



Aerogel keystones: Extraction of complete hypervelocity impact events from aerogel collectors

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Abstract—In January 2006, the Stardust mission will return the first samples from a solid solar system body beyond the Moon and the first samples of contemporary interstellar dust ever collected. Although sophisticated laboratory instruments exist for the analysis of Stardust samples, techniques for the recovery of particles and particle residues from aerogel collectors remain primitive. Here, we describe our recent progress in developing techniques for extracting small volumes of aerogel, which we have called “keystones,” which completely contain particle impacts but minimize the damage to the surrounding aerogel collector. These keystones can be fixed to custom-designed micromachined silicon fixtures (so called “microforklifts”). In this configuration, the samples are self-supporting, which can be advantageous in situations where interference from a supporting substrate is undesirable. The keystones may also be extracted and placed onto a substrate without a fixture. We have also demonstrated the capability of homogeneously crushing these unmounted keystones for analysis techniques that demand flat samples.

INTRODUCTION: STARDUST SCIENCE GOALS AND REQUIRED ANALYTICAL TECHNIQUES

In this first paper in an anticipated technical series from the Bay Area Particle Analysis Consortium (BayPAC)¹, we describe our recent efforts in the development of advanced techniques for the extraction of hypervelocity impacts in silica aerogel collectors and the subsequent preparation of these extracted residues for detailed laboratory analysis. In subsequent papers, we will discuss the analysis of keystone-extracted impacts using a variety of analytical techniques.

¹Building on our existing extraction development effort (Westphal et al. 2002) and leveraging the extensive analytical capabilities available in the San Francisco Bay area, we have formed a consortium of national laboratories and universities: Lawrence Livermore National Laboratory (LLNL), Space Sciences Laboratory (SSL) at the University of California at Berkeley, the Advanced Light Source (ALS) at the Lawrence Berkeley National Laboratory (LBNL), and the Stanford Synchrotron Radiation Laboratory (SSRL) at the Stanford Linear Accelerator Center (SLAC), with a focus on preparation for the return of the Stardust samples.

The Stardust mission, which was launched in 1999, is expected to collect cometary dust from the coma of comet Wild-2 in January 2004. The aerogel collector will be returned to earth for laboratory analysis in January 2006. If successful, Stardust will return the first samples from a planetary body beyond the moon. The importance of this mission to planetary science cannot be overstated. Wild-2 is a long-period comet that was placed into a short-period orbit, with perihelion just inside the orbit of Mars, through a fortuitous encounter with Jupiter in 1974. Wild-2 is probably composed of extremely primitive material that has suffered very little alteration since its accretion into a solid body ~4.6 Gyr ago; indeed, it is not impossible that it is composed of nearly pristine interstellar material. By the time of the Stardust encounter, Wild-2 will have orbited the Sun only ~5 times since the beginning of its residence in the inner solar system.

Since Stardust will return the first samples known with certainty to be cometary material, the zeroth-order questions for Stardust will be:

- Have we seen this type of material before?

- Is it similar to meteorites, micrometeorites, or interplanetary dust particles (IDPs)?
- Is it solar system material, presolar material, or both?
- Are there organics, and if so, what are they (aliphatic, aromatic, N-containing)?

The broader scientific questions that Stardust will eventually address are:

- To what degree was material from the inner solar system mixed with that of the outer solar system in the presolar nebula? Are the reservoirs of material for the two regions distinct?
- How is cometary material related to interstellar dust? What is the origin of crystalline silicates in cometary material, given that interstellar silicates appear to be amorphous?
- What is the origin of organics in cometary dust? Are the organics interstellar in origin, or were they formed in the presolar nebula?

The analyses directed at each of these questions will require overlapping and complementary analytical techniques. For example, to address the first two zeroth-order questions listed above, detailed mineralogical/petrological analysis using analytical electron microscopy will likely be required. But measurements of the elemental compositions using for example proton-induced X-ray emission (PIXE) or synchrotron-based X-ray fluorescence (SXRF) will also be required. The third zeroth-order question will require ion microprobe analyses to measure the isotopic compositions of individual grains. Identification of organics will require sophisticated analytical techniques, e.g., synchrotron infrared spectroscopy or Raman spectroscopy, possibly coupled with isotopic analyses. As an example of what can be learned by doing sequential analyses on individual samples, Floss et al. did nanoscale secondary ion mass spectrometry (nanoSIMS) and synchrotron-based Fourier-transform infrared (FTIR) spectroscopy on the “Benavente” IDP. They showed that a correlated carbon and nitrogen isotopic anomaly in that IDP is

associated with an organic phase (Floss et al. Forthcoming). Sample preparation, therefore, must be as universal as possible to take full advantage of the complement of analytical techniques. As we developed particle extraction techniques, we kept this requirement fully in mind.

The Stardust collector consists of 130 aerogel tiles, with dimensions of 42 mm × 21 mm on the collecting face, 30 mm thickness, and density in a gradient from ~10 mg cm⁻³ at the collecting surface to ~50 mg cm⁻³ at the bottom of the collector. The goal of Stardust is to collect more than 1000 particles at least 15 μm in size and many millions of smaller particles. Because of large uncertainties in the modelling of dust in the cometary coma, this estimate of the statistics is subject to order-of-magnitude uncertainties. Large deviations in these statistics in either direction could pose a problem for Stardust analysis. A yield much smaller than expected could compromise the scientific yield of Stardust. A yield much larger than expected could compromise the structural integrity of the collector and could make in situ characterization of individual particles difficult or impossible. We address this possibility later in this paper.

