

**THREE-DIMENSIONAL VISUALIZATION OF POROUS STRUCTURE OF A CLUSTER INTERPLANETARY DUST PARTICLE.** Z. W. Hu<sup>1</sup> and R. Winarski<sup>2</sup>, <sup>1</sup>XNano Sciences Inc., 8401 Whitesburg Dr. # 12852, Huntsville, AL 35802, USA (zwhu@xnano.org). <sup>2</sup>Center for Nanoscale Materials, Argonne National Laboratory, Argonne, IL 60439, USA.

**Introduction:** Sets of interplanetary dust particles (IDPs) collected from the stratosphere provide samples of parent bodies and/or astromaterials unlikely available in collections of conventional meteorites [1-3]. Several techniques have been used to measure or infer the bulk density of IDPs [3-6], an important property that may have important implications for the origin of IDPs. These measurements [3-6] have shown that the density varied from one particle to another, particularly with IDPs probably from different sources, with low average density values of 0.6-1.0 g/cm<sup>3</sup> for typical IDPs of likely cometary origins compared with a high mean density value of 3.3 g/cm<sup>3</sup> for dust particles likely derived from asteroids [6].

Chondritic IDPs with low bulk density may well have relatively high porosity or a combination of abundances of pore spaces and low-density components (e.g., carbonaceous material/organic matter). Void spaces are frequently exhibited in TEM images of microtomed thin sections of aggregate IDPs. However, much remains unknown concerning three-dimensional (3D) details of porosities of IDPs, including the morphology and types of pores, how they are distributed throughout individual particles, and whether and to what extent pore spaces are connected to one another. Elucidating these questions are of importance to better understanding the formation and evolution of IDPs and their parent bodies as well as the origin and history of pores in IDPs. Given that typical IDPs are fragile aggregates of submicron and nanoscale grains, we have been developing and applying an X-ray phase contrast nanotomography approach to nondestructively characterizing IDPs, aimed at mapping the porous structure and spatial distributions of grains and other features of interest in intact IDPs in 3D nanoscale detail by taking advantage of its high resolution and high sensitivity.

**Principles and Methods:** X-ray imaging with phase contrast is utilized to enhance imaging sensitivity to features of interest including pores and grains. The interaction of X-rays with a heterogeneous structure can be described by a complex index of refraction  $n$ ,  $n=1-\delta-i\beta$ , where  $\delta$  and  $\beta$  are the refraction (phase shift) and absorption terms, respectively. Phase contrast imaging, unlike the conventional X-ray imaging technique relying solely on absorption, utilizes the refraction or phase shift of the transmitted X-rays,

which occurs upon X-rays travelling across interfaces that define features of interest such as pores, mineral grains and cracks. It transforms the phase modulation into intensity variations through wave interference, as a result, enhancing image contrast, for example, by edge enhancement (Fig. 1). Moreover, for light-element materials (e.g., carbon material/organic matter), the phase shift term  $\delta$  is dominant in the hard X-ray regime, say, on the order of  $10^{-6}$ – $10^{-7}$  for  $\delta$  compared with  $10^{-9}$ – $10^{-10}$  for  $\beta$ . Hence, X-ray phase contrast imaging allows pores and individual grains throughout a particle to be mapped in more detail, making it possible to detect subtle structurally/compositionally related features that may otherwise be difficult to resolve with absorption contrast alone.

Advanced X-ray optics and a lens-coupled CCD system were combined, which allowed of resolving details of  $\sim 10$  nm under favorable conditions. The sample was carefully mounted on the top of a tungsten pin with Scotch double-sided tape such that the entire volume of a particle can be scanned for tomographic imaging while high resolution was maintained. Prior to being reconstructed, transmission images were manually aligned by taking positional shifts into account, which occurred during data acquisition.

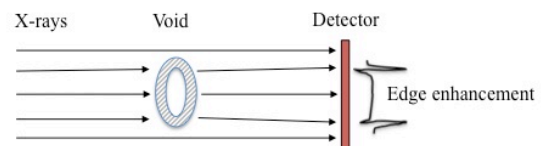


Fig. 1. A varying refractive index across an X-ray wavefront at a void interface results in a slight change in the direction of wave propagation, which provides the basis for phase contrast imaging of the void.

