

3D Nanoscale Analysis Using Focused Ion Beam Tomography of Carbonaceous Nanoglobules in Matrix Materials from the Tagish Lake Meteorite

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Focused Ion Beam (FIB) slice-and-image tomography enables imaging of carbonaceous nanoglobule distributions in meteorites at a high resolution [1]. This method permits accurate determination of pore volume, atomic number contrast imaging of individual constituent phases and their volume fractions, and spatial correlations among phases. These parameters are important for understanding the formation and processing histories of the Tagish Lake meteorite and its constituent phases. In particular, we seek to extend the FIB tomography method to a resolution of 10 nm or smaller voxel dimension, to improve our understanding of the distribution of the carbonaceous nanoglobules and their origins.

Matrix material from the Tagish Lake (TL) 5b sample described in [2] was chosen for the initial study because TL 5b contains abundant nanoglobules. We selected 10 to 50 μm matrix fragments and attached them to a silicon support wafer by overcoating with a ~ 10 nm layer of sputtered Au. The samples were then examined in a Helios FIB-SEM at NIST.

We acquired a slice-and-image tomographic dataset with a nominal 10 nm slice resolution, as measured with top-down fiducial alignment marks. The image series was obtained in backscatter electron mode (BSE, 2 keV) to provide atomic number sensitivity. The set consisted of ~ 1000 images with voxel resolution of $5.6 \text{ nm} \times 7.1 \text{ nm} \times 10 \text{ nm}$, sampling 10 μm depth. Images from a 2 μm section of that volume were segmented for quantitative analysis. Figure 1 shows a BSE image from a grain on the exposed particle face at 10 μm depth.

The dataset segmentation was primarily targeted towards examining the morphology and contiguity of the carbon present in the matrix. In order to classify the data, the images were thresholded to a contrast level that corresponded to carbon BSE signal. The features were then segmented manually as the backs of pore volume often had the same contrast level as the carbon at the surface of the milling front. Segmentation into multiple morphological categories was possible: individual hollow, solid, and agglomerated nanoglobules interspersed in the silicate matrix were each identified. In addition, a large, distinct volume of carbonaceous material that appears to be a separate clast in the matrix, was also identified (Figure 2).

One carbonaceous particle, indicated with * in Fig. 2, shows the appearance of a mineral filling a globule. However, three-dimensional reconstruction (shown in Fig. 2b), shows that the feature is in fact not a globule filled with silicate but rather a bunch of solid globules that had agglomerated in a rounded fashion.

Segmentation of the nanoglobules shows that the majority of the globules are solid, rather than hollow. Figure 3 compares the size distribution of the hollow and solid type. The lack of internal minerals in the

nanoglobules indicates that they are formed by processes other than as icy rinds on interstellar silicate grains.

References:

[1] Stroud R. M. et al. (2013) Proceedings of the 44th Lunar and Planetary Science Conference, Abstract #1719. [2] Herd C. D. K. et al. (2011) *Science* 332:1304-1307

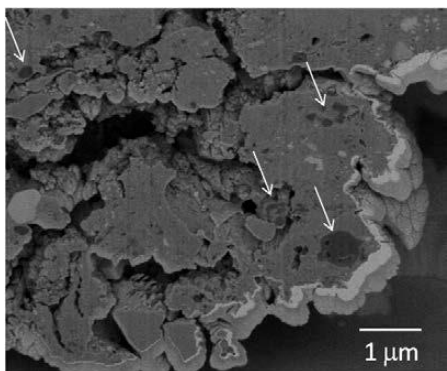


Figure 1. Example BSE image acquired during the tomographic series. These images are uncorrected for the 52° imaging angle with respect to the milled face. Arrows point to nanoglobule features.

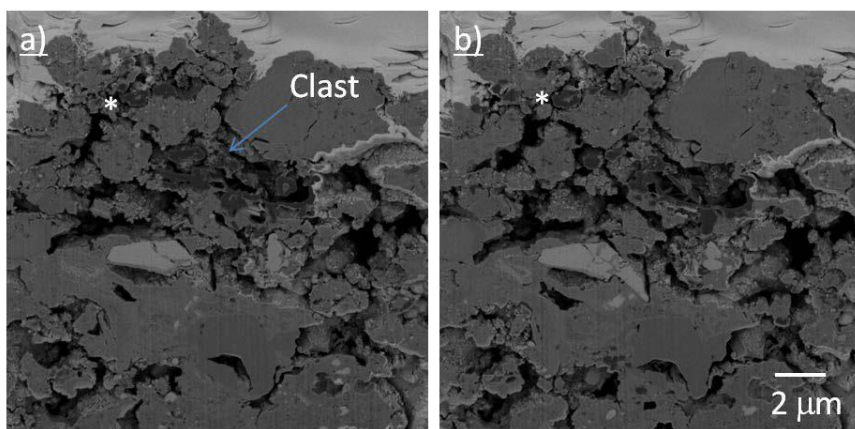


Figure 2. Extracted BSE images from a focused ion beam (FIB) slice and image tomogram series separated by a depth of 160 nm. Agglomerated solid nanoglobules and a large carbonaceous vein or clast is visible in these sections.

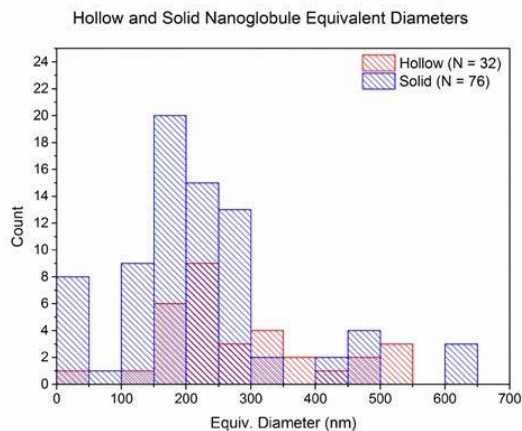


Figure 3: Histogram of nanoglobule equivalent diameter, classified by morphology. This graph is evidence of a much higher proportion of hollow to solid nanoglobules, as well as a slightly higher average equivalent diameter of hollow nanoglobules.