Hypervelocity Impacts of Friable Chondritic Particles in Aerogel

Particles with speeds of a few km sec⁻¹ or greater typically produce characteristic carrot-shaped tracks when they stop in aerogel collectors (Fig. 1). Particles with initial sizes ~10 μm in size typically produce tracks that are several hundred microns long. Near the entry point, the particle moves hypersonically and produces a strong, cylindrical shock, forming a tubular cavity (the “carrot”). As the shock expands into the aerogel collector, it weakens and finally stops when the shock pressure no longer exceeds the crushing pressure of the aerogel. If the particle is sufficiently fragile, it is disrupted into fragments during the hypervelocity phase. As each surviving particle slows, the shock near the particle

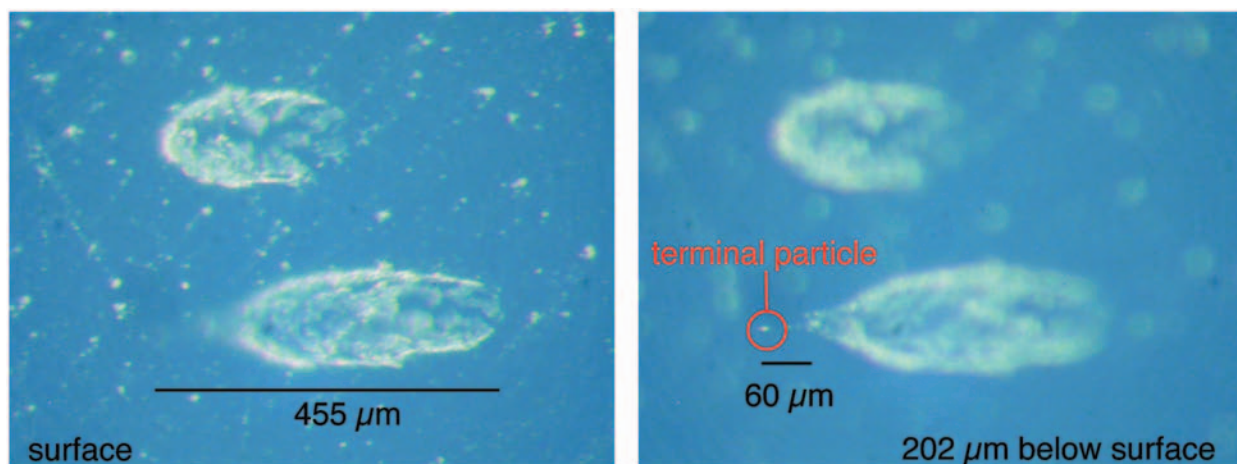


Fig. 1. Anatomy of a hypervelocity particle impact track in aerogel.

weakens until it is either too weak to crush the aerogel or becomes an acoustic wave. In this region, one or more thin “whisker” tracks are produced. Eventually, the surviving particles stop when their ram pressure no longer exceeds the crushing pressure of the aerogel. Dominguez and Westphal (Forthcoming) have developed a detailed model incorporating these physics that accurately predicts the shape and size of these impact tracks for non-fragmenting projectiles. This simple picture is complicated by three effects: ablation, which can be severe for particles with a large volatile component; fragmentation, which we discuss below; and accretion, in which molten and compressed aerogel accretes onto the stopping particle. Aerogel accretion has been reported to partially protect the projectiles from damage (Barrett et al. 1992), although this effect has not been studied extensively. Dominguez and Westphal (Forthcoming) speculate that the large dispersion in observed range is due to repeated cycles of gradual accretion and abrupt, random shedding of heated aerogel by stopping hypervelocity particles.

In our approach to the development of particle extraction techniques, we have assumed that a reasonable analogue for cometary particles collected from the Wild-2 coma will be anhydrous IDPs, which are generally suspected of being cometary in origin (Brownlee et al. 1994). These particles, which have been collected in the stratosphere for many years, are fractal-like aggregates of small grains but often include large, relatively refractory grains (Fig. 2). These particles (or

their artificially-synthesized analogues) are so fragile that they do not survive the strong shocks present inside hypervelocity gas guns, so we have no empirical information on survivability of such particles when captured by aerogel. However, we have assumed that even the mild shock pressures experienced by the particles during capture in aerogel are very likely to disaggregate these fragile particles into their components. Since the range of particles in aerogel is dependent mostly on the particle size, the small components stop quickly, while any large and robust particles present have the longest range and form the track termini. At first inspection under an optical microscope, the resulting track can give the mistaken impression that the entire particle was captured intact, or at least disintegrated into only a few fragments, since the small components are difficult to image and easy to overlook. Since the large, robust components are typically mineral grains, an analysis of the track that does not include the distributed material along the track will be seriously biased toward robust minerals. If only the terminal particles from the Stardust samples are to be recovered and analyzed, we might conclude that comet Wild-2 is composed entirely of forsterite!

The assumption that captured chondritic particles readily shed small fragments on capture is at least partially confirmed by a population of impact events in the Orbital Debris Collection Experiment (ODCE) collector. ODCE was deployed on the Russian space station Mir for 18 months