**Results & Discussions:** A cluster IDP of  $\sim 10$   $\mu\text{m}$  in length, U2015-M-1, was selected for examination. This elongated chondritic particle has a bulky middle and two somewhat tapered ends (Fig. 2). Part of the interior of the particle, boxed with green markers (Fig. 2), is highlighted in Fig. 3, which appears to be composed of extremely-irregular, submicron domains or grain clusters that contain embedded mineral grains

(brighter grainy features with typical sizes of  $\sim 15\text{-}50$  nm) and smaller nanoscale-diameter pits or intra-cluster pores (darker features), together with much larger submicron pore spaces present between the clusters. The domains or clusters are morphologically consistent with carbonaceous material. Moreover, the submicron pores are in effect connected to one another, forming like a fairy-castle maze in 3D space, which, together with the irregular submicron clusters, tends to suggest that the particle constitutes sophisticated, fractal-like structures. It is noted that the fractal aggregate model of cometary nuclei was proposed to account for the observed properties of comets [7].

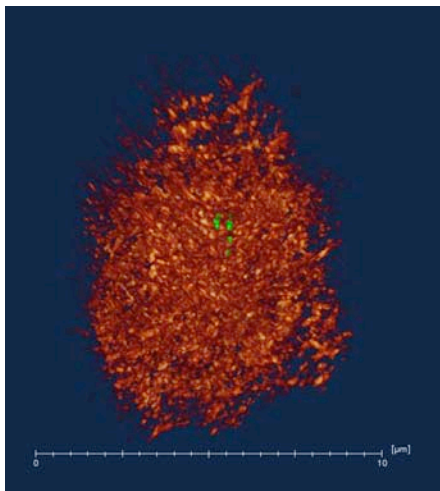


Fig. 2. 3D rendering of the reconstructed volume of an entire cluster IDP from an X-ray phase contrast nano-tomographic data set.

In two-dimensional (2D) settings, void spaces, particularly larger ones, are also discernable, but it is difficult to appreciate the essence of porous structure as well as the spatial distributions of void spaces, not to mention loss of the porous details both morphologically and texturally, a likely key piece of information on the history of the pores and particle. The maze-like porous structure adds an extra challenge to accurate porosity measurement. Both 2D and 3D preliminary analysis, nevertheless, gives an average porosity estimate of  $\sim 50\%$ . No cracks have thus far been detected inside pore walls, which may imply that no appreciable compaction or density alterations occurred from atmospheric deceleration and sample collection. The achieved imaging capabilities permit examination of potential features of interest throughout the entire volume of the particle, including amorphous radiation-damaged rims as well as magnetite rims arising from

atmospheric entry heating and cracks or damage caused by the aerodynamic and impact forces [3]. Analysis is ongoing, progress in this aspect will be updated.

**Summary:** Porous structure is an essential part of the intriguing story of IDPs. X-ray phase contrast nano-tomographic imaging of an intact cluster IDP reveals a small porous world that appears to be structurally more complex and texturally richer than thought. The observation provides information complementary to other studies [e.g., 8] to help better understand the origin, formation and evolution of IDPs.

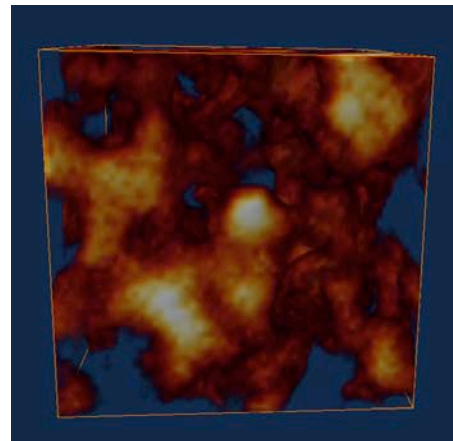


Fig. 3. A close-up of part of the interior enclosed with green markers (Fig. 2) with the volume of  $945 \times 945 \times 945 \text{ nm}^3$ .

**References:** [1] Brownlee D. E. (1985) *Ann. Rev. Earth Planet. Sci.* 13, 147-173. [2] Warren J. L. & Zolensky M. E. (1994) *AIP Conf. Proc.* 310, 245-254. [3] Flynn G. J. & Sutton S. R. (1991) *LPS XXI*, 541-547. [4] Fraundorf P. et al. (1982) *LPS XIII*, 225-226. [5] Zolensky M. E. et al. (1989) *LPS XX*, 125-126. [6] Joswiak D. J. et al. (2005) *Proc. Dust in Planetary Systems (ESA SP-643)*, 141-144. [7] Donn B. D. (1990) *A&A* 235, 441-446 and references therein. [8] Matrajt G. et al. (2011) *LPS XXXXII*, Abstract #1049.

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