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Scanning Electron Diffraction of 'Soft' Materials – Application to Organic and Hybrid Systems

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There have been some remarkable advances in electron microscopy over the past ten years or so, including the advent of aberration correctors and monochromators on commercial systems. However, equally important has been the increase in the speed and efficiency of cameras, detectors and spectrometers, coupled with a rise in associated computational power for image and spectral processing, and more generally, for handling and mining large multi-dimensional data sets.

The relative ease with which such multi-dimensional data sets can now be processed has been a critical factor in the increasingly wide adoption by the microscopy community of crystallographic mapping, or 'crystal cartography', using the (scanning) transmission electron microscope, (S)TEM. This has gone hand-in-hand with the increasing use of more image-focussed techniques such as electron ptychography, but whether the purpose is to acquire high fidelity and quantitative images or spatially-resolved diffraction patterns, both require the acquisition of large 4-dimensional data sets composed of diffraction patterns acquired at every real space pixel within a scan of a region of interest.

For high resolution image reconstruction, for example, electron ptychography may be used, in which a highly coherent convergent probe is used to form CBED patterns with disc overlaps encoding relative phase changes between diffracted discs. By contrast, for crystallographic mapping at the nanoscale, a near diffraction-limited (almost parallel) beam is used to form 'spot' patterns which extend out in reciprocal space (with a resolution often beyond $1Å^{-1}$) but are formed using real space probes ca. 3-5nm in diameter; this type of mapping is often called 'scanning electron diffraction', or SED [1]. A series of patterns recorded by SED may be used to determine relative changes to the pattern geometry across the region of interest and those changes used to determine crystal phase, orientation and strain, see Figure 1.

In this paper we will show how SED may be used to provide nanoscale crystallographic information not readily obtained using other techniques and, in particular, focus on how fast, low-dose acquisition of SED patterns allows for the study of highly beam-sensitive 'soft' materials [2], such as polymeric [3,4], organic and hybrid inorganic-organic systems [5]. A number of examples will be presented, including: (i) using SED to probe the nature of defects causing non-radiative losses in halide perovskite thin films; (ii) combining 3D-ED with SED to determine molecular packing and (optically important) stacking defects in perylene diimide (PDI) organic semiconductor nanobelts; (iii) using SED to probe domain structures in UiO-66 metal-organic frameworks (MOF) structure, originating through missing metal clusters; (iv) revealing the sensitivity of SED to determine defect ordering in 'condis' polyethylene [6].

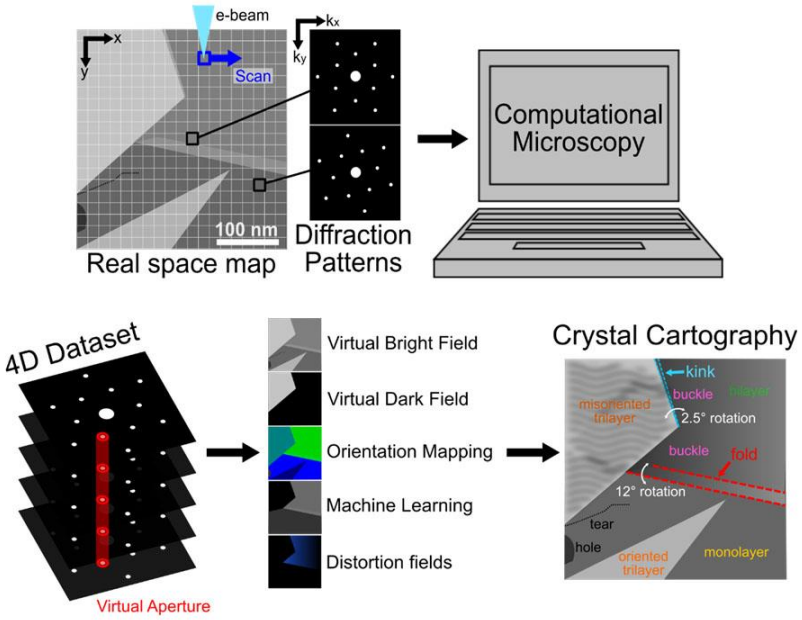

Figure 1. Schematic representation of the method of scanning electron diffraction with an example showing the possible analysis of microstructural features in a low-dimensional material.

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