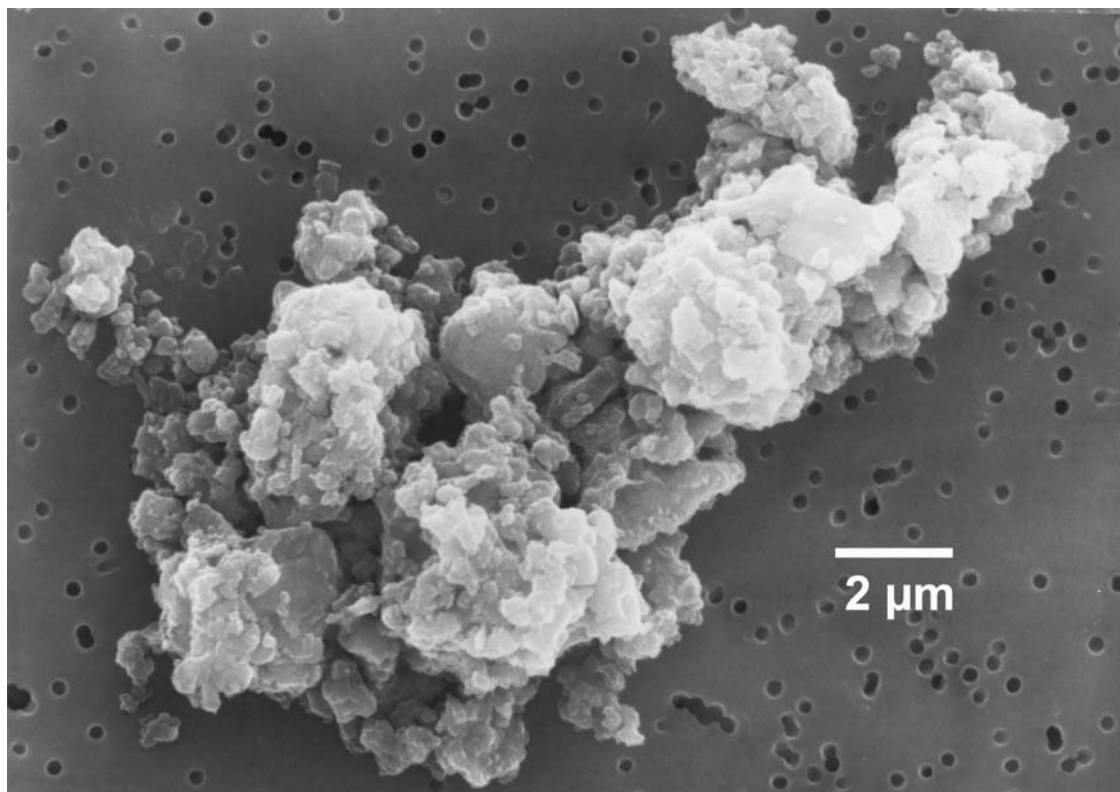


Fig. 2. Image of a typical anhydrous cluster IDP.

starting in March 1996. Zolensky et al. have made a detailed examination of the impacts in this collector (Hörz et al. 2000). A fortunate happenstance has produced a large number of parallel impacts with chondritic composition in the ODCE collector. The working hypothesis is that a relatively large chondritic ($\gg 100 \mu\text{m}$) particle, tentatively classified by Zolensky et al. as CV3 (Hörz et al. 2000), suffered a glancing collision with a component of the space station; this collision was sufficiently strong to disrupt the particle into numerous fragments but was sufficiently peripheral that it produced very little ejecta from Mir. These particle impacts have proven to be very useful as targets for the development of extraction techniques, since their typical sizes ($10 \mu\text{m}$ or smaller) are probably typical of the sizes of particles to be extracted from Stardust, and they are known to be chondritic. Detailed examination of these impacts in an optical microscope (Fig. 3) and using a nuclear microprobe (Graham et al. Forthcoming) shows the presence of a substantial amount of material distributed along the tracks of these impacts. Since CV3 meteorites are probably much less friable than cometary material, we expect cometary impacts to be even more strongly biased away from large, intact terminal particles. We have speculated that cometary impacts may, in fact, resemble the descriptively named hedgehog events observed in ODCE (Hörz et al. 2000). These events resemble small explosions in the aerogel, perhaps consistent with flash-vaporization of volatile materials. Although particles are observed in hedgehogs, usually at the terminus of radial tracks, no distinct terminal particle is usually present.

We conclude that a conservative approach to the preparation for Stardust analysis is to assume that much—perhaps most—of the cometary material will be distributed along impact tracks, and that if one neglects this material, one may introduce serious systematic biases into the analysis. Thus, the entire impact event should be recovered if possible. It may also be important to disturb the remainder of the collector as little as possible. These requirements have driven our development effort.

End-to-End Stardust Analysis

In Fig. 4 we show a draft flow chart for the analysis of Stardust samples, from the initial examination of collectors and identification of particle impacts through detailed measurements in laboratory instruments.

The protocol for initial examination of collectors will be developed by the JSC curatorial facility. Tools for initial examination are well-developed. Westphal et al. have previously demonstrated that an automated scanning microscope can be used to identify particle impacts in aerogel collectors (Westphal et al. 2002). Whether or not automated scanning will be required for the Stardust cometary collector has not yet been established. Other tools that may be useful for distribution of Stardust samples exist now, for example,

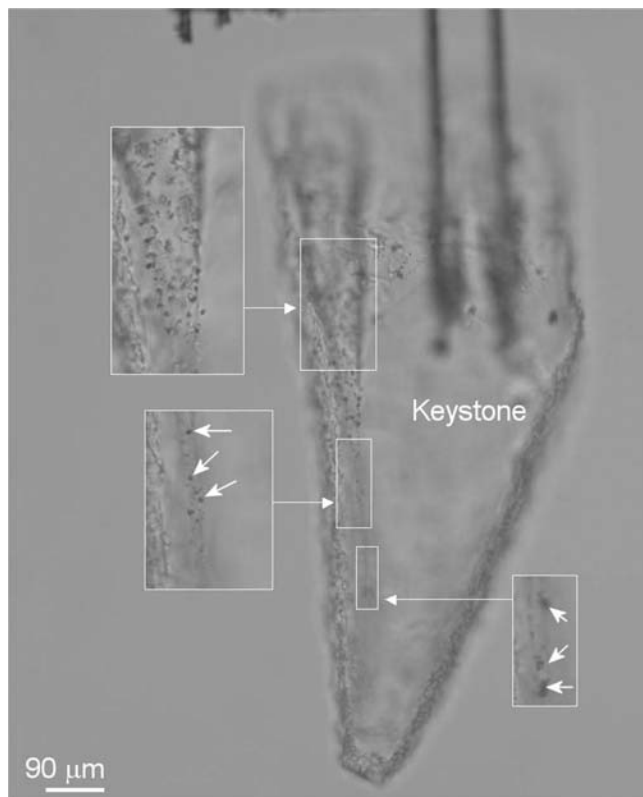


Fig. 3. A “chondritic swarm” impact event observed in transmitted light in an optical microscope. In addition to the large terminal particle, fine-grained particle residue is distributed along the length of the impact track.

large-scale cutting of aerogel cells into smaller pieces using a pulsed UV laser has been demonstrated by Graham et al. (2003). Preliminary in situ characterization of particles could be done by Raman microscopy (Kearsley et al. 2001) or SXRF (Brennan et al. Forthcoming; Flynn et al. 2003).

