

## Focused Ion Beam Slice-and-View Tomography and Correlative Electron Microscopy of Multiphase Meteorite Particles

N.D. Bassim<sup>1</sup>, R. M. Stroud<sup>1</sup>, K. Scott<sup>2</sup>, L. R. Nittler<sup>3</sup> and C. D. K. Herd<sup>4</sup>.

<sup>1</sup>Naval Research Laboratory, Code 6366, 4555 Overlook Ave. SW, Washington, DC 20375, USA

<sup>2</sup>National Institute of Standards and Technology, Gaithersburg, MD 20899, USA.

<sup>3</sup>Department of Terrestrial Magnetism, Carnegie Institution of Washington, 5241 Broad Branch Rd. NW, Washington, DC 20015, USA.

<sup>4</sup>University of Alberta, Department of Earth & Atmospheric Science, Edmonton, AB T6G 2E3, Canada.

Focused Ion Beam slice-and-view tomography is a useful tool for characterizing nano-to-meso length scales in materials. To date, this length-scale characterization has been employed in geological materials in order to understand pore volume and distribution for such applications as hydraulic fracturing. It may be applied as well for the fine-grained mineralogy of primitive meteorites, in which the feature size of interest is smaller than that accessible via synchrotron-based X-ray methods. The 3-dimensional distribution of organic matter, nanoscale minerals and pore space can provide insight into the formation temperatures, accretion characteristics in the protoplanetary disc, and the extent of hydrothermal alteration during its history. In particular, we seek to observe primitive organic matter and determine any microstructural context for their formation. These carbonaceous materials often form in spherical shapes (known as nanoglobules) and primarily have a sub- $\mu\text{m}$  diameter. Nanoscale metal-rich inclusions, such as Fe-Ni sulfides can also be studied for agglomeration due to hydration.

A tomogram of material from the Tagish Lake (TL) 5b meteorite sample was chosen for the initial study because TL 5b contains abundant nanoglobules, and its powdery nature enables easy extraction of suitably-sized matrix fragments. We selected 10 to 50  $\mu\text{m}$  matrix fragments and attached them either to a silicon support wafer or an Omniprobe grid (to reduce redeposition and enable easy TEM liftout) with crystal bond adhesive. Slice and View tomograms were obtained using an FEI Nova 600 FIB-SEM with 50 nm thick slices, usually with about 100 slices. SEM images were acquired at 5keV in backscatter detector mode for Z-contrast within the sample. The acquired images were then aligned using a cross-correlation algorithm in ImageJ software and the image stacks were cropped, corrected for tilt, segmented and visualized using Avizo Fire software. After slice-and-view, EDS maps of the exposed faces were obtained, and, in some cases, a 1  $\mu\text{m}$  thick section was extracted by in situ lift-out and thinned to electron transparency for high-resolution TEM examination on a JEOL 2200FS field emission scanning transmission electron microscope.

Figure 1 is a plan view SEM image of the test particle with five cross-sectional images from a tomographic dataset at 1  $\mu\text{m}$  intervals. Some redeposition of sputtered material from the silicon support and carbon protective mask is visible, most notably in the first image of the series. Despite the redeposition artifacts, we clearly observe the evolution of internal pores, as well as large ( $\mu\text{m}$ -sized) and small (20-50nm) Fe-rich grains. We also observed diffuse local variation in the image contrast (bright charging effects), which likely reflects variation in the water content of phyllosilicate phases. This contrast changes with continued imaging, as the water is driven off. This charging also induces image artifacts and makes segmentation more problematic.

Figure 2 shows a specific, extracted image slices that contains two types of carbonaceous nanoglobules: solid and hollow-cored. It also shows charging effects within several grains, around a high-z coarse grain, which may be sulfonated Fe-Ni inclusions. Figure 3 is a TEM montage correlated with the backscattered SEM image to identify phases. Dark field STEM imaging clearly reveals the presence of nanoscale sulfides, in addition to the large Fe-rich grains observed by SEM BSE imaging of the section face. The fibrous structure of the phyllosilicate phase which dominates this particle and interior nanoparticle phases are visible in bright field TEM images, and the composition of individual phases can be confirmed with EDS.

The proof-of-concept work presented here is the first step towards obtaining a full 3D rendering of the phases in fine-grained matrix. Since Tagish Lake is a highly-altered meteorite, we can place microstructural context of the nanoglobules and sulfide size and distribution as compared to less altered meteorites. We plan studies of additional matrix samples to examine the spatial relationships between minerals and organic matter.

