FOCUSED ION BEAM RECOVERY AND ANALYSIS OF INTERPLANETARY DUST PARTICLES (IDPs) AND STARDUST ANALOGUES.

G. A. Graham[1], J. P. Bradley[1], M. Bernas[2], R. M. Stroud[3], Z. R. Dai[1], C. Floss[4], F. J. Stadermann[4], C. J. Snead[5] and A. J. Westphal[5]. [1]Institute for Geophysics and Planetary Physics, Lawrence Livermore National Laboratory, Livermore, CA 94551, USA (graham42@llnl.gov), [2]FEI Company, Hillsboro, OR 97124, USA, [3]Naval Research Laboratory, Washington DC 20375, USA, [4]Laboratory for Space Sciences, Washington University, St Louis, MO 63130, USA. [5]Space Sciences Laboratory, UC Berkeley, Berkeley CA 94720.

Introduction: Meteoritics research is a major beneficiary of recent developments in analytical instrumentation [1,2]. Integrated studies in which multiple analytical techniques are applied to the same specimen are providing new insight about the nature of IDPs [1]. Such studies are dependent on the ability to prepare specimens that can be analyzed in multiple instruments. Focused ion beam (FIB) microscopy has revolutionized specimen preparation in materials science [3]. Although FIB has successfully been used for a few IDP and meteorite studies [1,4-6], it has yet to be widely utilized in meteoritics. We are using FIB for integrated TEM/NanoSIMS/synchrotron infrared (IR) studies [1].

FIB Instruments: The results discussed in this abstract were obtained using a FEI dual beam Strata 237 workstation that consists of a field emission electron source and a Ga+ (liquid/metal) source. The workstation was also fitted with a 30 keV scanning transmission electron microscope (STEM) detector, x-ray energy-dispersive spectrometer, and a nanomanipulator. A FIB will be delivered to Livermore in March 2004. It will be equipped with secondary and backscattered detectors, an energy-dispersive x-ray spectrometer, cathodoluminescence and electron backscattered diffraction detectors, X-ray tomography, and a nanomanipulator.

Application of FIB to IDPs: FIB has been used to prepare electron transparent TEM sections of geological samples [4-6]. We are using FIB to produce thin sections of IDPs. An anhydrous IDP U2022 G14 mounted on a polyethylene IR substrate was coated with film of Pt deposited in the FIB first using the electron beam and then using the ion beam. The Pt film or "strap" protects the specimen by reducing excessive surficial ion etching. The Ga^+ ion beam operating at currents ranging from 10 to 20,000 pA is then used to ablate material on either side of the protective "strap". The result is a thin section approximately 1µm thick. The section is then subjected to further low current and low voltage milling to produce a TEM section ~100nm in thickness. The section is removed from the trench using the "lift-out" technique [5,6]. Unfortunately, the lift-out procedure consumes most of the IDP. Only one or two TEM sections are recovered with the rest of the particle destroyed by ion milling, in stark contrast to ultramicrotomy where a large number of electron and optically transparent sections can be harvested from a single IDP [7].

A unique advantage of the FIB method is "site-specific" sampling where regions or particles as small as ~100 nm or less can be extracted and thin-sectioned. We are using site-specific sampling to recover isotopically anomalous "hotspots" from ion probe samples (Figs. 1 & 2). To analyze samples in the ion microprobe, IDPs are usually pressed into high-purity gold foils. Using site-specific sampling we can correlate isotope measurements and mineralogy with nanoscale precision (Fig. 2). The sections are extracted from the ion probe foils using the lift-out technique, examined using TEM and, if necessary, re-examined using NanoSIMS.

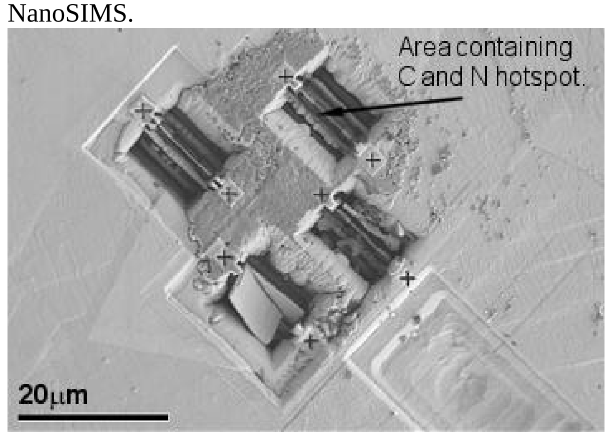

Fig. 1. Secondary electron micrograph of IDP Benavente (L2036-G16) from which multiple FIB sections have been harvested.

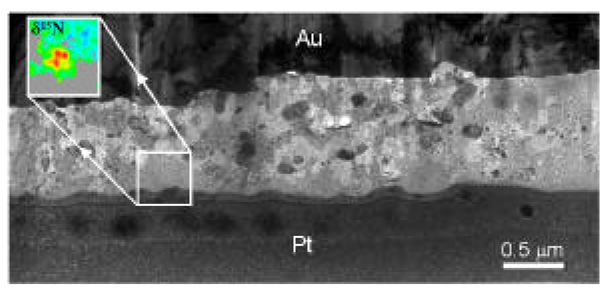

Fig. 2. Bright-field micrograph of FIB extracted section of Benavente. Inset (upper left) is a "hotspot" ($d^{15}N$ = 1110 ± 98 ‰) obtained from boxed area in section using NanoSIMS.