In 2000, Zolensky et al. (2000) reviewed some of the analytical techniques that will be available for Stardust. Even in the short time since that review was written, spectacular progress has been made in many analytical techniques. For example, using the new Cameca nanoSIMS, Messenger et al. have identified presolar grains in IDPs (Messenger et al. 2003). The spatial resolution of the ^{17}O maps of IDPs in their analyses was $\sim 100 \text{ nm}$. Scanning transmission X-ray microscopy (STXM) can now routinely map various bound states of carbon, nitrogen, and oxygen using near-edge X-ray absorption fine-structure (NEXAFS) with better than 100 nm resolution (Kilcoyne et al. 2003). Further advances are anticipated in the next few years.

But, techniques for extraction of hypervelocity particle impacts from aerogel and subsequent sample preparation for analysis in advanced laboratory instruments remain primitive. In this paper, we focus on the steps between in situ particle identification and analysis—the extraction of impact residues and the preparation of extracted samples for analysis.

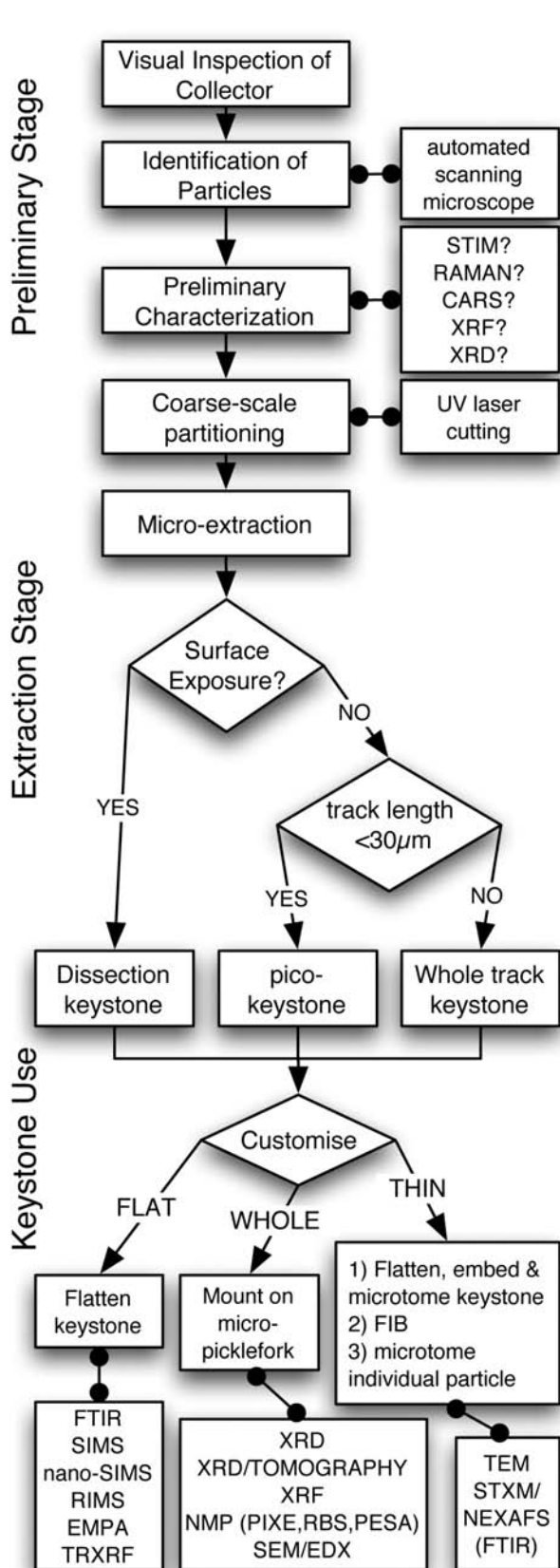


Fig. 4. Draft flowchart for Stardust analysis.

Precision cutting of aerogel on the micron scale is a difficult challenge (Westphal et al. 2002). To “machine” aerogel, we used borosilicate glass microneedles manufactured in our laboratory using a commercially available micropipette puller. We mounted the needles on commercially-available 3-axis micromanipulators (Sutter Instruments MP-285), which were robotically controlled by an independent computer (Sun Sparc 1). The cutting action consists of repeated small axial poking motions of the aerogel by the microneedles. A slice can be produced in the aerogel by poking repeatedly in a line, with increasing depths between each sequence of pokes. We empirically determined the parameters of the moves (speed, spacing between pokes, increase in depth between each sequence of pokes, etc.), and found that the optimal parameters are different for aerogels from different manufacturers.

To extract a particle impact, we first align the impact along the x-axis of the microscope stage. Then, we undercut the impact using a straight needle mounted at a shallow angle (typically 27°) to the horizontal. (Fig. 5). Using the same needle, we cut two small tunnels that we use in a later step for mounting the keystone in a micromachined fixture. We then cut the event out of the collector using a microneedle bent so that its tip is oriented normal to the collector surface. Any shape can be defined for this cut. Typically, we follow the particle impact track on one side, leaving $\sim 10\ \mu\text{m}$ of aerogel between the wall of the impact track and the vertical cut. This results in an aerogel sample (a “keystone”) that completely contains and conserves the impact event. The damage to the surrounding aerogel is minimized. As of this writing, we have extracted approximately 100 keystones from various aerogel collectors. Early failures were caused by insufficient care in aligning the needles. The failure rate with our current extraction procedure is negligible.

For analyses in which it is desirable to expose the residue on the inside wall of the track—for example, for scanning electron microscopy with energy dispersive spectroscopy (SEM-EDX) mapping of individual particles or microbeam two-step laser desorption mass spectroscopy ($\mu\text{L}^2\text{MS}$) organic analysis—it is also possible to cut vertically along the axis of the hypervelocity section of the track. (Fig. 6). This results in a “dissection keystone;” this approach has the advantage that, while half of the hypervelocity region track is extracted for analysis, the remainder of the track is left in the collector for later extraction. The terminal particle and any particle residue in the track whisker cannot be divided by this method and are either removed in dissection of the keystone or are left in the collector.

