UNLOCKING THE NANOSCALE FLUFFY STRUCTURE IN INTERPLANETARY DUST WITH HARD X-RAY PHASE CONTRAST NANOTOMOGRAPHY. Z. W. Hu[1] and R. Winarski[2], [1]XNano Sciences Inc., P. O. Box 12852, Huntsville, AL 35815, USA (zwhu@xnano.org). [2]Center for Nanoscale Materials, Argonne National Laboratory, Argonne, IL 60439, USA.

Introduction: Interplanetary dust particles (IDPs) collected from the stratosphere [1] are the least expensive extraterrestrial samples, yet many of them such as chondritic porous (CP) IDPs are the most primitive extraterrestrial materials currently available for laboratory study [2-5]. CP IDPs are believed to be of cometary origin based on their porosity, ultrafine structure, mineralogy, and compositions, with an abundance of surviving presolar interstellar material preserved [2-5]. Hence, IDPs provide an excellent window into the structure and properties of interstellar grains, interstellar processes, and the origin and evolution of their primitive parent bodies and the solar system. Much of our knowledge about the early solar system has come from the laboratory analysis of the mineralogy, chemistry, and isotopic compositions of IDPs.

Complementary to analytical tools used for characterizing microtomed thin sections of IDPs minerallogically and compositionally, nondestructive analytical techniques have been employed or are being developed to analyze IDPs, including synchrotron X-ray microtomography to visualize the internal structure of bulk IDPs [6]. Given that many IDPs are delicately-bound and texturally-rich porous aggregates of predominantly submicron or nanoscale grains, applying nondestructive three-dimensional (3D) imaging techniques with high resolution and high sensitivity may be essential to extracting both the fine structure of tiny interstellar grains and the integral structure of IDPs morphologically and textually. Moreover, some indigenous or diagnostic features preserved in cometray dust may be subtle and easily altered by destructive processing. The fluffy structure on a submicron level has been widely suggested to be a characteristic of interstellar material and cometary dust based on spectroscopic data [7-8], although the degree of surviving interstellar grains may vary in different comets and locations. 3D mapping of the structure of intact IDPs at the nanoscale may provide additional constraints on the sources of IDPs and the origin and evolution of comets and asteroids.

We have been exploiting hard X-ray phase contrast nanotomographic imaging capabilities to nondestructively map the original structure and properties of intact IDPs in 3D nanoscale detail. In this abstract we briefly present results obtained from the initial X-ray imaging experiment on interplanetary dust and discuss their potential implications and a potentially new opportunity for extraterrestrial materials research.

Sample and Experimental Methods: An irregularly-shaped micron-scale particle from L2036B1, which is related to particle L2036B2 from a small cluster IDP L2036#7, was selected for nondestructive examination. The particle was mounted on top of a needle nano-tip, which was then positioned into a nanotomography sample holder for subsequent alignment and measurement. The imaging experiment was performed with monochromatic coherent X-rays at 10 keV under a full-field transmission mode at the Center for Nanoscale Materials (CNM)/XOR Hard X-ray Nanoprobe Beamline (26-ID) at the Advanced Photon Source (APS), Argonne National Laboratory [9]. A capillary with high focusing efficiency was used as the condenser to focus the X-rays onto the sample. A high-resolution Fresnel zone plate was employed as the objective lens to magnify the projection images of the sample, which, combined with a lens-coupled CCD imaging system positioned downstream from the sample, results in an effective pixel size of 15×15 nm^2 on recorded images. A Zernike phase ring was placed on the back focal plane of the zone plate for phase contrast imaging. The projection images were taken at an angular interval of 0.1^{o} over a scan range of 180^{o}, from which three-dimensional (3D) tomographic images were then reconstructed. Positional shifts due to rotational wobble and vertical drift were corrected prior to image reconstruction. Reconstructed 3D images have a voxel size of $15\times15\times15$ nm^3, yielding an effective spatial resolution of 30 nm or better. A large depth of focus provided by the hard X-ray microscope used, > 18 μm in the present case, combined with the high penetrating power of the chosen hard X-rays, makes the particle well suited for a challenging 30-nm-resolution phase contrast tomographic imaging experiment.