Application of FIB to Stardust Particles: The successful encounter of Stardust with comet Wild 2 provides new impetus for refining our methods for extracting particles from aerogel [8]. We have obtained detailed optical images of impact tracks preserved in aerogel (density 0.02 g/cm^3) that was exposed on the Russian *Mir* Space Station as part of NASA's Mir Environmental Effects Package [9]. Most tracks show evidence of extreme projectile fragmentation. It is possible that the collected Stardust particles will have suffered the same fate. The development of a technique to recover aerogel keystones is an important milestone [8], but the particles within keystones *remain embedded in aerogel*. The focus of our current Stardust activities is advanced particle management where we are developing techniques to routinely remove micron and sub-micron fragments from tracks within keystones.

A small fragment of aerogel containing an impact track was attached to an SEM stub using an adhesive carbon pad and investigated under the dual beam instrument. The tungsten needle nanomanipulator (in conjunction with secondary electron imaging) was the primary tool employed for particle recovery. The needle was used to remove the "roof" of the impact track after the ion beam had been used to make strategic cuts in the aerogel. Backscattered electron imaging revealed that the freshly exposed track "floor" was covered with sub-micron fragments of projectile debris (Fig. 3).

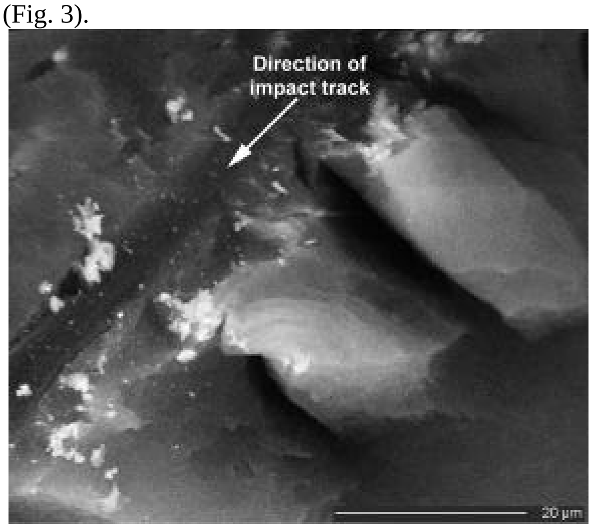

Fig 3. BSE micrograph of the preserved impact path on the aerogel. A number of sub-micron particulates are in the field of view.

One of the particulate fragments identified using backscattered imaging was picked up and extracted using the tungsten needle. The particle was welded to the needle using Pt. Normally the Pt is deposited using the ion beam but to reduce the potential damage to the particle, the electron beam was used instead. The needle with the particle attached was then extracted from the debris field (Fig 4). The particle was transferred to a TEM grid within the chamber. Using Pt as a weld, the particle was attached to the TEM grid. The particle was thinned to electron transparency (~100nm thick) using the conventional FIB milling protocols. It is also possible to transfer particles to a number of substrates for other microanalysis techniques.

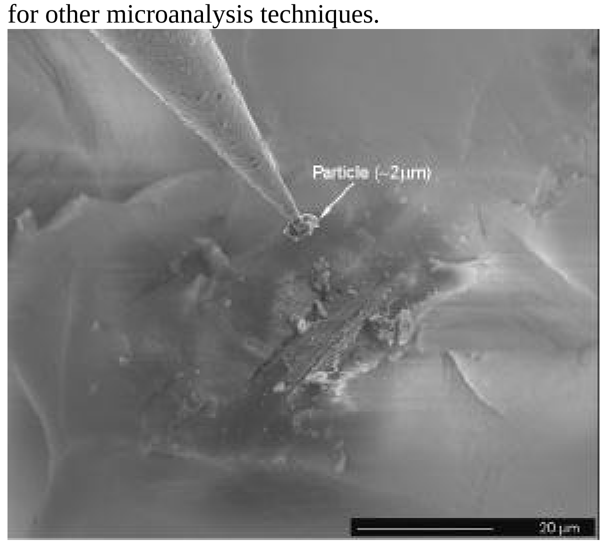

Fig. 4. Secondary electron micrograph of the Omniprobe^{TM} tungsten needle nanomanipulator extracting an individual grain (~2 µm in diameter).

Conclusions: We believe that FIB will play an increasingly prominent role IDP research, although ultramicrotomy is less destructive and the more efficient method for harvesting large numbers of thin sections from a single IDP. It is likely that FIB will play an important role in the recovery of cometary particles and a pivotal role in the recovery of individual interstellar grains from the Stardust aerogels.

References: [1] Floss et al (2004) submitted to *Science*. [2] Bradley J.P. et al. (2004) this volume. [3] Giannuzzi L. A. and Stevie F. A. (1999) *Micron, 30*, 197-204. [4] Stroud R. M. et al. (2000) *MAPS, 35*, A153-154. [5] Lee M. R. et al. (2003) *Min Mag, 67*, 581-592. [6] Heaney P. J. et al. (2001) *American Mineralogist*, 86, 1094-1099. [7] Bradley J. P. and Brownlee D. E. (1986) *Science, 251*, 1542-1544. [8] Westphal A. J et al. (2003) submitted to *MAPS*. [9] Hörz F. et al. (2000) *Icarus, 147*, 559-579.

Acknowledgement: The work carried out by GAG and JPB was performed under the auspices of the U.S. Department of Energy, National Nuclear Security Administration by the University of California, Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.