The extracted keystones are small, fragile, and susceptible to loss due to even very gentle air currents. Working with Christopher Keller at MEMS Precision Instruments (MEMSPI), we have developed a set of micromachined fixtures

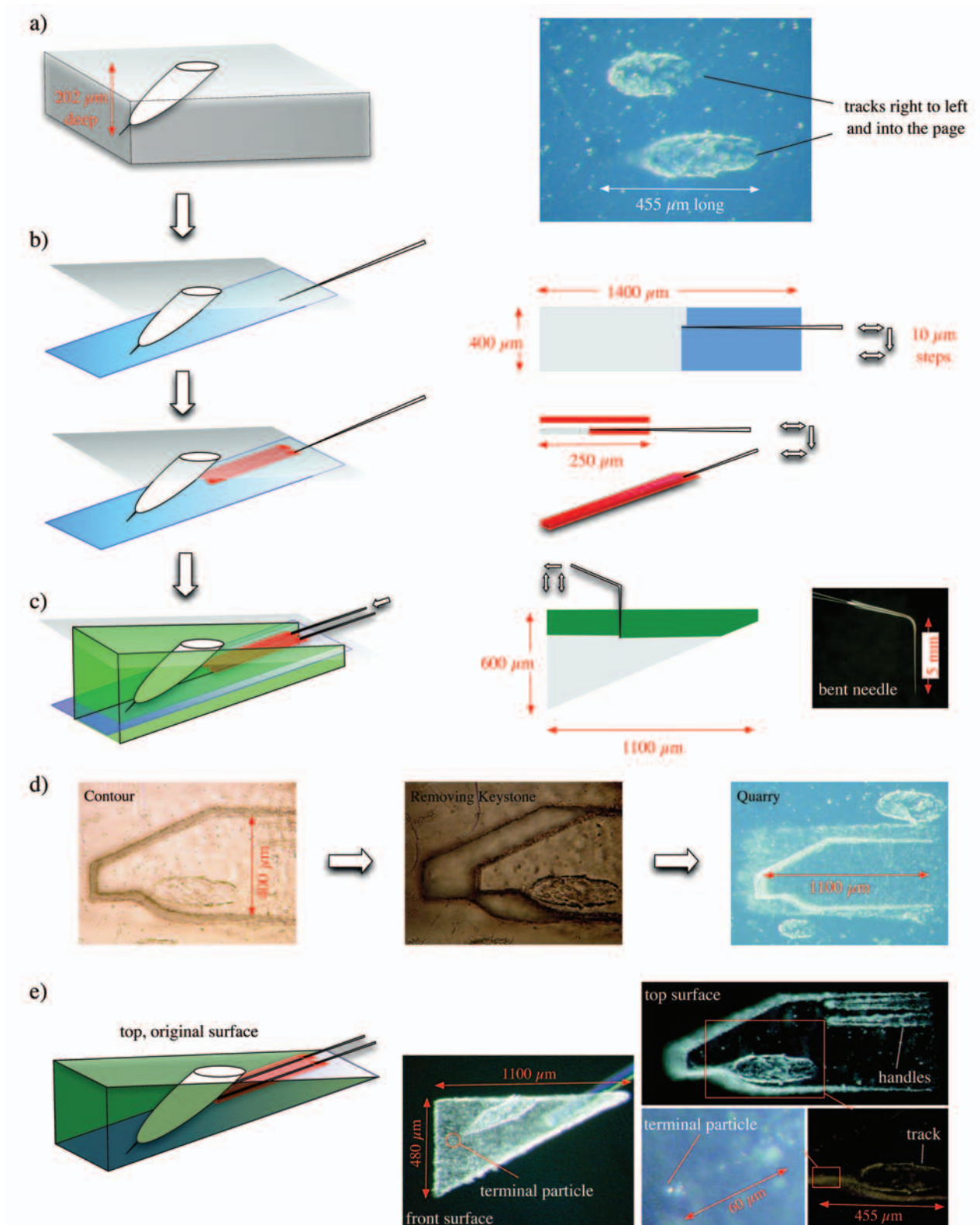


Fig. 5. Pictorial description of the procedure for complete extraction of impact residue in an aerogel keystone: a) original impact: in this case, a “chondritic swarm” impact in the ODCE collector (Mir); b) undercut of impact event and mining microforklift shafts; c) vertical contour cut and installation of microforklift; d) extraction from collector; e) final self-supporting keystone. The small terminal particle, though present in all images apart from that of the quarry, is visible only in part (e).

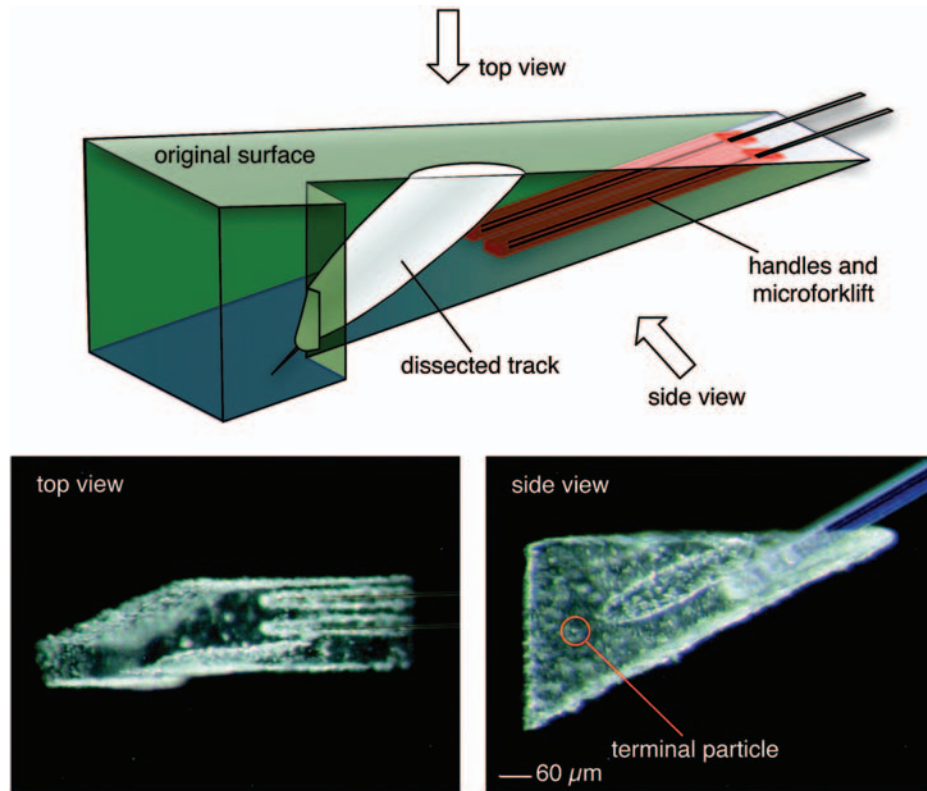


Fig. 6. Extraction of an impact track in a dissected keystone, exposing particle residue for analysis by surface techniques. The particle depth is 198 μm .

for keystones, which we call “microforklifts.” Keystones fixed on the microforklifts (Fig. 5) are self-supporting, so there is no need for mounting on any additional interfering substrate that could complicate some microanalytical techniques. The microforklifts, in turn, are mounted on 1 mm-diameter glass rods that can be readily handled.

