angles.

Atomically resolved interface in TiN/HfO2 sample

Fig. 3 shows a set of virtual STEM images generated from atomically resolved imaging of TiN/HfO2 interface. High angle annular dark field (HAADF), annular dark field (ADF), annular bright field (ABF) and bright field (BF) images were generated from corresponding areas of the detector which are shown in insets to the right of each image. The images follow the basic characteristics of each detector type: HAADF resolves high atomic number species as the brightest in the bottom half of the image following the adsorptive-contrast characteristics; ADF and ABF show a varying strength mixture of adsorptive- and phase-contrasts and BF shows mostly phase-contrast. The advantage of 4D-STEM here is that we can redefine each detector's area to achieve ideal imaging for a given material type and even explore this after the experiment. This offers a maximum versatility together with options to explore the data further with modalities like DPC, Centre of Mass, electromagnetic field imaging and others.
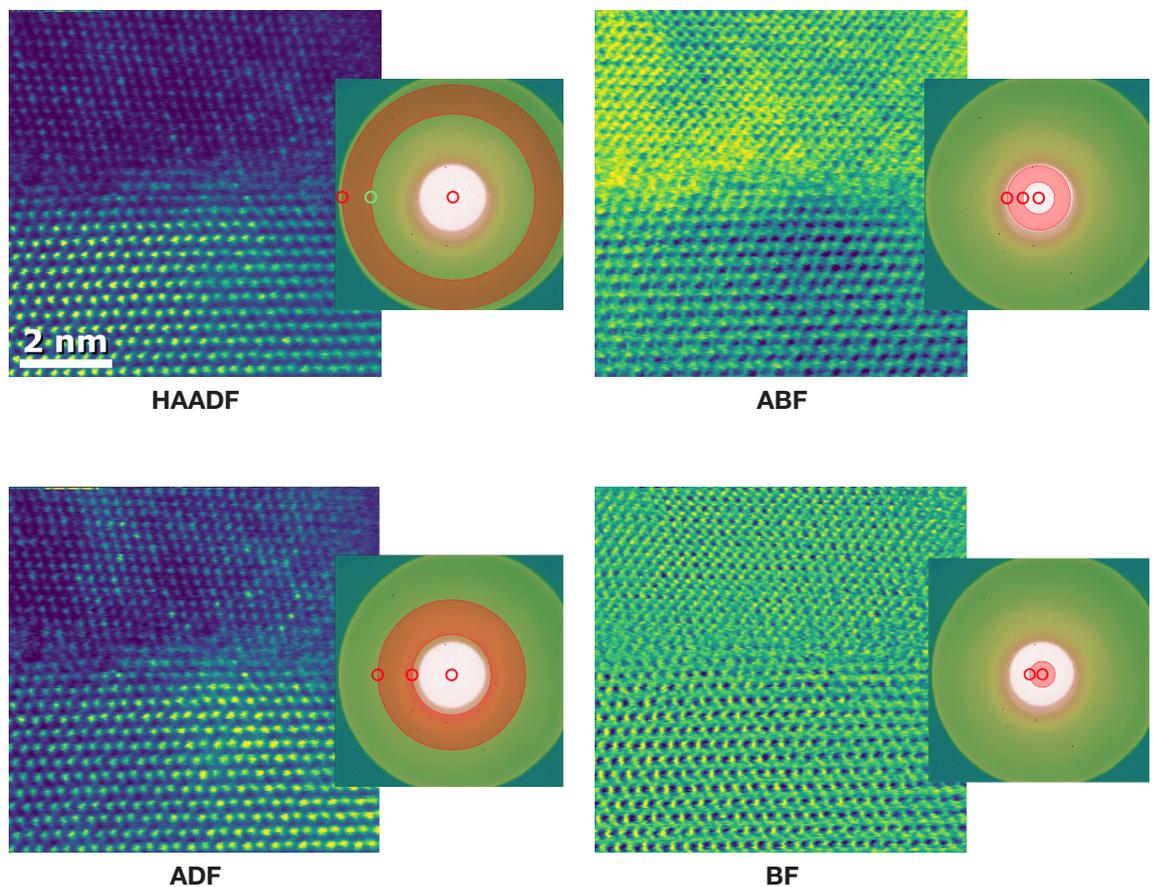
Fig. 3: Demonstration of atomic resolution virtual STEM imaging in 4D-STEM of TiN/HfO2 interface. The summed parts of the detector are shown in the insets next to the figures. Data and sample courtesy of AEM group at TU Darmstadt by Alexander Zintler and Leopoldo Molina-Luna.

