Wide Dynamic Range, 10 kHz Framing Detector for 4D-STEM

Hugh Philipp¹, Mark Tate², Katherine Shanks³, Luigi Mele⁴, Maurice Peemen⁵, Pleun Dona⁵, Reinout Hartong⁵, Gerard van Veen⁵, Yu-Tsun Shao⁶, Sol Gruner³, Julia Thom-Levy⁷ and David Muller⁸

¹Laboratory of Atomic and Solid State Physics (LASSP), Cornell University, Ithaca, NY, USA, Ithaca, New York, United States, ²Laboratory of Atomic and Solid State Physics (LASSP), Cornell University, Ithaca, NY, USA, ITHACA, New York, United States, ³Laboratory of Atomic and Solid State Physics (LASSP), Cornell University, Ithaca, NY, USA, United States, ⁴Materials and Structural Analysis Division, Thermo Fisher Scientific, Achtseweg Noord, Eindhoven, 5651 GG, Netherlands, Netherlands, ⁵R&D Laboratory, Thermo-Fisher Scientific, Netherlands, ⁶School of Applied and Engineering Physics, Cornell University, Ithaca, NY, USA, United States, ⁸School of Applied and Engineering Physics, Cornell University, Ithaca, NY, USA, United States, ⁸School of Applied and Engineering Physics, Cornell University, Ithaca, NY, USA, Ithaca, New York, United States

Hybrid pixel array detectors (PADs) have expanded the fidelity, frame rate and dynamic range for diffractive imaging in electron microscopes [1,2]. With their high detective quantum efficiencies (DQE), the noise performance limited is by counting statistics. Consequently, improving the precision and quality of high-speed images requires increasing the linear dynamic range and the maximum usable beam current. Here we describe a new prototype hybrid PAD, our next-generation electron microscope pixel array detector (NG-EMPAD) that expands both the framing speed and dynamic range of our earlier EMPAD design [2]. Like the EMPAD, electrons are detected directly in a 500 µm thick silicon sensor bonded pixel-by-pixel to a custom signal processing integrated circuit (IC). Each pixel in the IC integrates charge carriers produced by the high-energy incident electrons. In-pixel circuitry uses a combination of techniques to extend the dynamic range, including adaptive gain switching [3] and the quantitative excess charge dumping similar to the EMPAD [2]. In-pixel buffering of signals during readout allows acquiring a new image while reading out the previously collected image, yielding an image collection duty cycle of nearly 100%.

The NG-EMPAD is capable of continuous imaging at 10 kHz. Detector performance was tested on a TFS Titan Themis from 80-300 keV (the detector is capable of imaging down to 30 keV). Figure 1 (left) shows the spectral response of the detector at low fluence, showing that single electrons are easily distinguished from background. Figure 1 (right) shows that the linear response extends to 180 pA of beam current/pixel at 300 keV. Figure 2 shows high-precision maps of ferroelectric polarization and strain from the same data set recorded on the NG-EMPAD using a 2 nA beam current and dwell time of 100 µs. The high signal to noise ratio across the full diffraction pattern made it possible to use the Kikuchi bands to measure polarity, and cepstral-STEM [4] processing of the diffraction peaks to map strain simultaneously.

The 10 kHz imaging of the detector was synchronized with STEM rastering to quickly collect high resolution data sets for annular dark field, bright field, differential phase contrast, and ptychographic imaging in a single pass exposure. The high duty cycle and detective quantum efficiency minimizes sample dose and the fast frame rate enables us to outrun environmental noise that reduces coherence and stability of very high spatial resolution imaging methods. This has greatly improved our yield for deep sub-Angstrom reconstructions of thick samples using multislice ptychography[5].

Detector development was supported by Thermo Fisher and DOE- DE-SC0017631. Cornell microscope facilities supported by NSF DMR 1719875 and DMR 1539918.

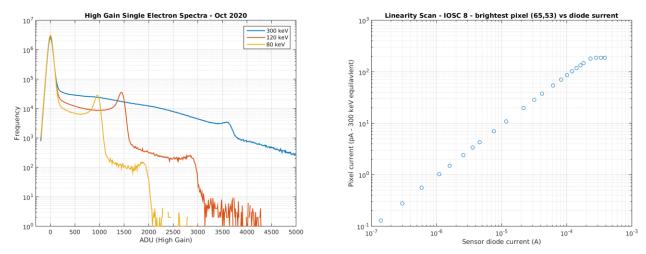


Figure 1. (left) Histograms of the pixel response at 80, 120, and 300 keV. Each dataset is comprised of 30,000 frames of low-fluence, uniform illumination (3 orders of magnitude and monitored with the current flowing from the sensor power supply. Saturation current for 300 keV is 180 pA/pixel or 1.125x109 e-/s/pixel.

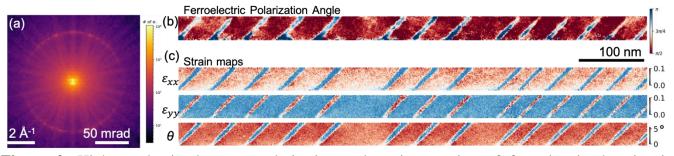


Figure 2. High-speed, simultaneous polarization and strain mapping of ferroelectric domains in PbTiO3/DyScO3 using the NG-EMPAD, acquired with a 512×512 scan and over a region of 715×715 nm2, recorded in less than 30 seconds using a 2 nA probe current and 100 μs/pixel dwell time. (a) Single CBED pattern from the scan, on the DyScO3 substrate along the [] zone axis recorded at 300 kV. The HOLZ ring can clearly be resolved. Within the same dataset from the PbTiO3 film, (b). ferroelectric a/c domains and (c) lattice strain determined using the polarity-sensitive Kikuchi bands and EWPC transform, respectively.

References

- [1] C. Ophus, Microscopy and Microanalysis 25, (2019): 1–20.
- [2] M. W. Tate et al.," Microsc. Microanal., vol. 22, no. 1, pp. 237–249, (2016)
- [3] B. Henrich et al.," Nucl. Instrum. Methods Phys. Res. Sect. Accel. Spectrometers Detect. Assoc. Equip., vol. 633, pp. S11–S14, 2011
- [4] E. Padgett, *Ultramicroscopy* **214**, (2020): 112994
- [5] Z. Chen et al., arXiv:2101.00465. (2021). https://arxiv.org/abs/2101.00465