Extraction of individual grain fragments at the track terminus or along the track may be required after the initial characterization of all preserved material within the aerogel keystone has been achieved (Fig. 7). We have demonstrated that individual particles can be extracted from keystones using custom-developed micromachined silicon microtweezers developed by us in collaboration with MEMSPI. These tweezers are able to routinely handle micron-scale particles. We are continuing the development effort on these tweezers, including an embeddable, encoded tweezer than can be used for precise positioning during microtoming. However, we repeat our cautionary statement here that exclusive extraction of individual, large particles may bias the analysis toward robust minerals.

SUMMARY OF TECHNIQUES AND SAMPLE PREPARATION METHODS

Several analytical techniques, e.g., scanning transmission ion mapping (STIM), proton elastic scattering analysis

(PESA), Rutherford backscatter (RBS) analysis, and proton-induced X-ray emission (PIXE) on the nuclear microprobe are described in a companion paper in this series; SXRF and synchrotron-based X-ray diffraction (SXRD) require no further sample preparation beyond the extraction of an event in a keystone and mounting on a microforklift. However, other analytical techniques require further sample preparation. We show a summary of techniques and sample preparation methods in Table 1.

Analytical Techniques Requiring Flattened Keystones

Secondary ionization mass spectrometry (SIMS) and resonance ionization mass spectrometry (RIMS) require physically flat samples because of the strong electric field at the sample. Because of its extremely short working distance, the nanoSIMS requires a particularly flat ($\leq 1 \mu\text{m}$) sample. For different physical reasons, FTIR analysis also requires a flat (or dispersed) sample to avoid interference effects. We have found that aerogel keystones can be readily flattened between glass slides without shattering. The collapse appears to be essentially homologous: the projected images of the keystones appear to be nearly identical before and after flattening (Fig. 8). For techniques in which material is ablated either slowly (e.g., nanoSIMS) or not at all (electron microprobe analysis, EMPA), it may be desirable to prepare a

Table 1. Summary of particle and impact residue sample preparation techniques for some common analytical techniques.^a

Analytical goal	Analytical technique	Sample preparation technique					
		Keystone (containment or dissection)	Flattened keystone	Microtomed flattened keystone	FIB-mined keystone	Microtomed/FIB-thinned particle	Aerofilm
Elemental	SEM/EDX	R	•	•	•	•	•
Composition	Nuclear microprobe (STIM/PESA/RBS/PIXE)	R	•	•	•	•	•
	EMPA	X	R	•	•	•	•
	SXRF ($Z \geq 6$)	R	•	•	•	•	•
Min/Pet	SXRD	R	•	•	•	•	•
	Modal mineralogy	R	•	X?	•?	X	X
	Tomography	R					
	FTIR	X?	R	•	•	•	X?
	Raman	•	•	•	•	•	•
	STEM	X	X	R	X	R	•
Chemistry	STXM	X	X	R	X	R	•
	FTIR	X?	R	•	•	•	X?
	$\mu\text{L}^2\text{MS}$	X	R	•	•	•	•
Isotopes	SIMS	X	R	•	•	•	•
	RIMS	X	R	•	•	•	•

^aSymbol key: R = minimum requirement; • = possible but not required; X = not possible.

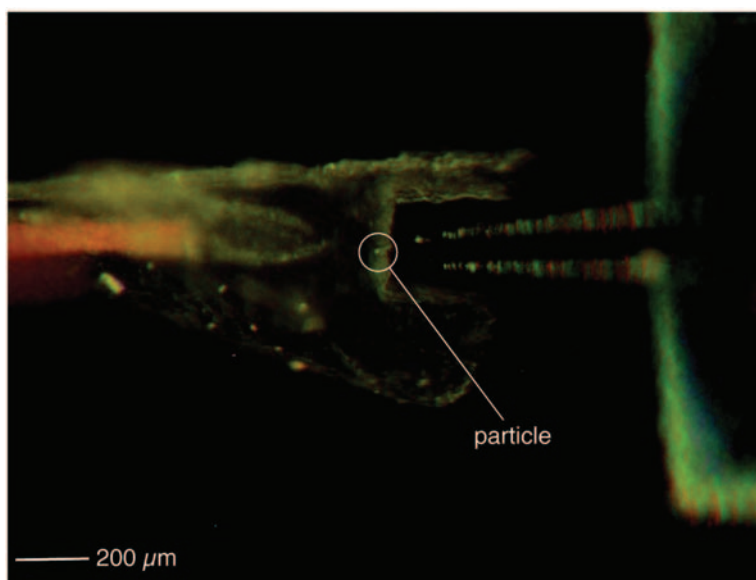


Fig. 7. An extraction of an individual particle from an aerogel keystone.

flattened dissection keystone, so that the particle residue is completely exposed for analysis.

Analysis Techniques Requiring Thin Sections

Scanning transmission electron microscopy (STEM) analysis requires ultrathin sections, which may be prepared

from aerogel keystones in several different ways. We have demonstrated that aerogel keystones may be flattened by a factor of ~ 50 , embedded in epoxy (e.g., EMBED 812), and ultramicrotomed. As a next step, we plan to develop the capability of ultramicrotomy of keystones embedded in sulfur. Individual grains, extracted using actuated microtweezers, may also be embedded and microtomed.

