LOCATING STARDUST-LIKE PARTICLES IN AEROGEL USING X-RAY TECHNIQUES. A. J. G. Jurewicz[1], S. M. Jones[1], A. Tsapin[1], D. T. Mih[1], H. C. Connolly Jr[2,3], and G. A. Graham[4], [1]JPL/CIT (m/s 183-501, 4800 Oak Grove Ave., Pasadena CA 91109), [2]Kingsborough College - CUNY (Dept. of Phys. Sci., 2001 Oriental Blvd., Brooklyn, NY 11235), Dept. Earth Planet. Sci., [3]American Museum of Natural History, New York, NY 10024, [4]The Open University, Milton Keynes MK7 6AA UK.

Introduction: Silica aerogel is the material that the spacecraft STARDUST *[1]* is using to collect interstellar and cometary silicates. Anticipating the return of the samples to earth in January of 2006, MANY individual investigators and, especially, the investigators in NASA's SRLIDAP program *[2]* are studying means of both *in situ* analysis of particles, as well as particle extraction.

Thus far, the best contender for *in situ* particle compositional analysis is the use of synchrotron-derived x-rays produced by advanced photon sources in national laboratories, *e.g.[3]*. Many, if not all, of the returned cells of aerogel will have preliminary examinations done in these specialty laboratories.

Soon thereafter, aerogel containing extraterrestrial particulates will be allocated to individual PI's within the planetary materials community for study. Many investigators will want to extract the particles from the aerogel; others will want to precisely locate one of (hopefully) many particles for specific *in situ* analyses. For both expediency, and to minimize the amount of transport that these unique and irreplaceable samples must endure, all investigators need a means of identifying and locating the particles in their own laboratories.

If the aerogel remains optically clear -- as it was prior to launch -- large particles can be located in the laboratory using an optical microscope equipped with a long-working distance objective. Small particles deep in the aerogel may be difficult to locate (and more difficult to identify) with optical microscopy because, for example: (1) tracks from other particles may obscure the line of sight, (2) even clear aerogel has some intrinsic absorbance and refraction of light that can cause distortion, and (3) the captured particles will have an accreted "shell" of aerogel surrounding them. Moreover, if the clarity of the aerogel has degraded during the particle collection -- say, because of exposure to cometary volatiles or incorporation of carbon-rich fines -- optical microscopy may not be an option for a majority of the particles.

Accordingly, to help individual PI's with extraction of particles from aerogel in their own laboratories, we are exploring the use of standard laboratory x-ray equipment and commercial techniques for precisely locating specific particles in aerogel.

Background: Radiography has been applied to particles in aerogel on a limited basis e.g., *[4,5,6]*. However, until recently, the resolution of densities has been too limited and the spatial resolution within the images has been too coarse for common commercial x-ray techniques to be applied to STARDUST returned-samples. That is: (1) if you can't see the track leading to the particle in the aerogel, you can't prove that the particle is extraterrestrial, and (2) if the grain-size of the particle is smaller than the resolution, you can't characterize it.

Advances in commercial x-ray imaging technology and related software may have strongly mitigated those limitations. These innovations include: real-time digital imaging; high-contrast detectors and image intensifiers; as well as high spatial-resolution, "nanofocus" instruments, giving spatial resolution *below 1.0μ*, *[7]* and software that can calculate 3-D images using reference points in sets of 6 or more images taken at multiple, known angles, using radiography or other techniques.

Approach: We approached the evaluation of commercial x-ray techniques as follows. First, we determined the most appropriate detector for use with aerogel and particulates. Then, we compared and contrasted techniques useful for university laboratories.

Direct comparison of detectors. One vendor (VJ Technologies, www.vjt.com) provided simultaneous access to three X-ray systems with different detectors, so that direct comparisons could be made in real time. The detectors available were: an amorphous silicon detector, a camera-scintillator system, and a CMOS detector. Standard radiography (2-dimensional) of representative samples was performed in all three systems.

Test samples used to evaluate the relative density resolution (and range) of the detectors included:

- gradient-density (flight-like) and layered-density (prototype) silica aerogel made for STARDUST;
- aerogel with particles; e.g., 20 mg/cc silica aerogel shot in light-gas gun test testing, and aerogel exposed on the MIR spacestation;
- C-Si aerogel (~80-100 mg/cc) that had been arc-jet tested in the ARC (Ames Research) facility;
- silicate charges from high-temperature diffusion and phase-equilibria experiments.

In most cases, the camera-scintillator detector combination provided the broadest, most useful range in density contrasts for these samples. However, all were adequate for some purposes. Comparative radiographs (MIR sample; commercial microfocus sources, ~1μ to 3μ spot size) are given in Figure 1. Because of it's high sensitivity, the camera-scintillator system also gave the best results using the recently-developed nanofocus (~0.1μ to 1μ spot size) x-ray source (Figure 2).

Comparison of commercial, 3-D x-ray techniques. The three techniques investigated are CT, laminography, and *Digitome®*'s 3-d reconstruction technique.

(a) Micro CT -- was successfully performed on shot aerogel using a standard commercial *Skyscan-1072* unit (www.skyscan.be) at JPL. This tomographic x-ray unit has a

relatively large x-ray spot size (~2 μ) compared to what is now available, as well as an older camera-scintillator detector. The aerogel fragment studied had been shot with particles ≤13μ using the light gas gun at ARC). A photograph of the specimen as well as planes computed from reconstructed 3-d images are given in Figure 3.

