LABORATORY CAPTURE, ISOLATION AND ANALYSIS OF MICROPARTICLES IN AEROGEL: PREPARATION FOR THE RETURN OF STARDUST

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Introduction: Over the past 30 years geochemical studies of cosmic dust have focused on material retrieved or captured from terrestrial locations, e.g. the stratosphere and Antarctic ice [1,2] and from low Earth orbit (LEO) [3-4]. Extensive investigation, using a wide range of analytical techniques [5] has resulted in substantial data on mineralogical and chemical properties of particles [e.g. 6]. Yet, despite detailed studies [7] it is generally not possible to state unambiguously the parent body origin (in terms of cometary versus asteroidal) for a given class of particle [8]. In 2006 dust particles collected from Comet Wild 2 should be returned by NASA's Stardust spacecraft [9]. Aerogel is the primary capture cell media [9], however other surfaces on the spacecraft will also preserve particle remnants. Laboratory simulations and LEO exposure [10-12] have shown that particles impacting into aerogel at hypervelocity (e.g. 6 km s^{-1}) can be preserved as almost intact pristine material. After recovery and preliminary investigations of Stardust at the NASA Johnson Space Center curation facility, samples will be made available to the international community for analysis [9]. It is therefore essential that groups prepare for the return of samples, in particular developing extraction techniques and analytical strategies so that the maximum yield of information can be obtained from every particle examined. Herein we discuss observations made during laboratory experiments focussing on a possible extraction technique and in-situ mineralogical analysis.

Experimental: Impact experiments were carried out using a two stage light-gas-gun (LGG). The aerogel targets had a density of 96 kg m^{-3}. A range of projectile compositions were used, from individual, homogeneous minerals (e.g. olivine, pyroxene, etc.), to complex, crushed meteorite powders (matrix material from Murchison, Allende and Orgueil carbonaceous chondrites). In a 'buck-shot' technique [13] projectiles were sabot-mounted and accelerated to approximately 5.1km s^{-1} to impact the aerogel targets.

Raman spectroscopy was carried out on an Instruments SA (now Jobin Yvon) model HR640 spectrograph with a liquid nitrogen cooled CCD detector, linked to an Instruments SA Raman scattering module based on an Olympus BX40 microscope. The analytical conditions for the work were: spot size of approximately 25μm; a He-Ne laser (wavelength 632.8 nm) at 20 mW power. A full description of the technique is given in [14].

Electron microscopy was carried out using a Jeol 840 scanning electron microscope with an Oxford Instruments eXL energy-dispersive spectrometer microanalyser (EDS). Samples were carbon coated. Typical analytical conditions were 32 mm working distance, 2 nA beam current and 20 kV. High resolution imaging was carried out using a Philips XL FEG-SEM with an accelerating voltage of 5-7 kV and a working distance of 10mm.

Ablation of aerogel was carried out using a 266 nm quadrupled Nd-YAG laser operated at 10 Hz. The aerogel block was subjected to pulses of variable energy (e.g. 0.5 to 6 mJ/pulse) over variable exposure times (e.g. 5 to 30 seconds).

Discussion:

Simulation of Impact Features. The LGG shots proved to be highly successful in generating both track features and capturing the projectile particles at impact velocities similar to those of the encounter velocities of the stardust collectors with Comet Wild 2 [9].

Extraction Technique. Probably the most significant challenge to overcome before the actual return of stardust samples is the extraction procedure of particles from the aerogel. Several novel techniques have previously been applied [15-16] with varying degrees of success. We have carried out preliminary experiments using a Nd-YAG laser to ablate aerogel, with the aim of either cutting a captured particle out of the aerogel, or removing excess aerogel so that in-situ analysis can be achieved. Our initial experiments have shown that different combinations of pulse energy and duration allow fine control of ablation by moving the sample relative to the fixed beam on a computer controlled x-y stage, creating 'pits' (nm to μm in depth) and 'tracks' (μm in depth). The technique will now be used to attempt isolation of impact-captured olivine particles.

Bulk Analysis. It is important to ascertain which analytical techniques should be used following extraction of captured particles, and especially, in what sequence. Stable isotopic measurements on extraterrestrial materials are clearly of major importance, as demonstrated by the long-established record of the PSSRI [e.g. 17]. However, whilst such analyses provide valu-

able information on light elements, they are by their very nature essentially destructive. Clearly, such measurements must be acquired at the end of a sequence of analytical protocols. The first fundamental task is identification of bulk chemistry and mineralogy. Analytical SEM and electron microprobe analysis are the usual workhorse techniques [5]. Unfortunately, both require that samples be given a conductive carbon coat, contaminating the sample surface and precluding the reliable use of stable isotopic mass spectrometry. Advances in microscopy have removed the need for the conductive coat in some circumstances, see discussion in [5]. Notwithstanding the potential contamination issue, conventional SEM does enable imaging and detailed X-ray microanalysis of impact tracks and captured projectiles. Nevertheless, initial characterization of particles needs a non-destructive, and non-contaminating technique.