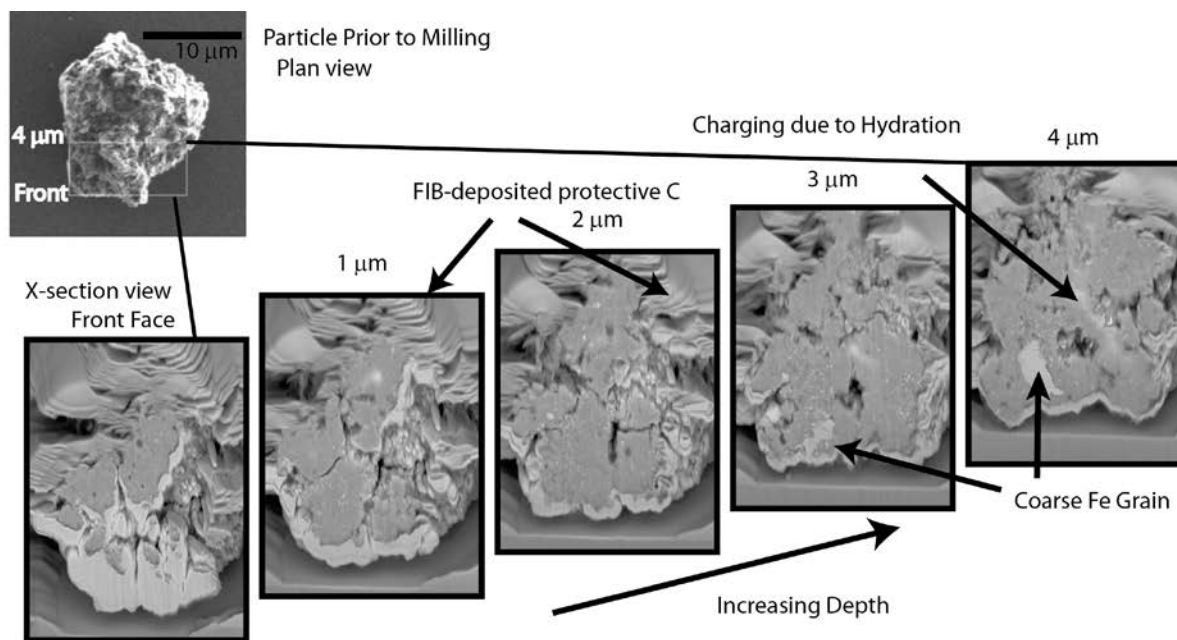


Figure 1. Extracted BSE images from a focused ion beam (FIB) slice-and-view tomogram series at milling depths of 0 to 4 μm. The emergence of internal void space and a Fe-rich grain are visible in the series. The images are corrected for the 52° imaging angle with respect to the milled face.

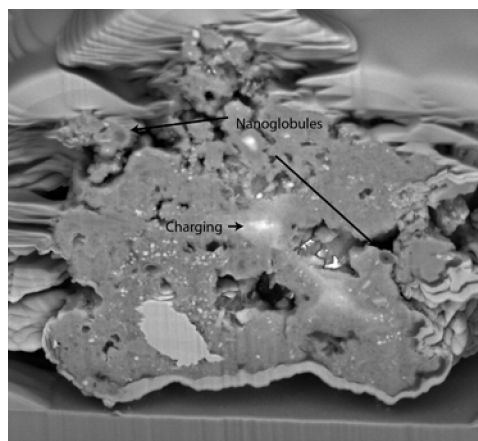


Figure 2. Extracted Image Slice that contains two types of carbonaceous nanoglobules: solid and hollow. Also, charging due to hydration surrounding a high-Z phase is present. Image is not corrected for tilt and is 15 μm on across.

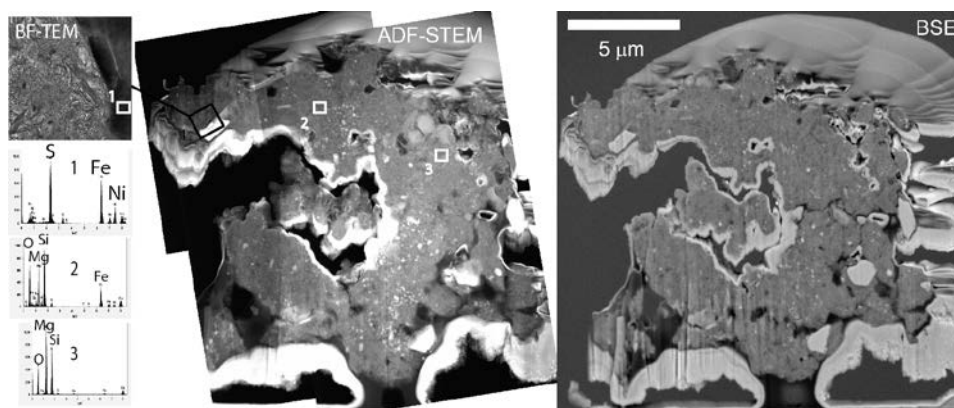


Figure 3. Correlation of the BSE image of the terminal face of the tomographic series (right) with dark field STEM (middle), STEM-EDS and TEM (left) analyses of the final thinned section. White boxes indicate the areas of EDS collection.