Complex sample with depth and material changes

Another desirable feature of 4D-STEM is the ability to characterise complex samples. This is demonstrated in **Fig. 4** where multiple position averaged convergent beam diffraction patterns (PACBED) are shown with the respective area they were taken from. The sample is conventionally prepared lead free antiferroelectric. The overview on the left in **Fig. 4** is a bright field image generated by summing the central disk area. Numbered PACBED patterns show:

- high order Laue zone lines in thicker parts of the sample in **1, 2** and partially over interface in **6,**

- first order Laue zone ring in **3** and **4,**

- a thin part of the sample showing multiple diffracted disks in **5.**

Interestingly, other effects like shifts of the whole pattern (between **3** and **4**) are visible which makes the classic STEM imaging difficult - the probe cannot be positioned in the centre of the detector for the whole sample. This kind of effect might be present due to the local differential of the thickness, crystal bending or possibly strain.
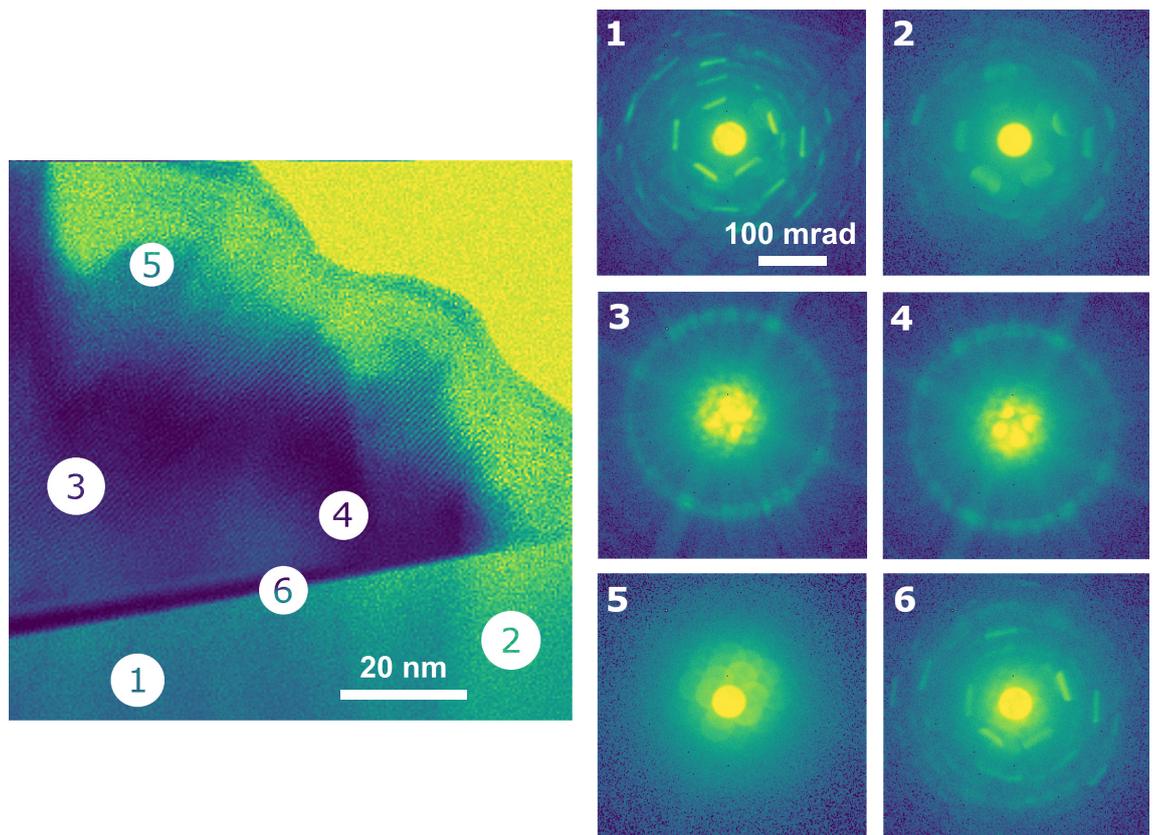
Fig. 4: Example of 4D-STEM data in a conventionally prepared lead free antiferroelectric sample. Diffraction patterns from each numbered areas were summed and their logarithm was plotted to show differences in local crystal structure. The size of the white circle corresponds to the size of the area summed. Data and sample courtesy: Alexander Zintler, Hui Ding, Leopoldo Molina-Luna, AEM group, TU Darmstadt.