Results and Discussions: The small particle, with its seemingly rather smooth outward appearance under a scanning electron microscope, is a nanoporous, nanograined aggregate with rich, well-defined textures throughout. It contains two morphologically different porous "segments", with "component 1" (C1, Fig. 1) being ribbonlike and extraordinarily fluffy at the nanoscale. This fragile, fluffy component, hardly visible without invoking phase contrast, is approximately 1.5 μm long, 0.6 μm wide, and 0.1 μm thick, consisting of a bunch of extremely porous sub-aggregates of ~ 50-100 nm grains (grainy features in C1, Fig. 1). The nanograins themselves appear to form the boundaries of the similarly sized pores of 200-300 nm in diameter. These nanograins are irregularly shaped but tend to be elongated, probably belonging to amorphous silicates like those present throughout “component 2” (e.g., brighter features G in C2, Fig. 1). That these seemingly discrete nanograins (at first glance) are held together implies that there is material lighter than silicates, more likely organic matter as observed on grains in ultramicrotomed CP IDPs [10], serving as the glue for holding together such a sophisticated porous aggregate (C1, Fig.1), although small voids on the scale of tens of nm exist among the nanograins. Indeed, examination tends to show that the nanograins are mantled, with a brighter core-fainter mantle structure, e.g., a silicate core of ~ 50 nm in diameter or width and a 30-40 nm thick mantle shown in Fig. 2. The observed features are in favor of the view that the refractory organic mantle results from the irradiation induced alteration of carbon-bearing ices that were initially condensed on interstellar grain surfaces [11-12]. The core-mantle structure appears uneven in general. But the averaged size of a core-mantle structure is quite close to that proposed nevertheless [7, 11].

The examined small particle has survived intact during atmospheric entry as indicated by the indigenous textures preserved throughout [13]. The revealed 3D structural details of the extremely fluffy, sub-100-nm -100-nm grain aggregate provides evidence that the dust particle more likely came from a comet and the amorphous nanograins are of interstellar origin. There are several possible scenarios for the formation of such nanoporous structure (C1, Fig.1): (1) The nanoporous, nanograined aggregate was formed in the interstellar medium and was subsequently incorporated into its parent cometary body. (2) It occurred or developed later, i.e., before and/or after interstellar grains were incorporated into the parent body. While the initial results have provided significant clues, further study would allow us to elucidate the exact nature of such fluffy structure and its formation processes. Hard X-ray phase contrast nanotomography, complementary to other analytical tools, is opening up a new opportunity to examine various processes or hypotheses concerning interstellar grains, ices and nonvolatile dust aggregation, and primitive bodies to help us better understand the origin and evolution of the solar system.

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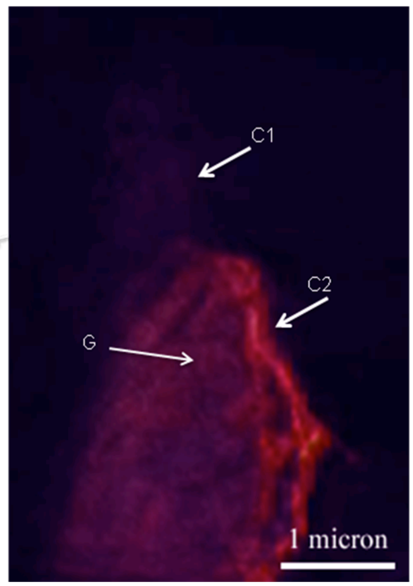

Fig. 1. Phase contrast X-ray 3D image of an intact IDP revealing an unusually fluffy structure at the nanoscale (C1). See text for detail.

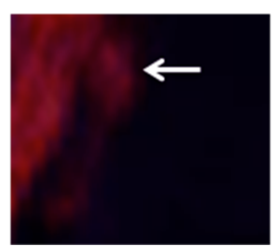

Fig. 2. A further enlarged phase contrast 3D nanoimage showing nanograins with a brighter core-fainter mantle structure (arrowed).