Aberration correction and scanning transmission electron microscopy (STEM) tomography; consequences and opportunities

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Electron tomography (ET) is now a well-established technique for the determination of 3D structure in materials at resolutions on the order of a nanometre [1]. In the last decade a wide range of contrast modes have been employed for reconstruction, of which high angle annular dark field (HAADF) scanning transmission electron microscopy (STEM) is arguably the most versatile and widely applied. The application of STEM ET is increasingly moving in two directions; higher resolutions and towards full quantitative analysis [2]. Unfortunately, prior art for ET is less than well suited to either of these goals, in particular the sample geometry and nature of STEM illumination are particularly limiting. A further complication is that aberration corrected instruments are increasingly being employed to carry out ET. While these instruments offer many advantages, in terms of imaging resolution and stability, they also offer significant pitfalls for ET.

Shown in Fig. 1 a) and b) are frames from a HAADF STEM tilt series from an Al-Cu-Sn alloy, acquired using a dual-aberration corrected FEI Titan^3 80-300, operated at 300kV with a convergence semi-angle of 15.1 mrad. This alloy has been rapidly quenched leading to the formation of precipitate decorated voids [3]. At zero tilt, Fig. 1 a), microstructural features are very clear, however when tilted to -72° the image clarity is much reduced. This reduction in quality is from a combination of two factors; an increase projected thickness of the foil specimen and the limited depth of focus (DOF) caused by the large convergence angle. As a result the ***sampling*** of the volume in reciprocal space is significantly reduced at high tilt angles; this reduction of sampling, from the ideal, is illustrated in Fig. 1 c). This is exactly the opposite of what is required for tomographic reconstruction where the high frequency information at high tilts is the most valuable.

In a conventional foil type specimen the increase in projected thickness to unusable levels can only be avoided by selection of a thin region of the specimen. Unfortunately this will reduce the sampled volume, a critical consideration for quantitative analysis. If however a "needle" geometry specimen is employed [2] the projected thickness changes very little with tilt, allowing equal sampling in all directions and a return to the 'ideal' case in Figure 1 c). In addition this geometry allows complete 360° rotation, with a suitable specimen holder, extending the angular sampling leading to homogeneous tomographic reconstructions. Specimens that have been prepared for three dimensional atom probe (3DAP), either by electropolishing or focused ion beam milling, are ideal "needles" for this kind of acquisition.

In a conventional STEM instrument the convergence semi-angle (α) is set to give optimal resolution as a function of the Cs of the probe forming lens [4]. In an aberration corrected instrument the α used is a function of the residual higher order aberrations after the corrector tuning [5]. While selecting the smallest possible probe size results in the highest "resolution", it is increasingly becoming apparent that a more sensible approach is to choose α to give an optimum contrast/resolution balance for the experiment being undertaken [6]. This is particularly the case in ET; as an increased α is partnered by a loss in DOF [7]. Shown in Fig. 2 are calculated probes with defocus for three different α values for a 3rd order aberration corrected STEM system; 15.1 mrad, as used above, 10.7 mrad and 2 mrad tuned for maximum DOF. While the absolute value for the DOF (in terms of the z-FWHM) of these probes is small, especially for 15.1 mrad, the relevant DOF for ET is rather that given a particular pixel size the DOF in which a significant proportion of the probe current remains within that pixel. Choosing a, somewhat conservative, value of 80% of the probe current the 1nm DOF for the three conditions in Fig. 3 are 70 nm, 130 nm and >500 nm respectively. The DOF for the 15.1 mrad case is still clearly too small given the sample thickness at zero tilt is ~ 50 nm (150nm at 70°). It is also obvious that the optimal α for 3D spatial resolution is vastly different than that for 2D spatial resolution. However, modern STEM's, while clearly offering little advantage in terms of resolution, offer a great flexibility in terms of illumination; with multiple condenser lenses allowing selection of a range α for a single condenser aperture size.

