SYNCHROTRON X-RAY ANALYSIS OF CAPTURED PARTICLE RESIDUE IN AEROGEL KEYSTONES. A. L. Butterworth[1], A. J. Westphal[1], C. J. Snead[1], N. Tamura[2], S. Bajt[3], G. A. Graham[3] and J. P. Bradley[3]. [1]Space Sciences Laboratory, University of California at Berkeley, Berkeley, CA 94720, USA (annab@ssl.berkeley.edu), [2]Advanced Light Source, Lawrence Berkeley National Laboratory, Berkeley, CA 94720 [3]Institute for Geophysics and Planetary Physics, Lawrence Livermore National Laboratory, 7000 East Avenue, Livermore, CA 94550, USA.

Introduction: On January 2nd this year, Stardust successfully passed through the trail of Comet Wild-2 and harvested cometary particles. These particles, in addition to the contemporary interstellar grains captured during the spacecraft's journey to Wild-2 will be returned to Earth in 2006 [1]. In preparation for this return it is important to evaluate and develop suitable sample analysis techniques for these unique particles. The micrometeoroids and orbital debris captured by NASA Orbital Debris Collector Experiment (ODCE) exposed on the *Mir* space station has provided suitable analogues of the typical impact features that maybe preserved in the Stardust collectors [2]. Detailed optical microscopy of impact features in the ODCE aerogel collectors has indicated that particles can undergo fragmentation during capture (Fig. 1).

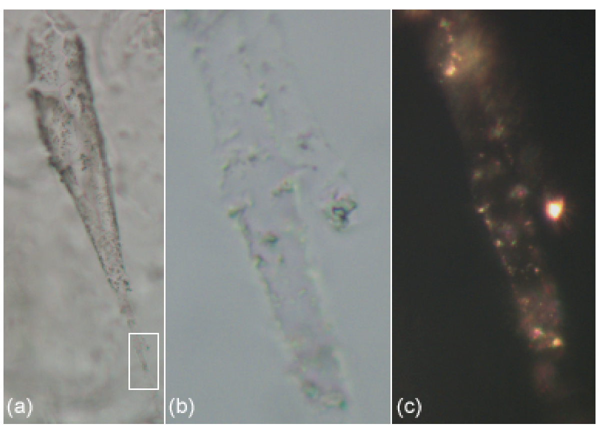

Fig 1. (a) Transmitted light (TL) micrograph of an extracted impact track. (b) TL micrograph of the region of interest located in (a). (c) Dark-field optical micrograph of the same region of interest identifying a number of finely dispersed particulates. All images are acquired using a Lecia DM RXA2 microscope fitted with a Lecia DC 500 digital camera.

Our existing research efforts have focused on the extraction of these entire impact features in the form of keystones [3]. These keystones are the first step in the extraction process that will eventually lead to "naked" particle recovery [e.g. 4]. To enable the recovery of the "naked" particle or particulates it is important that the distribution of projectile debris along the track is known. It can be difficult to discriminate between finely dispersed projectile debris and fragments of aerogel that have condensed or crystallized during the capture event using optical or secondary electron imaging. It is essential that impact features be subjected to elemental and mineralogical characterization before individual particulates are recovered. Previous work has shown that synchrotron techniques are well suited to the in-situ analysis of embedded particles [5-6]. In this abstract we report on the preliminary investigation of the suitability of the synchrotron micro X-ray Diffraction beamline (7.3.3) at the Advanced Light Source at the Lawrence Berkeley National Laboratory.

Experimental: For this experiment we used an aerogel keystone (~500 x 200 x 200 µm) containing an impact track approximately 200µm in length. Preliminary optical imaging identified a number of particulate remnants dispersed along the length of the track. The keystone was mounted in air, supported by a two-pronged silicon fixture, a "microforklift" [2], which was secured by a handle to a copper plate. As the keystone is mounted on the microforklift, there is no need for any supporting substrate and therefore no contributing background signal. The Micro X-ray Diffraction beam line utilizes the synchrotron source and operates within the 6-12 keV energy range. An X-ray CCD and a fluorescence detector are available with the capabilities of 1 µm spot analysis and larger area mapping. As the purpose of the experiment was to map the fragmented particulate debris we chose an 80 x 80 µm area upstream of the terminal particle. We made fluorescence maps of calcium and iron of the selected area in order to locate particle fragments. We then mapped the selected area by collecting a grid of 53 by 53 points with a 1.5 µm spacing. At each point we collected Laue X-ray diffraction patterns (Fig. 2) and X-ray fluorescence spectra. A white-light source was used for mapping.

