Striving for precise metrology with the modern STEM

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The aberration corrected STEM's ability to produce so many signals from every raster point makes it an indispensable tool to the materials scientist. However, somewhat paradoxically, the enormous magnifications achievable, and the sensitivity of this instrument's scanning system, means that great care is needed when seeking to extract quantitative information. In this talk, some recent approaches which maximise the quantitative utility of results while minimising sample damage will be presented; including from single-scan annular dark-field (ADF) frames with absolute contrast (2D), ADF frame-series (3D), spectrum-image series (4D), and multi-pass electron ptychography and EMCD focal-series (5D). Examples will be presented from particular materials systems, but the approaches for optimising experiment design are widely transferable.

The ADF signal's high-angle scattering offers a route to obtain both atomic-number and/or sample-thickness information at high-resolution. However, this first needs to be normalised by the sensitivity of the detector to obtain 'fractional scattering' data suitable for comparison with simulation. Necessarily then, any study of quantitative ADF data requires an awareness of the inhomogeneities within, and variability between, ADF detectors [1]. Even with the sensitivity of the detector mapped, it is not trivial to determine the correct collection angles to use in reference simulations, and recent calibration approaches have now allowed these crucial parameters to be obtained with increased reliability [2]. With these careful calibrations it becomes possible to both analyse the precision of ADF measurements (such as atom-counting), and to inform acquisition design in advance if the experiment [3].

As serial-scanned data is necessarily recorded over a finite time, environmental effects can lead to scanning-artefacts [4]. However, it has been shown that by using multi-frame acquisition and alignment (non-rigid registration), the retrievable ADF intensity-precision, and strain-precision both improve with approximately 1/√number-of-frames. Even where a finite total electron budget is available, this can be optimised and lead to picometre precision imaging [5]. Such quantitative data can then be used (with energy minimisation) to retrieve atomistic models of nanoparticles for catalytic studies [6].

This same multi-scan approach can also be extended to spectrum image data (4D: x,y,E,t) improving both the spectral signal-noise ratio and lattice fidelity, while also reducing the effects of beam-damage and enabling digital super-resolution [7]. Still further, with the advent of electron counting cameras a new possibility for full-field recording with pixelated detectors makes possible electron ptychography across multiple passes (5D: x,y,kx,ky,t), enabling atomic-scale phase mapping with high-precision [8]. Looking to the future, opportunities for multi-frame EMCD focal-series and ultra-low-dose STEM operation, ADF acquisition and image reconstruction will be discussed.

References:

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