

Getting the Best from an Imperfect Detector – an Alternative Normalisation Procedure for Quantitative HAADF STEM

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Recently, in high-angle annular dark-field scanning transmission electron microscopy (HAADF STEM), normalising the recorded image data into units of ‘fractional beam-current’ has become increasingly popular [1]. This technique, sometimes called quantitative-STEM or quantitative-HAADF, is extremely useful as it facilitates the direct comparison of experimental and simulated data with both expressed on identical absolute scales [2-3]. To enable an accurate measurement of the scattering, it becomes crucial to study the collection efficiency of HAADF detectors, and we find that across instrument manufacturers many have significantly asymmetric response behaviours [4]. Most significant is the variation in collection efficiency with scattering angle, and importantly what overall ‘effective efficiency’ should be chosen to normalise experimental data. Here we discuss a refinement to the method of analysing detector efficiency, that better describes the interaction between asymmetric detectors and the electron-flux distribution across the detector plane.

Generally, comparing experimental HAADF data with simulation requires careful measurement of the specific detector’s radially varying sensitivity, and to simulate a library of images to match. This requirement to recalculate library values for every specific detector is at best time-consuming and at worst can cause discrepancies when collaborating with, or verify the findings of, others. Our alternative method is to calculate the simulated reference data using a ‘perfect’ detector (totally uniform sensitivity response). The detector is then only defined by its inner and outer angles (i.e. the camera-length chosen) and is reproducible across different instruments. To compare between experiments using an imperfect detector, and the simulated library, the ‘effective sensitivity’ must be calculated, which describes the average efficiency expressed as a single number. To calculate this we first perform an experimental detector sensitivity scan, Figure 1 a), from which we identify the active area, Figure 1 b). Next using either simulated or experimental camera-length series, or direct recording on a CCD, the distribution of the detector plane electron flux is determined (Figure 2). For angles beyond the size of the bright-field disk this is reasonably well described by a power law (Figure 2, dashed line). Using the measured flux distribution, the flux across the active region of the detector is normalised so that it integrates to unity, Figure 1 c). Finally this flux distribution is multiplied by the experimental efficiency scan to give the ‘flux-weighted detector efficiency’, Figure 1 d). Integrating this plot then gives the appropriate value to normalise experimental data by, accounting for asymmetry in both the detector and the electron flux.

To verify this new approach, scattering cross-sections were calculated for the case of a perfect detector and also for the asymmetric experimental detector, with poorer efficiency at low scattering angles (Figure 3). Simulations reflect increasing numbers of platinum atoms imaged at 300 kV, using a 21 mrad semi-angle, and 60-190 mrad collection angle, using the multi-slice absorptive-potential simulation method in STEMSIM [5]. Using the classic approach of normalising by the average sensitivity of the detector’s active area the cross-sections were significantly underestimated, however, normalising by the flux-weighted detector efficiency the correct values were indeed found. The benefit

of this method is most clear when it is considered that the majority of the electron flux falls at the low angle regions of the detector where most sensitivity variability often exists [4].

In conclusion, by considering the electron-flux distribution across asymmetric annular detectors, we find a robust quantitative STEM normalisation method that allows for reference data to be calculated more simply and for easier comparison of results between research groups [6].

References:

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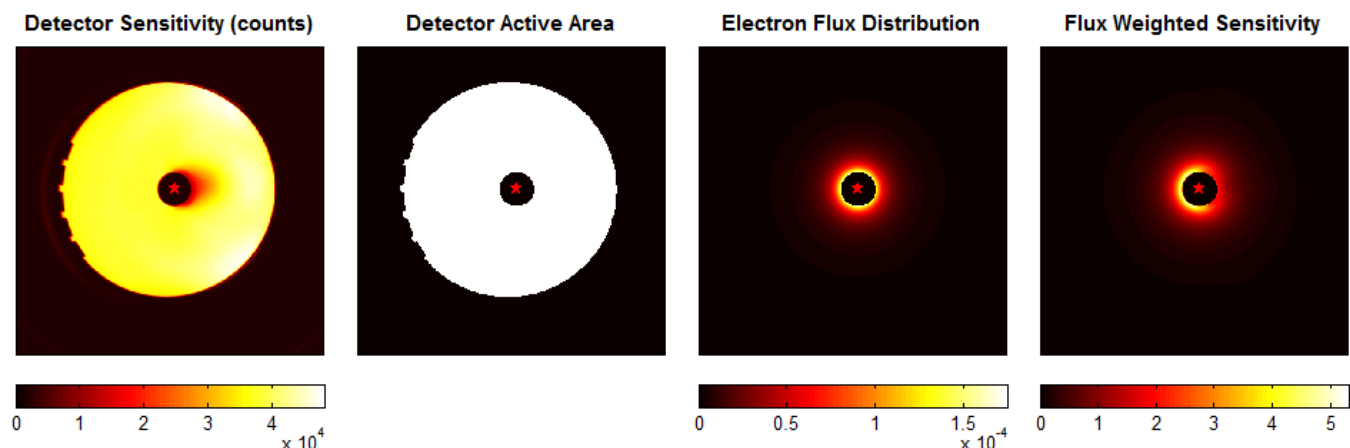


Figure 1. Sensitivity calculation stages; a) experimental detector map, b) the active area, c) the determined electron flux pattern, and d) the product of plots a) & c) giving the flux-weighted sensitivity.

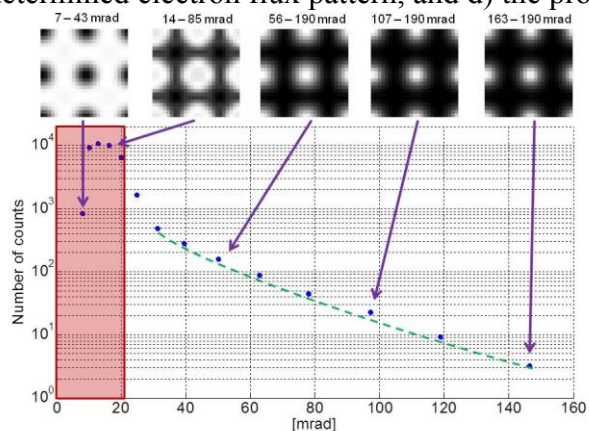


Figure 2. Measured flux distribution (dashed line) from simulated [100] oriented platinum camera-length series (inset). Red box indicates the size of the bright-field disk (21 mrad).

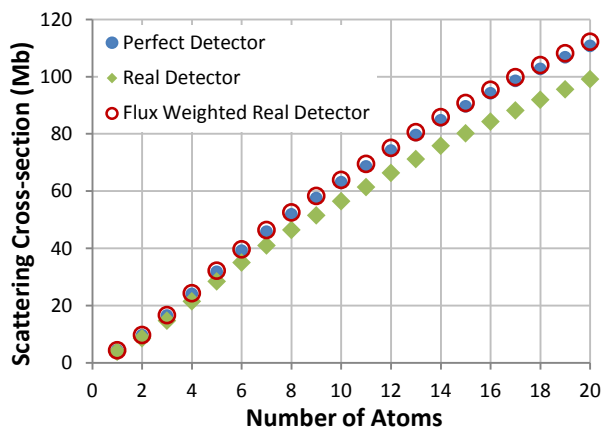


Figure 3. Comparison between three different simulated data; perfect detector (filled circles), real detector (diamonds) and a real detector with flux-weighted normalisation (open circles).