CT was also performed using a system that had a newer camera scintillator detector (as per Figure 1, courtesey *VJ Technologies*); however, there was some wobble in the scans because the system was still in the process of being aligned for shipment. We note that the wobble would have been insignificant for most industrial CT applications, and demonstrated the need for exquisite alignment for viewing the ultra-small particles expected in STARDUST aerogel. Still, major features (cracks) were visible despite the wobble.

One minor problem using CT (*vs.* alternate for 3-d reconstruction, e.g., the *Digitome®* technique discussed later) is that dense points in a low density matrix tend to make "ring"-artifacts in the computed image because of their ability to block the x-rays. These artifacts were prominent for both particles in aerogel as well as the experimental silicate charge (which contained platinum wires) in the CT in which the stage had the small wobble. Better system alignment and longer exposure times would certainly have mitigated the rings, as was done with the CT in Figure 3.

(b) Laminography – was also tested on aerogel containing fragments of particles. The commercial unit provided for the test contained an amorphous silicon detector, not a camera-scintillator combination. In laminography, the sample stays stationary and the detector itself swings up to ~(90°) to get multiple radiographs at varying, known angles. The primary problem observed with the commercial unit was the need for exacting alignment of the detector: again, what was more than adequate for most commercial applications gave unacceptable shadowing for aerogel samples.

(c) *Digitome®*'s 3-d reconstruction technique *[8]* – is designed for radiographs but, in theory, can use any any set of images having multiple, known reference points. The more images, the better the spatial resolution of the final reconstruction; 6 images are minimum, 8 are usually used. It is based on the shadowing of dense, known objects, and so has great potential for locating dense silicate particles in low density aerogel. Moreover, the company claims that determining special coordinates (rather than composition, etc.) is it's strong suite. Currently, we are negotiating with *Digitome®* for the chance to test their reconstruction techniques on aerogel (with particles) on which we place fiducal marks.

References: [1] Brownlee D. E. et al. (1996) *Proc., ASP Conf. Ser., 104*, 223-226. [2] i.e., *NRA-01-OSS-01-SRL*. [3] Flynn G. (2002) *34th COSPAR* Abstract. [4] Jurewicz A.J. G. (2000) *LPS XXXI*, Abstract #1836. [5] Graham G. A. (2001) *LPS XXXII*, Abstract #1637. [6] Bish D. L. (2001) *LPS XXXII*, Abstract #1416. [7] www.semiconductoronline.com (phoenix x-ray). [8] www.digitomecomp.com.

Acknowlegements: This work was supported by NASA under RTOP 624.13.55.01, JPL/CIT. Thanks to *VJ Technologies*, *Phoenix X-ray*, and *Skyscan* for their help and cooperation.

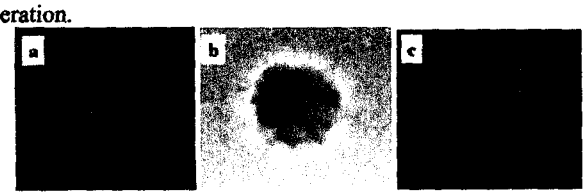

Figure 1. Comparing detectors: radiographs of a ~3mm pit in the MIR aerogel. (a) amorphous silica; (b) camera scintellator; (c) CMOS. In a, c dark is high density; in b, light is high density. Note that all three systems show texture at bottom of pit; (b, c) are sharpest images.

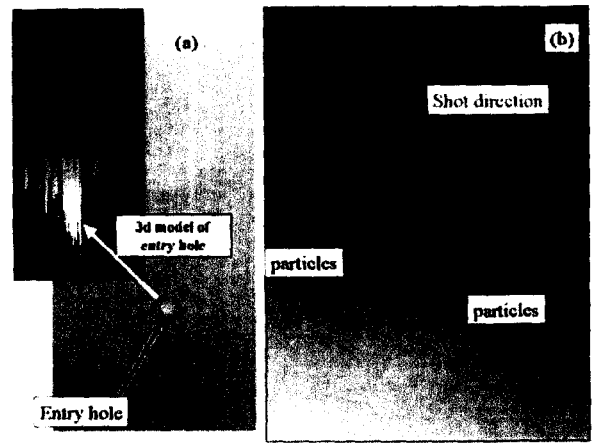

Figure 2. Radiographs by nanofocus x-ray tube but different detectors. (a) entry hole imaged using CMOS detector and converted to 3-d; (b) particles imaged using camera-scintillator – black arrows point to example particles. All shot particles were nominally ≤13μ (see (a) in Figure 3).

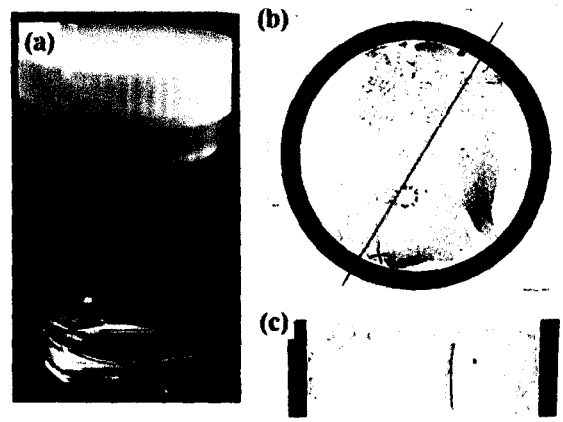

Figure 3. Micro-CT on shot aerogel (~≤13μ particles). (a) shell vial with shot aerogel; (b) horizontal section ~1mm below shot surface; (c) vertical section (top is shot surface) along orange line in (b). Heavy dark lines (b,c) are the shell vial, ~12mm inside diameter; tracks and cracks in aerogel are white. This CT used the *Skyscan-1072* CT unit at JPL.