

Nanobeam 4D STEM with MerlinEM: at the intersection between crystallography and microscopy

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Scanning/transmission electron microscopes (S/TEMs) are invaluable tools for the structural and spectroscopic characterisation of nanosized matter, in no small part due to their flexibility to be configured to perform a wide variety of experiments. In recent years, the development of hybrid pixel direct electron detectors (HPDEDs) and dedicated data acquisition and processing software has led to a resurgence of interest in three dimensional electron diffraction (3D ED) and four dimensional STEM (4D STEM) methods. 3D ED is an umbrella term for a set of protocols that involves the collection of three dimensional electron diffraction data from several crystallographic orientations for complete, and potentially absolute, structural elucidation [1]. In contrast, in a 4D STEM experiment the entire 2D diffraction pattern is collected at each probe position in a 2D scan [2]. The 4D STEM dataset is therefore richer in crystallographic information than a conventional 2D STEM image, and is ready for processing and analysis by a host of algorithms and software. Researchers have used 4D STEM to measure the electric and magnetic fields of materials [3], and to map the crystalline phase and orientations of polycrystalline domains [4], as well as the sample strain [5].

3D ED and 4D STEM are different facets of the same gem. They are complementary techniques that can provide different insights into the properties of a sample. The structural information obtained from 3D ED methods can facilitate 4D STEM data interpretation. For example, for nanometer scale crystallographic phase and orientation mapping from 4D data collected with a scanning quasi-parallel nanobeam, it is necessary to have a structural file from which to simulate diffraction patterns for comparison with experimental data. Orientation and phase are assigned to patterns that best match simulation. These structure files can be provided by 3D ED methods. Nanobeam 4D STEM is the intersection of 3D ED and microscopy.

As mentioned above, both 3D ED and 4D STEM have benefited from novel HPDEDs, owing to their high detective quantum efficiencies (DQEs), readout speeds, dynamic ranges and radiation hardnesses. Here, we describe a typical nanobeam 4D STEM setup with MerlinEM, including precession using NanoMEGAS hardware and software. We demonstrate how the MerlinEM HPDED significantly enhances data quality, enabling improved signal-to-noise ratios and more accurate diffraction pattern acquisition. Finally, we highlight the advantages of these improvements in the context of both standard and emerging data processing workflows, including 3D ED-assisted virtual imaging and phase and orientation mapping, applied to both organic and inorganic systems.

[1] M. Gemmi et al., ACS Central Science, 1315-1329 (2019)

[2] C. Ophus, Microscopy and Microanalysis 563-582 (2019)

[3] W. Lijun et al., Ultramicroscopy 113745 (2023)

[4] E. Thronsen et al., Ultramicroscopy 113861 (2024)

[5] P. Crout et al., arXiv:2307.01071v1 (2023)