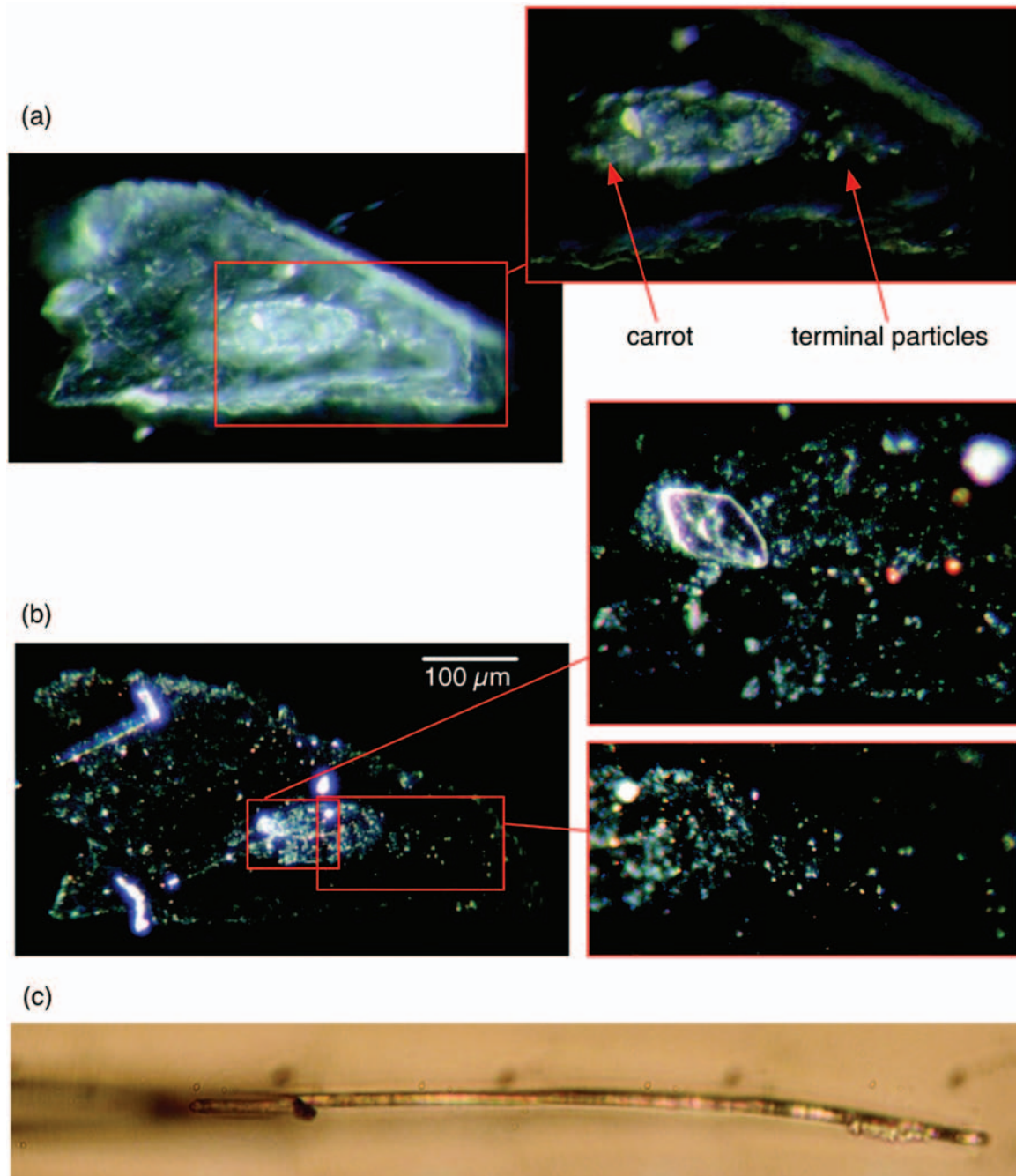


Fig. 8. Demonstration of flattening of an aerogel keystone containing a chondritic swarm impact. The keystone was flattened between two glass slides coated with non-adhesive coatings of carbon and Au + Pd, respectively: a) keystone before flattening. The thickest part of the keystone wedge (into the page) is $\sim 300 \mu\text{m}$ thick. Multiple terminal particles are visible in whisker tracks downstream of the carrot; b) face-on image of flattened keystone, held in microtweezers. In addition to the terminal particles, individual small particles distributed along the particle track can be resolved. The residual particles exhibit a striking variety of colors, so they appear to be heterogeneous; c) edge-on mosaic image of the flattened keystone held in a pair of micromachined tweezers. The flattened keystone thickness is $6 \mu\text{m}$, a factor of ~ 50 thinner than the unflattened keystone. The observed warpage in this keystone is not typically observed when flattening onto polished Al metal or EMBED 812 epoxy.

Graham et al. have demonstrated that thin sections from samples may be prepared using focussed-ion beam (FIB) milling (Heaney et al. 2001). We have demonstrated that individual thin sections of aerogel only $20 \mu\text{m}$ thick can be

extracted from aerogel collectors and placed into TEM grids for analysis (Fig. 9). This may be useful for the analysis of very small ($<100 \text{ nm}$) particulates in aerogel, in which embedding may be undesirable.