By carefully tuning the STEM convergence angle with the specimen geometry and thickness will ensure optimum sampling and maximum resolution can be achieved across a wide range of sample types and features sizes [8]

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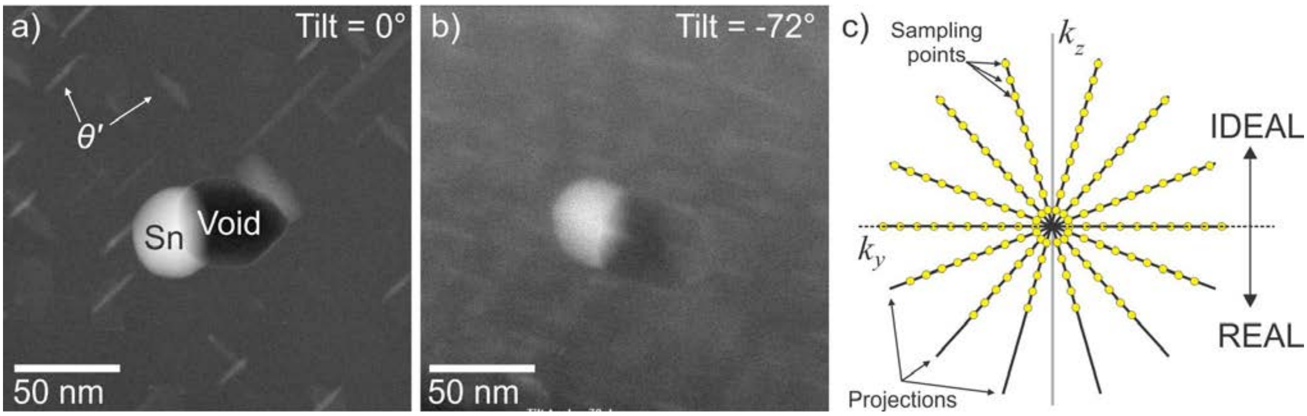

Figure 1. *Loss of information at high tilt. HAADF STEM image of a Sn/Void cluster in a Al-Cu-Sn alloy at a) 0° and b) -72° tilt. The loss of high frequency information is illustrated in reciprocal space in c). In the ideal case sampling is even at all tilts, but in reality DOF issues and sample thickness reduces the effective sampling in the* k_z *(depth) direction.*

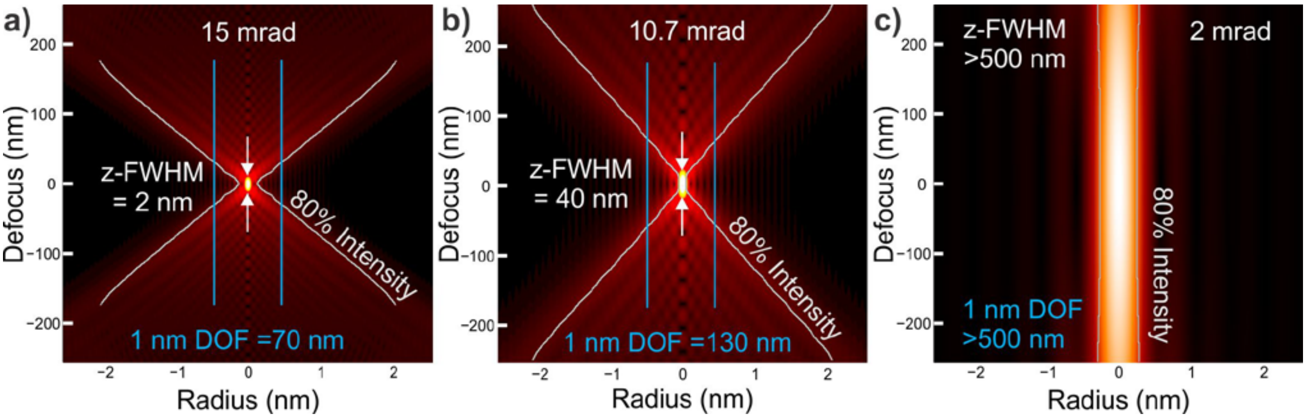

Figure 2. *Calculations of the 2D Integrated probe cross section with defocus for various convergence semi-angles, and measurements of the z-FWHM and 80% intensity 1 nm DOF. a) 15 mrad, as used for Fig. 1, b) An intermediate value of 10.7 mrad (similar to uncorrected systems) and c) illumination tuned for maximum DOF at 2 mrad.*