Raman Spectroscopy. We have used Raman spectroscopy. The technique obtains a vibrational spectrum of the crystal lattice of the sample by illuminating it with a laser, and collecting the spectrum of inelastically scattered photons. The detected spectrum contains a suite of peaks that are characteristic of the chemical structure of the sample. As each component of the sample that is illuminated by the laser may contribute to the spectrum seen, it is fortunate that the aerogel does not significantly absorb or scatter the laser light. Consequently, only the selected impacted particle material is analyzed (Figure 1).

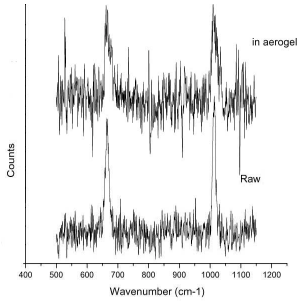


Figure 1. The Dispersive raman spectra for a pristine pyroxene grain and a grain captured in aerogel.

Our experience suggests that Raman spectroscopy is an extremely powerful technique for the non-destructive analysis of particles trapped in aerogel, especially as it allows the differentiation of materials of similar composition but widely differing states of shock deformation, not possible by X-ray microanalysis. This is an important ability as a previous investigation [18] has indicated that impacted particles may be subject to thermal and mechanical alteration. However there are several drawbacks with the technique. Localized small-scale thermal heating of the sample can occur, as observed during our analysis of matrix material from Allende. Also many Raman systems have relatively broad analytical spot size, resulting in spectra from more than one fine-grained mineral component. The recent purchase of a new Jobin Yvon LabRam HR Raman microscope gives a spot size down to 2 μm which will hopefully enable us to distinguish between the different fine-grained mineral components present within the captured particles. Furthermore Raman only works on samples that contain covalent bonds [5], so it is necessary to carry out complementary investigations, such as synchrotron X-ray microprobe studies [19]. We propose to use a new type of X-ray source microprobe [20]. Bland and Cressey, with industrial partners Bede Scientific, are developing a high-brightness X-ray probe that will allow both rapid phase identification and quantification of abundance in-situ for small samples, and has the potential for automated phase mapping, imaging and analysis of polished sections. Preliminary results [20] show that patterns can be acquired from 50 μm areas of carbonaceous chondrite matrix, and can identify individual phases. Improvements in resolution to 10 μm, and a higher X-ray flux, are anticipated when the instrument is completed.

Summary: Laboratory experiments have shown that we have made substantial steps toward isolation and characterization of aerogel-captured material. The techniques will now be applied to an aerogel block exposed in LEO [11].

References: [1] Bradley J. P. and Brownlee D. E. (1986) *Science*, 231, 1542-1544. [2] Maurette M. et al. (1991) *Nature*, 351, 44-47. [3] Zolensky M. E. (1994) *AIP Conf. Proc 310*, 291-204. [4] Graham G. A. et al. (2000) *Adv. Space Res.*, 25, 2, 303-307. [5] Zolensky M. E. et al. (2000) *Meteorit. Planet. Sci.*, 35, 9-29. [6] Messenger S. (2000) *Nature*, 404, 968. [7] Messenger S. and Walker R. M. (1998) *LPS XXIX*, #1906. [8] Kelm K. et al. (1999) LPS XXX, #1999. [9] Brownlee D. E. et al. (2000) *Meteorit. Planet. Sci.*, 35, A35. [10] Tsou P. (1995) *J. Non-Crystalline Solids*, 186, 415-427. [11] Hörz F. et al. (1998) *LPS XXIX*, #1773. [12] Burchell M. J. et al. (1999*) Planet. Space Sci.*, 47, 189-204. [13] Burchell M. J. et al. (1999) *Meas. Sci. Technol.*, 10, 41-50. [14] Burchell M. J. et al. (2001) *Meteorit. Planet. Sci.*, 36. [15] Raynal P. I. et al. (2000) *LPS XXXI*, #1319. [16] Stadermann F. J. and Floss C. (2000) *LPS XXXI*, #1372. [17] Graham G. A. et al. (1996) *Meteorit. Planet. Sci.*, 31, A53-54. [18] Bunch T. E. et al. (1991) *Icarus*, 91, 76-92. [19] Flynn G. J. et al. (2000) *LPS XXXI*, #1457. [20] Bland P. A. et al. (2000) *Meteorit. Planet. Sci.*, 35, A27.