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When will low-contrast features be visible in a STEM X-ray spectrum image?

Chad M. Parish[1]

[1.] Materials Science and Technology Division, Oak Ridge National Laboratory, Oak Ridge, TN USA

Under what circumstances will a low-contrast feature, such as a nanoparticle embedded in a foil prepared for transmission electron microscopy (TEM), be visible in an X-ray mapping experiment? This broad question does not have a general answer, but here I present a simplified model of oxide nanoclusters (NCs) embedded in a metallic matrix. This model allows an *a priori* prediction of what features will be visible under given experimental conditions (specimen structure, microscope X-ray collection efficiency, beam current and pixel dwell time, etc.). The ability to estimate what combination of X-ray collection efficiency (as solid angle Ω), probe current (i_b), and pixel dwell time (τ), is necessary for given features to become visible in an X-ray map.

First, NCs in the matrix are simulated (Fig. 1a) and their relative densities of Fe and Ti projected down the beam direction (Z-axis)(Figure 1b-c). Application of the fundamental X-ray detection equation [1] and parameters [2,3] allows calculation of the anticipated Fe and Ti X-ray maps (Fig. 2a-b), which can incorporate finite spot size and beam broadening [4]; no bremsstrahlung contribution is (yet) present in the calculation. DTSA-II [5] is used to calculate ideal $Fe_{85}Cr_{14}W_1$ and $Y_2Ti_2O_7$ spectra. By scaling the spectra relative to the specific Ti and Fe counts calculated from the X-ray detection equation, each individual pixel's spectrum can be calculated (Fig. 3). Because the time-consuming Monte Carlo X-ray simulation is only performed twice (for the idealized matrix and NC spectra), it is very computationally efficient to populate the entire spectrum image with individualize pixel spectra. Poisson noise is then used to produce the noisy point spectra; Fig. 3d). By generating full spectrum images (SIs), multivariate statistical analysis (MVSA) methods can be applied for datamining [6].

Comparisons to X-ray spectrum images taken on a Philips CM200, Hitachi HF3300, and FEI Titan G2 with ChemiSTEM are favorable when the input parameters to the model are well-matched to the experimental case. Fig. 5 compares Titan G2 data from simulation and experiment; the 4-detector SuperX system is well-suited to mapping small NCs embedded in a metallic matrix. Varying parameters such as SI size, probe current, pixel pitch, and pixel dwell time, all modify the visibility of the particles in a map, and are amenable to rapid screening by these calculations [7, 8].

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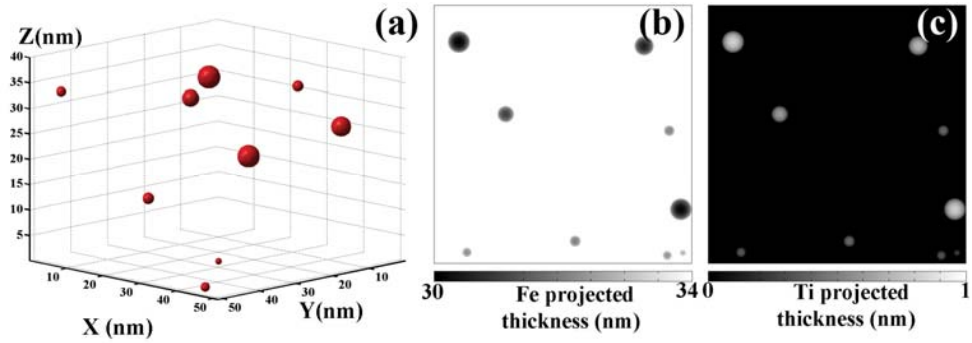

Fig. 1: (a) Simulated structure. Red are oxides, matrix invisible. (b-c) Z-projected thicknesses assuming $Fe_{85}Cr_{14}W_1$ and $Y_2Ti_2O_7$ stoichiometry, respectively.

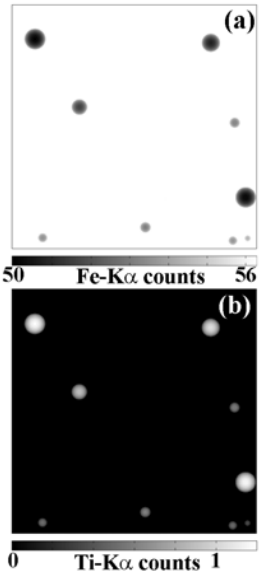

Fig. 2: Anticipated noise-free characteristic X-rays for (a) Fe (b) Ti.

