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Influence of Convergence Angle and Finite Effective Source Size for Quantitative Atomic Resolution EDXS

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Recent progress in energy dispersive x-ray spectroscopy (EDS) has enabled atomic resolution chemical mapping [1] which has become an invaluable tool for chemical analysis, particularly for identifying heavy elements [2]. Although recent development of Super-X technology has enabled qualitative identification of the atoms present in an atom column, quantitative atomic resolution analysis remains an active area of development. Many contributions to the atomic resolution signal have been studied, such as thickness and tilt; however, the effects of many other parameters remain unreported.

In this talk, we will first examine the effects of probe convergence angle on atomic resolution EDS signal via simulation and experiment. Using a probe corrected FEI G2 Titan 60-300 kV STEM equipped with a Super-X detector, atomic resolution EDS maps of <100> strontium titanate (STO) were collected using probe-forming convergence angles of 14 and 20 mrad with otherwise identical operating parameters. Each map was then lattice averaged using a template with the size of the unit cell translation vector in a similar approach as in Ref. [3]. The averaged unit cells were then replicated, producing a tiled representation of the averaged unit cell (Figure 1). By simulating the same structure using μSTEM [4], we observe agreement between simulated and experimental atomic maps. Unsurprisingly, both experiment and simulation reveals that 20 mrad produces maps with better spatial resolution; however, the signal from the atom columns appears stronger for the case of 14 mrad, especially for the light atom columns of pure O. Importantly, the difference in the O signal from the Ti/O columns is greater than O in pure O columns due to channeling [5]. We will discuss how the relationship between channeling and convergence angle raises important considerations for atomic level quantification.

Contrast dependence on the convergence angle is further explored in addition to finite effective source size and thickness. Simulations for a different system, Ni_3Al, display a similar behavior after considering the finite effective source size. By using a test example of <100> Ni_3Al, the maximum, mean, and minimum counts are plotted as a function of thickness for both 14 and 21 mrad, shown in Figure 2a. Without accounting for the finite effective source size, 21 mrad exhibits higher maximum intensity for the tightly spaced Ni (left). But incorporating the source size dramatically alters the signal from the atom columns (right), now displaying higher Ni maximum signal for 14 mrad rather than 21 mrad while the background remains lower. The influence of convergence angle and thickness will additionally alter the ratios of x-ray lines which directly impact quantitative elemental analysis. The impact on peak ratios and possible mechanisms for convergence angle will be presented [6].

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[6] JHD, WX, XS, and JML gratefully acknowledge support from the Air Force Office of Scientific Research (Grant No. FA9550-14-1-0182). SDF and LJA acknowledge support under the Australian Research Councils Discovery Projects funding scheme (DP140102538 and DP110102228). JHD acknowledges the National Science Foundation Graduate Research Fellowship (Grant DGE-1252376). The authors acknowledge the Analytical Instrumentation Facility (AIF) at North Carolina State University, supported by the State of North Carolina and the National Science Foundation.

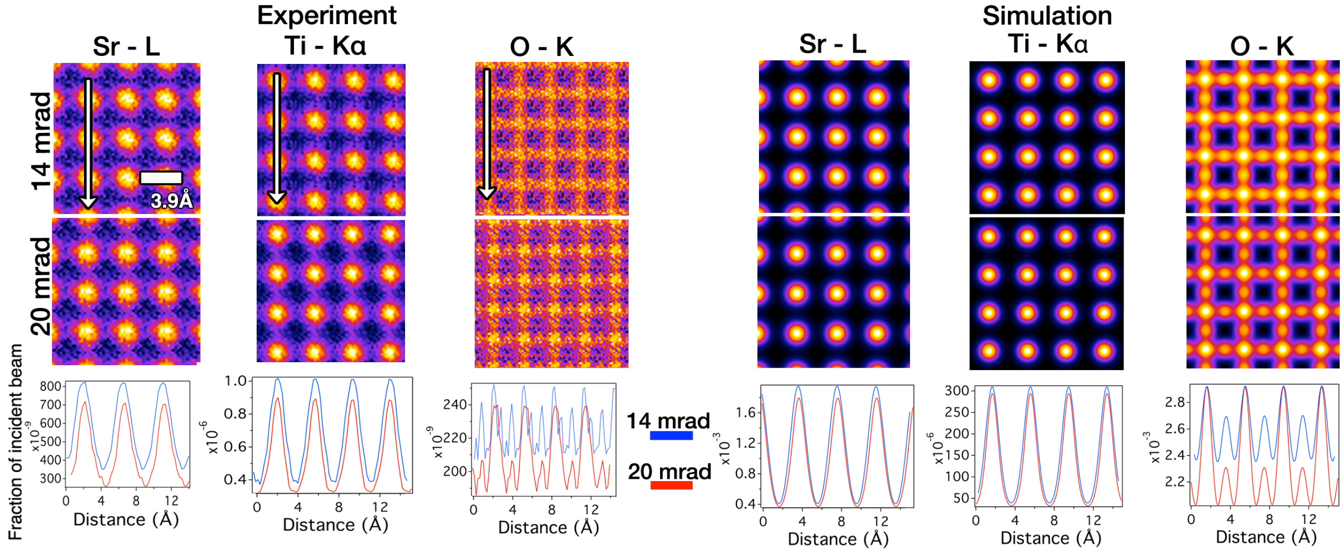

Figure 1. (a) Experimental EDXS lattice averaged maps replicated to a size of 4x4 unit cells. Line profiles display the intensity along atomic columns in the direction by the arrows. (b) Simulated EDXS maps with a 1 Å finite effective source size. Maps were filtered using a 'fire' colormap and no smoothing has been applied.

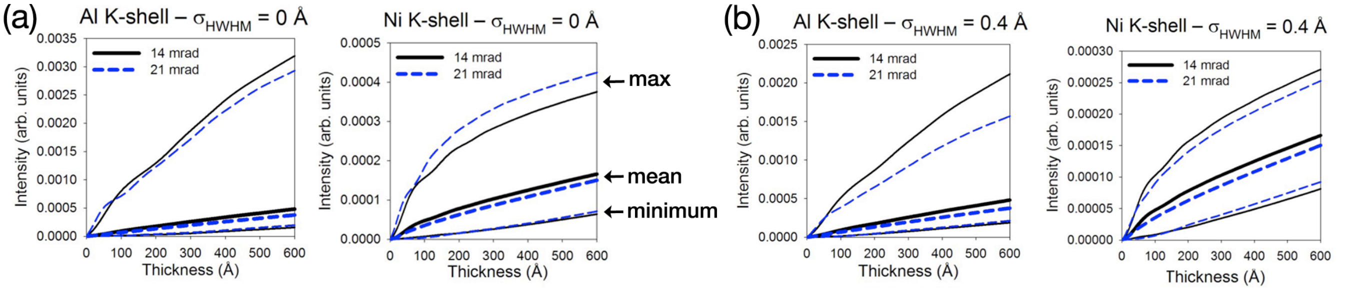

Figure 2. Simulated signal for $Ni_3Al_{<100>}$. (a) Maximum, mean, and minimum signals plotted without (a) and with (b) incorporating the finite effective source size.