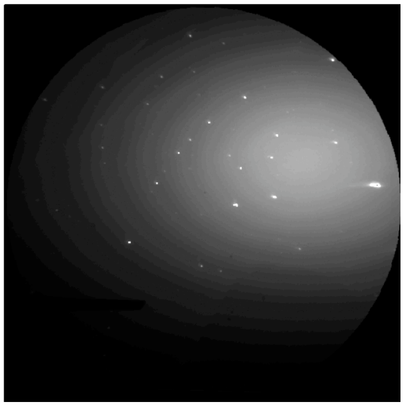

Fig. 2 Laue X-ray diffraction pattern and the distribution of peaks.

Results: The fluorescence maps and X-ray diffraction summary data are shown in Figure 3. The upper plots clearly show correlated hotspots of calcium and iron. It is highly likely that the Fe signal relates to the projectile material. However the Ca signal is more ambiguous as it may be a remnant of the gel forming process.

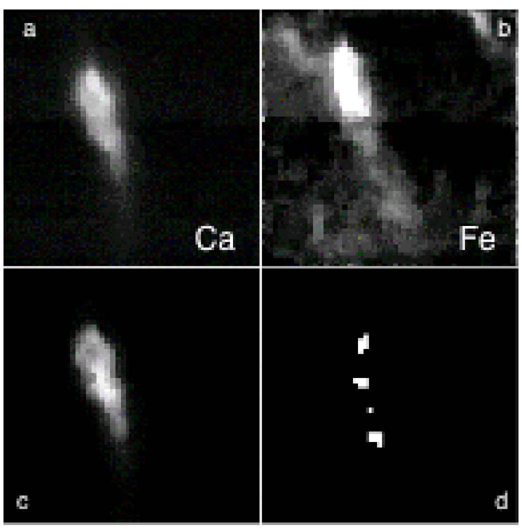

Fig 3. An 80x80 μm area of an impact track in an aerogel keystone (the track direction is left to right). a) relative XRF Ca intensity. b) XRF Fe intensity. c) The mapped XRD integrated peak intensities d) distribution of a possible pyroxene mineral phase as deduced from Laue patterns.

The lower left plot shows a summary of the integrated Laue pattern peak intensities, which is related to the amount of crystalline material in the X-ray beam. The brightest regions corresponds to those Laue patterns with the brightest and most numerous peaks. Most pixels contain no evidence for crystalline material, which is consistent with only the presence of amorphous silica. Finer grained material may have been below the current detection limit of the beamline setup.

The Laue patterns were indexed using lattice parameters for several likely mineral phases. It should be noted that the white-light scan cannot distinguish between very similar lattice structures, so the mineralogical composition of several of the embedded particulates can only tentatively be given as pyroxene. Future analysis using the beamline will utilize an energy scan with monochromatic light that would provide greater resolution and therefore enable enhanced identification of the mineralogy phases.

Discussion: For the preliminary investigations of the returned cometary particles there maybe an advantage in retaining the spatial information about the impact event and fragmentation of the grain. Therefore ability to acquire in situ chemical and mineralogical data is a significant step forward to developing an analytical strategy for Stardust. We concentrated on residue in the carrot-shaped hypervelocity region of the track, rather than a large terminal particle. Larger particles with unambiguous origins such as the terminal particle may be detected with optical microscopy, and can usually be removed by straightforward means [3]. Without analyzing fragments deposited in the track, it may be that a biased view will be obtained of Stardust sample composition. The combined ability to acquire both elemental and crystallographic data from the preserved particles is a significant advantage, as it will enable the location of both crystalline and amorphous materials.

We have shown in this preliminary study that the X-ray beamline at the ALS will be useful tool in analyzing material trapped in stardust aerogel by the Bay-PAC consortium [3]. In future we will determine the detection limit on grain size in aerogel that can be characterized by synchrotron XRD/XRF using artificially embedded particle standards.

References: [1] Brownlee D. E. et al (2000) *MAPS, 35*, A25. [2] Hörz F. et al (2000) *Icarus, 147*, 559-579. [3] Westphal A. J. et al., (2003) *MAPS*, submitted. [4] Graham G. A. et al., (2004) this volume. [5] Flynn G. J. et al., (2000) *LPS XXXI*, abstract #1457. [6] Flynn G. J. et al., (2001) *LPS XXXII*, abstract #1398.