CROSS-SECTIONAL ANALYSIS OF CARBONACEOUS NANOGLOBULES FROM THE TAGISH LAKE (C2) METEORITE. Laurence A.J. GARVIE,[1], Grant BAUMGARDNER[2], and Peter R. BUSECK[1,3], [1]School of Earth and Space Exploration, Arizona State University, Tempe, Arizona 85287-1404, USA. [2]School of Materials, Arizona State University, Tempe, AZ 85287-1704, [3]Department of Chemistry and Biochemistry, Arizona State University, Tempe, AZ 85287-1604, USA.

Introduction: Detailed knowledge of the physical and chemical properties of meteorite minerals is key to understanding the formation conditions of meteorites. The matrix of the carbonaceous chondrite (CC) meteorites is particularly intriguing since it is a fine-grained heterogeneous mixture including low temperature hydrous components, organic materials, and presolar and refractory grains. Determining the structures and compositions of the materials in the CC matrix necessitates the use of high spatial resolution analytical instruments such as scanning and transmission electron microscopes (SEM and TEM).

SEM with a field-emission gun (FEG) provides high brightness and spatial resolution of 1 to 10 nm. When combined with a focused ion beam (FIB) source, the FIB-SEM provides high spatial resolution with unique sample preparation capabilities. Arguably the most important application of the FIB-SEM methodology is the preparation of TEM samples for the semiconductor industry. The utility of the FIB-SEM technique for TEM sample preparation is especially advantageous for heterogeneous samples with widely differing physical properties.

Previous TEM work of the carbonaceous nanoglobules in CC meteorites indicates that many are vesicular or hollow [1-5]. Here we use FIB-SEM to provide a clearer understanding of the shapes and internal structures of carbonaceous nanoglobules from the Tagish Lake (C2) meteorite.

Materials and Methods. Several millimeter-sized pieces of the Tagish Lake meteorite were digested in HCl and HF in order to concentrate the carbonaceous materials. Previous TEM and EELS investigations show the residue is dominantly carbonaceous, with smaller amounts of S, N, and O [1, 2]. The SEM samples were prepared by adding small quantities of residue onto a TEM grid that supports a thin film of lacey C.

The TEM grid containing the carbonaceous residue was attached to an Al-SEM stub. Images were acquired with a Nova 200 NanoLab DualBeam FIB/SEM. The sample was imaged without coating. No significant charging was observed. Images were acquired with working distances between 0.5 and 5.4 mm. The latter distance was used when ion-beam thinning since this distance ensured that the areas imaged are at the eucentric height for both the electron and ion columns. Secondary-electron images were acquired with accelerating voltages and currents of 500V and 70 fA, 1 kV and 1.4 pA, and 5 kV and 98 pA, respectively. Use of low accelerating voltages reduces electron beam penetration depth, with concomitant enhancement of surface features [6]. The 500V and 1kV images were acquired with the down-hole visibility feature of the secondary electron detector.

Selected particles were milled with the Ga ion beam. Following secondary electron imaging, the sample was tilted 52° so that the sample plane was normal to the ion column. The Ga beam rapidly destroys the carbonaceous nanoglobules so focusing and astigmatism corrections were performed near the globule to be sectioned. The sample was then moved so that the globule was centered on the viewing screen, and a rectangular milling mask was set for the particle. The particles were milled at 30 kV with a 10 pA Ga beam.

Results and Discussion: Discrete particles with rounded morphologies are clearly visible and readily distinguishable from the bulk formless carbonaceous material. Upon first imaging the particles in the SEM, there is a rapid shrinkage with a decrease in their diameter by ca. 5%, although without visible changes to the overall shape or surface architecture. Following the shrinkage the particles are stable in the electron beam. We speculate that this shrinkage is associated with H loss.

The interiors of the particles are readily visible after milling with the FIB (Fig. 1). The top surface of the nanoglobules facing the Ga beam is slightly rounded following the milling and brighter in SE mode, presumably as a result of minor Ga implantation. The lower surface of the cut spheres show sharper features than the upper surfaces (Fig. 1d), presumably since the upper part of the shell acts as a mask to the Ga beam. There was no evidence of milled material depositing in the hollow spheres. The walls of the nanoglobules are featureless and lack entrained mineral grains and voids, consistent with the TEM observations [1-3].

The sectioned globules range from hollow to solid, although solid globules are rare in the Tagish Lake meteorite. The nanoglobules imaged using TEM commonly show vesicular cores (e.g., Fig. 1b in [2]). After sectioning, these particles show a central open core with adjoining smaller cores. These vesicular particles show a different internal structure from similar sized hollow latex particles [7].

