An Electron Microscope Pixel Array Detector as a Universal STEM Detector

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Complete information about the scattering potential of a sample is in principle encoded in the distribution of scattered electrons from a localized beam propagating through it. A new generation of high speed imaging detectors brings us closer to this goal and will allow us to explore practical limits and identify the most promising methods of analysis. We have recently developed an electron microscope pixel array detector (EMPAD) that functions as a compact and high-speed, high dynamic range electron diffraction camera (Figure 1a). It has single electron sensitivity with a signal/noise ratio of 140 for a single electron at 200 keV [1,2]. It has a dynamic range of \(10^6\) for primary electrons—i.e. a pixel can detect from 1 to 1,000,000 electrons, and reads out an image frame in 0.86 ms. These properties allow us to record essentially an image of all the transmitted electrons, from the unscattered beam to out beyond the HOLZ lines, and do so for every probe position in a real-space, atomic resolution image. Not only does this allow quantitative and simultaneous annular dark and bright field signals on an absolute scale, but from the analysis of the spatially-resolved diffraction patterns we can extract thickness, strain and tilt, octahedral rotations, polarity and even electric and magnetic fields.

As a practical demonstration of dynamic range Figure 1b and 1c show the diffraction patterns generated from 1ms and 100 ms acquisition with a sub-2A beam containing 10 pA probe current at 200 keV in a Tecnai F20. The high-angle scattering is detected with single electron sensitivity simultaneously with the unsaturated high-intensity central beam and crystallographic Bragg discs. Quantitative atomic resolution images from this data are given in [1,2]. Less conventional imaging modes are explored here. Generalized multipole imaging, from the \(n^{\text{th}}\) moment of the diffraction pattern directly gives the expectation value of the \(n^{\text{th}}\) moment of the scattered probe’s momentum, \(\langle k^n \rangle\). The first moment, or center of mass (CoM) is the probability current flow, which in a thin sample is the beam deflection from the Lorentz force [3,4]. Thus, changes in the scattered beam’s momentum are directly measured over all angles and a specimen’s in-plane magnetic and electric fields can be extracted. In thick samples, the influence of crystal tilt and absolute local sample thickness variations can also be accounted for by recording the full pattern. Fig 2a shows a \(k^2\)-weighted diffraction pattern which enhances the Kikuchi bands, isolating crystal tilt. Fig 2b shows the measured tilt of domains in multiferroic BiFeO\(_3\). From a naïve CoM analysis, this could be incorrectly interpreted as an electric field. Fig 2c shows the true x-component of the magnetic field in Co imaged by CoM Lorentz-STEM with the familiar magnetic ripple texture of the material.

Fast MMPAD readout allows 4D-STEM imaging at atomic resolution. Example data from our Cs-corrected Titan at 120 kV is shown in Fig 3 for SrTiO\(_3\). Fig 3a shows the annular dark field image from the EMPAD, which is similar in appearance to the \(2^{\text{nd}}\) moment signal \(\langle k^2 \rangle\) of Fig 3b. \(\langle k^2 \rangle\) is a measure of the scattering power of the sample. The CoM images (Fig 3c) give the x&y derivatives of the potential, which can be obtained by integration [5,6] (Fig 3d), showing the oxygen sublattice clearly, well beyond what the ABF signal provides. [7]
References:
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Fig. 1. a) EMPAD detector on FEI F20 showing compact design and sensor. CBED pattern of BiFeO$_3$ recorded in b) 1 ms and c) 100ms with 10 pA of beam current. 20 mrad scale bar.[2]

Fig. 2. Mapping crystal tilt from Kikuchi band shifts in multiferroic BiFeO$_3$. a) $k^2$-weighted diffraction pattern. b) x components of the crystal tilt. (65 sec acquisition). c) Magnetic field ripples in Co thin film mapped in field-free LM-STEM from CoM shifts of the central beam [2]. 65 sec acq.

Fig. 3. 120 keV EMPAD STEM data from SrTiO$_3$: Simultaneously extracted a) ADF, b) Second Moment, c) First moment and d) iCOM image from symmetrized integration of the first moment (c) to derive an effective projected potential, showing the oxygen sublattice. Blue circles- Sr, Red-O, Green- Ti+O. 9 second acquisition time on Cs-corrected Titan Themis 60-300.