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Electron Dose Management for High Angle Annular Dark Field Scanning Transmission Electron Microscope Tomography of Beam Sensitive Materials

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High angle annular dark field tomography is a technique which allows the acquisition of three-dimensional information of crystalline materials [1]. Although this technique is easily applicable to beam resistant materials such as metals and some semi-conductors, current materials of interest, namely in the field of power storage (i.e., battery materials) and power generation (solar panels and fuel cells), are not as resistant to electron beams. Figure 1 B shows an increase in surface roughness where the previous higher resolution image A) was taken.

As electron dose management is key for biological TEM imaging, Recent tools such as direct detection cameras and phase-plate imaging are used to significantly reduce the electron dose required for imaging [2]. However, many of these approaches are unfortunately not transferable to tomography of crystalline materials, where dynamic effects can generate complex contrast leading to reconstruction artefacts [1].

The simplest route to reduce the dose for crystalline material tomography is therefore to optimize acquisition parameters. Depending on the facility and user experience, some parameters are not accessible. However, a user can usually control: magnification, frame size, detector gain, idle beam shutter, dwell time, and focus. Among these, the critical parameters are: frame size, magnification, dwell time, and focus. Frame size and magnification both affect pixel size. The dwell time, or acquisition time per pixel, will affect the signal to noise ratio. The focus of the scanning probe will also affect the local dose on the sample, since a probe smaller than the pixel size can induce unnecessary damage to a sample. Simply put, the dose D, in electrons per unit area, is proportional to the square of the magnification, *Mag*, multiplied by the pixel count, *nx* and *ny*, probe current *I*, and dwell time *t*, per frame.

$$D \propto Mag^2 * nx * ny * I * t \qquad (1)$$

By carefully tuning these settings for each material, beam damage can be reduced without affecting the reconstruction results.

Using SmartJ [3], an ImageJ implementation of the Smart Routine [4], the resolution of images acquired with several settings is measured. This simple design of experiment (DOE) allows a user to test the varying conditions and assess the minimal possible dose to resolution ratio [5].

References:

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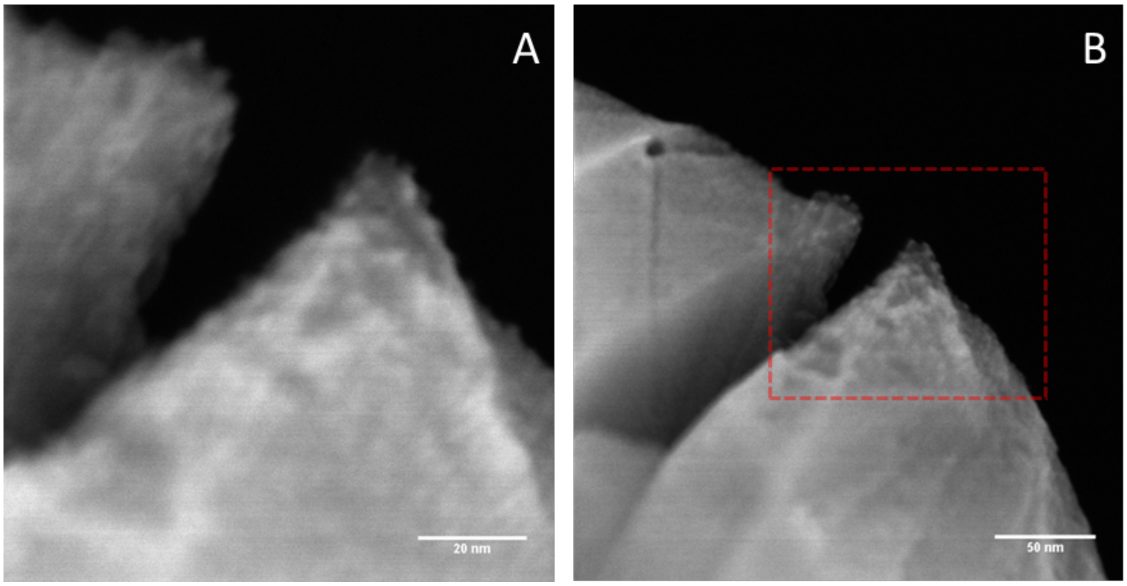

Figure 1. 300 keV secondary electron image (A) and resulting damage (B) during observation of a lithium bearing material, $Li_2FeSiO4$.