FIB with an SEM is a valuable tool for the analysis of extraterrestrial materials, even of sub-micron-sized “soft” carbonaceous particles. The rapid site-specific cross-sectioning capabilities of the FIB allow the preservation of the internal morphology of the nanoglobules, with minimal damage or alteration of the unsectioned areas.

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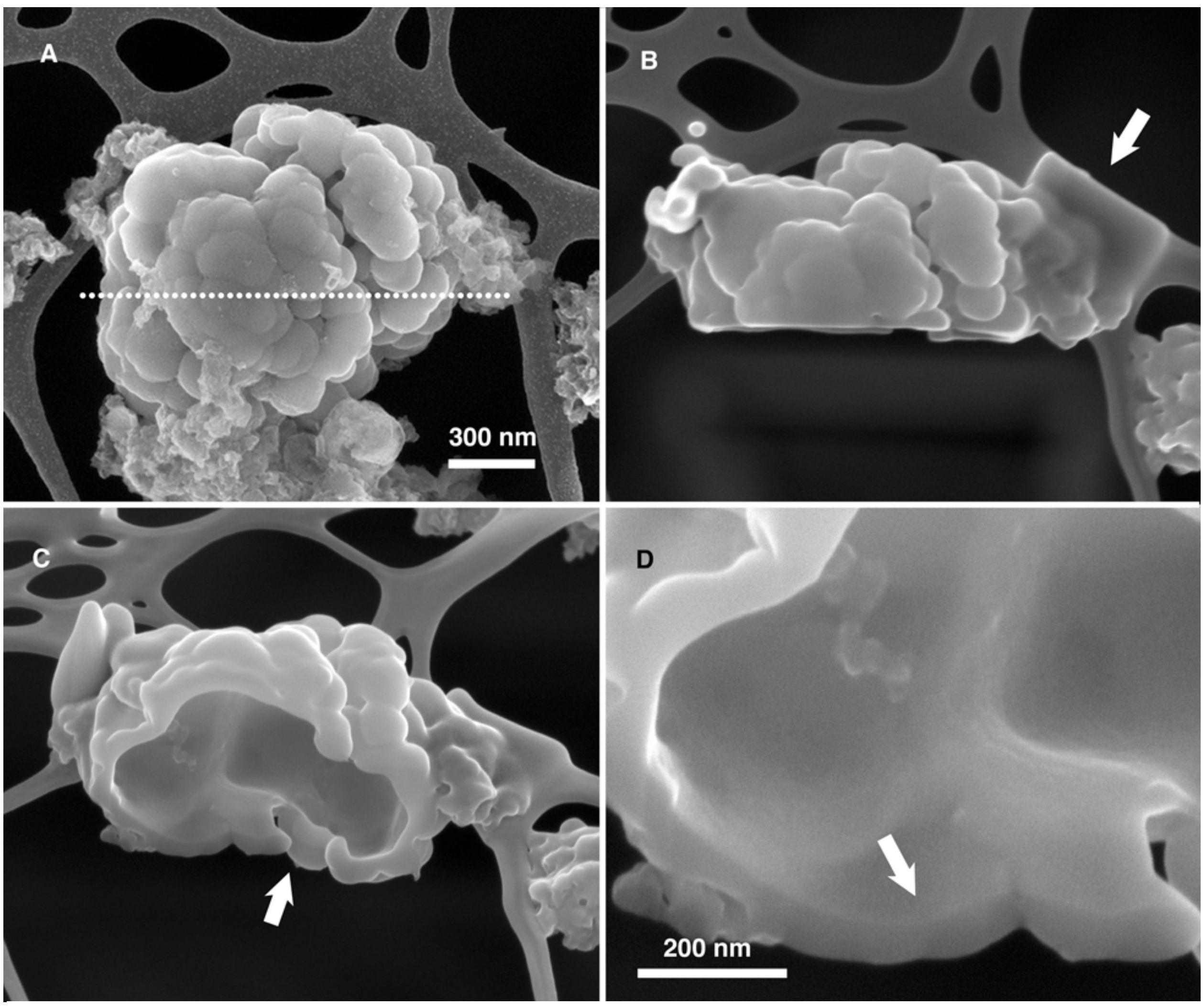

Fig. 1. SEM image (SE mode) of a large nanoglobule supported on a lacey C TEM grid (A). The dotted line shows where the globule was sectioned. B) Milled globule. Arrowed square area shows where Pt was deposited between the globule and the supporting film to prevent the particle falling away from the TEM grid during milling. C) Particle tilted 25° showing the hollow interior. The arrow points to an opening in the globule wall. D) Close up showing the details of the interior and globule wall.