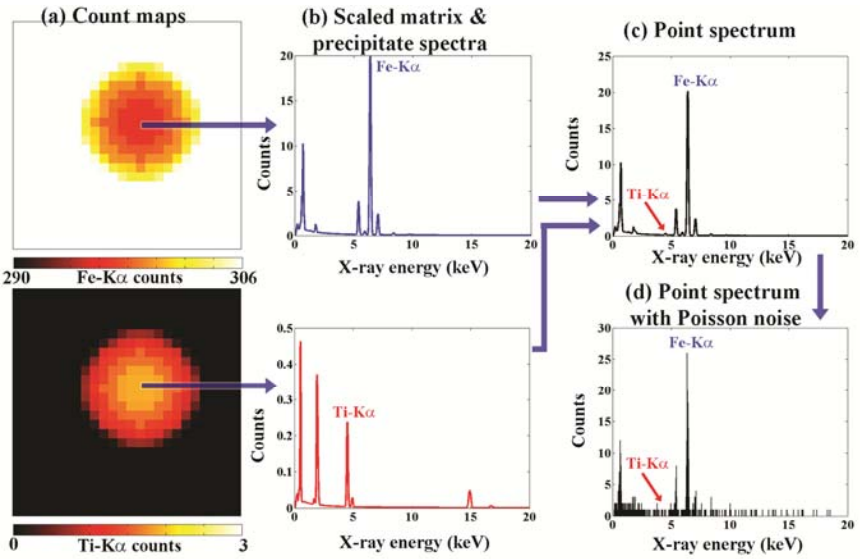

Fig. 3: (a) Details of simulated count maps. (b) Idealized DTSA-II calculated spectra for matrix and precipitates. (c) Summed noise-free point spectrum. (d) With Poisson noise added.

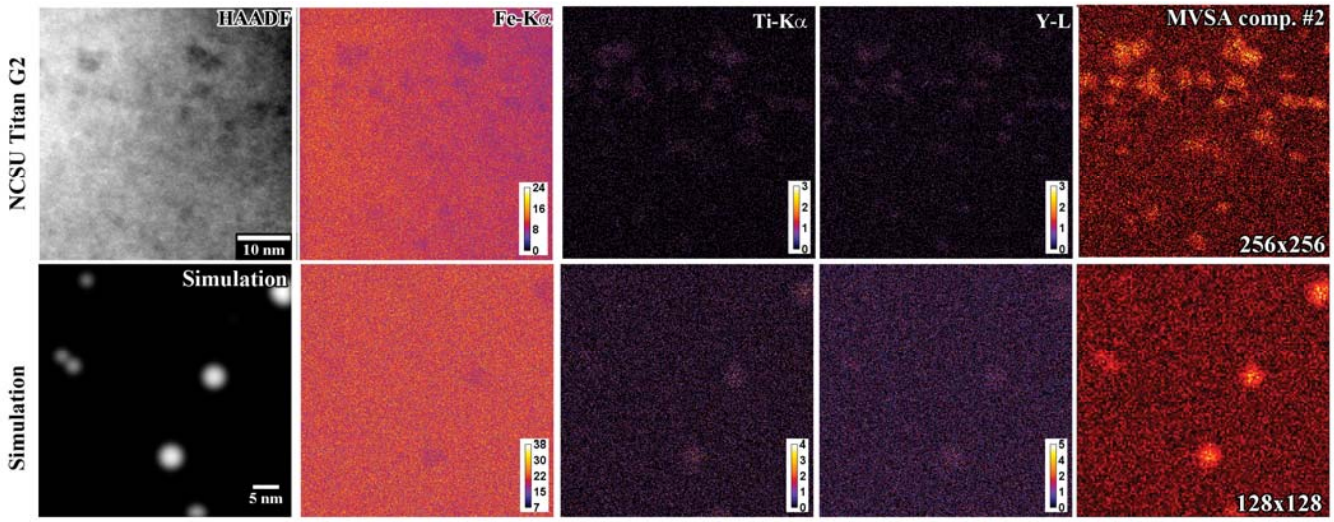

Fig. 4: Top row experiment (NCSU Titan G2 with ChemiSTEM) and bottom row simulation, showing similar signals and visibilities. MVSA comp#2 denotes MVSA score image for precipiates. Inset value (i.e., 256×256) is binned pixel size for MVSA of original 512×512 map.