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3D Chemical Imaging at the Nanoscale

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In addition to his well-known contributions to SEM and EPMA, Joseph Goldstein contributed significantly to the field of analytical electron microscopy (AEM), including early important work on the quantitative analysis of x-ray spectra in the scanning transmission electron microscope (STEM) [1]. In the last 30 years, AEM has advanced steadily via innovative electron optics, better spectroscopic detectors, improved computer control, and multi-modal data acquisition technology. The AEM analyst now has access to a wealth of high-quality, multidimensional data about the sample. As a result, the challenge now is to devise schemes to manage and exploit this flood of raw data and to synthesize the results into meaningful solutions to real-world problems. Recently, researchers have begun to explore the possibility of chemical tomography in the AEM, the determination of 3D elemental distributions in the sample based on tilt-series of energy-filtered TEM (EFTEM) elemental maps and high-angle annular dark field (HAADF) STEM structural data [2-5]. One of the chief barriers to this work is that for thicker samples EFTEM maps can show non-monotonic relationships between the observed signal in the 2D tilt images and the concentration of the analyte in that projected pixel. This violates the projection requirement, a key assumption in many 3D reconstruction algorithms. Because of this, most successful work to date has been limited to systems that (at least approximately) meet this requirement and do not exhibit the full complexity of interactions possible in the AEM. To get past this hurdle, it is necessary to construct new models for 3D reconstruction in the AEM that are not based on x-ray tomography predecessors and that explicitly account for effects such as beam spreading, multiple scattering, and through-sample self-absorption of the type described in the early work of Joe Goldstein [1].

Following the lead of the medical imaging community, one useful step towards the creation of effective 3D reconstruction algorithms is the analysis of synthetic datasets of phantoms with known properties. Figures 1, 2, and 3 show the results from 3D Monte Carlo simulations of three cylinders (Cu, Al, and SiO_2), each 600 nm in diameter and 1 μm long [6]. The electron beam (purple line) was rastered over 32 pixels parallel to the x-axis and the resulting x-ray spectra at the XEDS detector were calculated, resulting in spectrum-line profiles for each tilt from 0° to 180°. From the 5,760 spectra calculated, chemical sinograms were extracted by summing the intensities in 130 eV wide windows over the Cu Kα, O Kα, and Al Kα peaks and displaying them as the channels of an RGB image (Figure 4). These data display both beam broadening and self-absorption effects, but do not include Bragg scattering or dynamical interactions, two other effects that plague 3D AEM.

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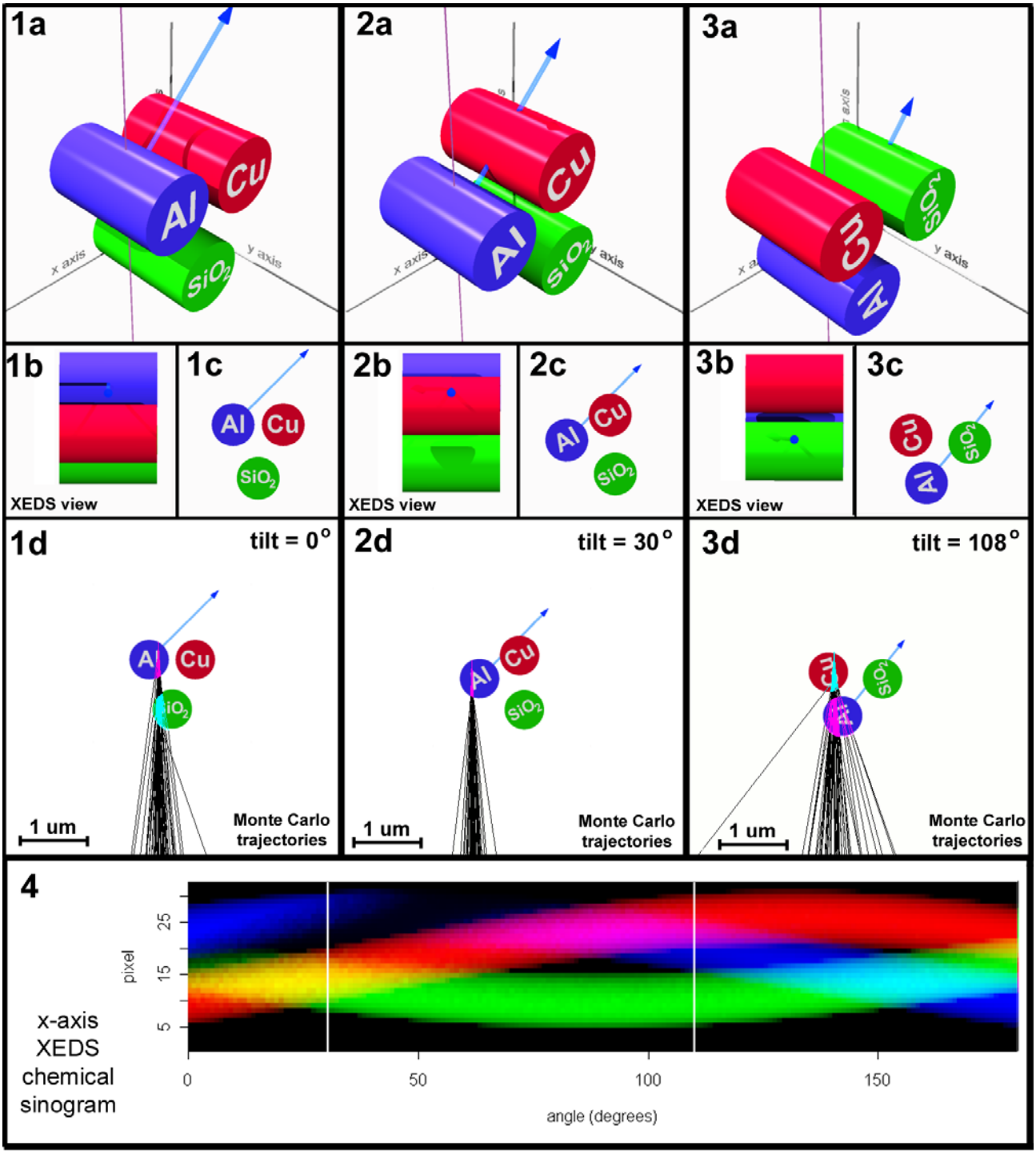

Fig 1.(a) 0° tilt perspective view of AEM cylinder phantoms (600 nm diameter by 1 μm long); blue arrow points to XEDS detector. **(b)** same phantoms as seen by XEDS detector. **(c)** view along tilt axis. **(d)** 300 keV Monte Carlo electron trajectories. 100 electrons displayed at 1 of 32 horizontal beam raster locations. **Fig 2 & 3.** same as Fig 1 but sample tilted to 30° and 108° respectively. **Fig. 4.** "Chemical sinogram" displaying XEDS line intensities vs. sample tilt angle (horizontal axis) and x-axis beam raster position (vertical axis). Red is Cu Kα, green is O Kα, and blue is Al Kα. Vertical white lines denote 30° and 108°. Note attenuation of Al Kα by Cu cylinder near 30° tilt.

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