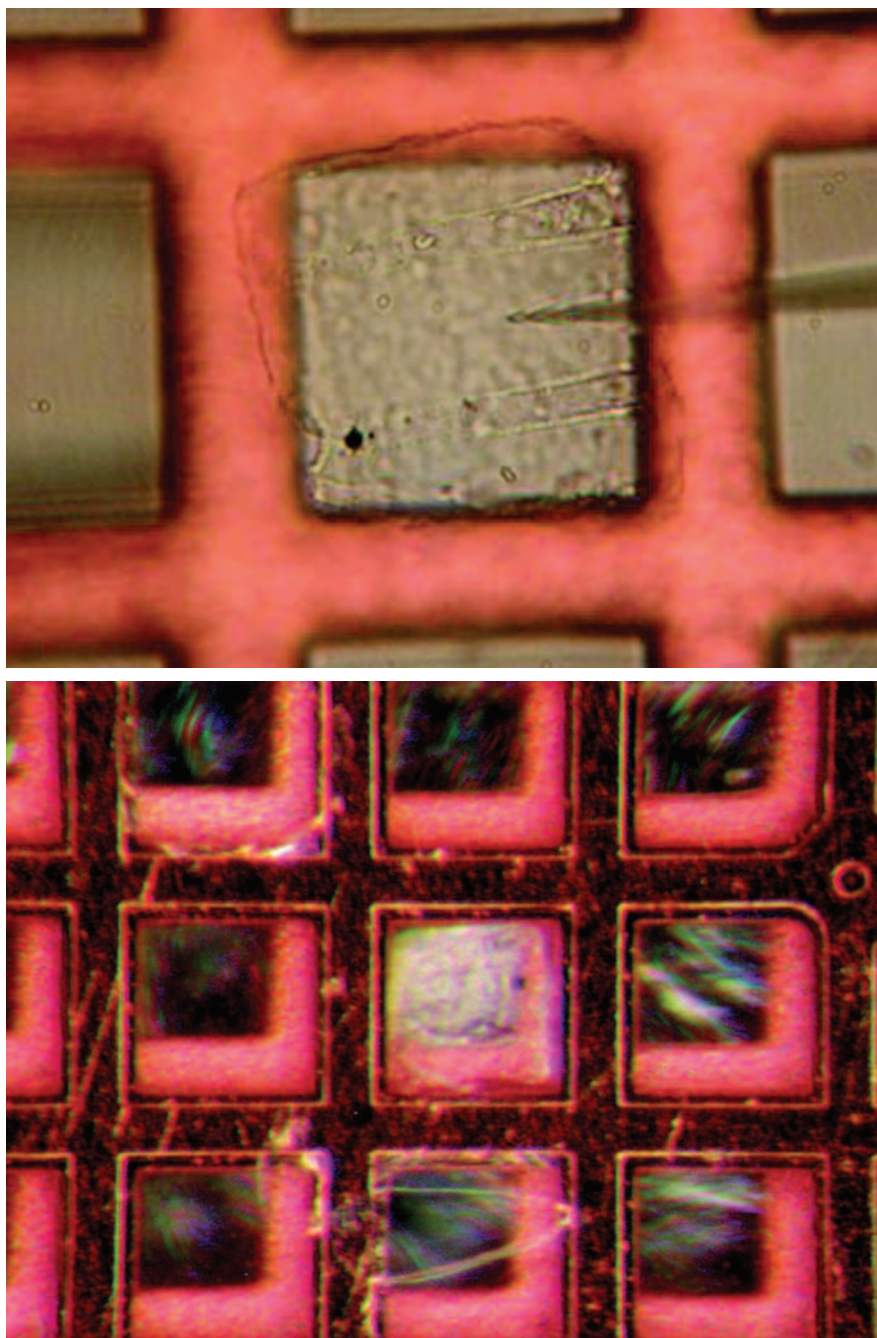


Fig. 9. A 20 μm -thick aerogel “film” placed into a 97 μm \times 97 μm TEM grid hole. Emplacement using a glass microneedle (top). An aerogel film trapped between two TEM grids (bottom).

Extraction of Interstellar Dust Impacts: Picokeystones

In addition to the cometary dust collector, the Stardust mission also carries an interstellar dust collector. Landgraf et al. (1999) have estimated that Stardust will collect 40 interstellar dust particles of a mass of 1 pg or greater. Because of the steeply falling mass distribution, most of these particles will be sub-micron in size. The identification, extraction, and analysis of these particles will be extremely challenging. We

have taken the first steps in the extraction of very small impact tracks that may be similar to those of IS grains. First, we machine away material on either side of a wedge-shaped volume of aerogel that contains the small track. Next, we extract the track from the collector in an ordinary keystone. The result is a stacked keystone (a “picokeystone,” since the projectile probably has a mass on the picogram scale) in which the large keystone serves as a carrier for the small keystone containing the track. (Fig. 10).

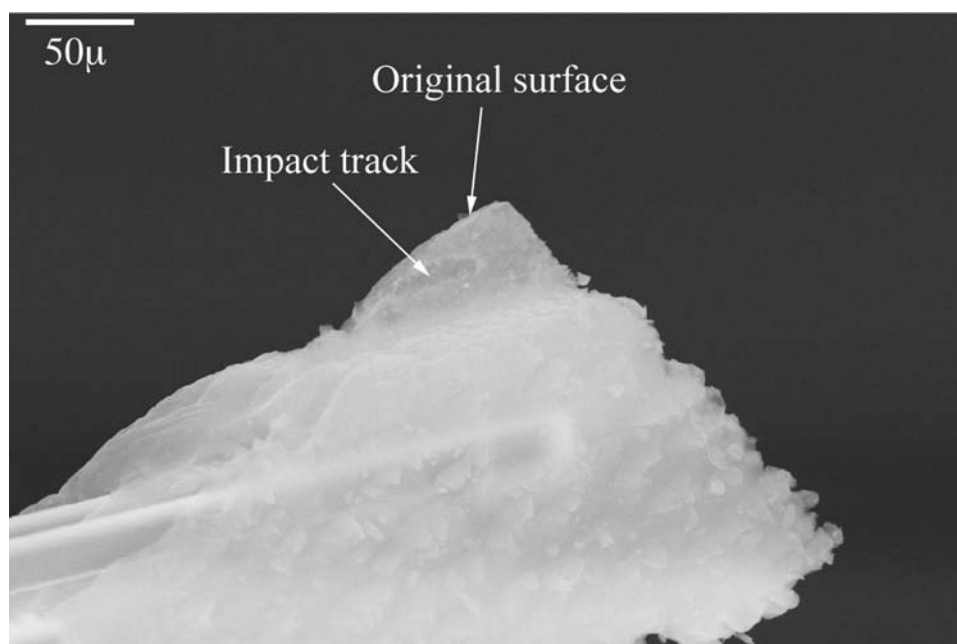


Fig. 10. A secondary electron micrograph of a $\sim 30 \mu\text{m}$ -long track extracted in a “picokeystone.” The track was not exposed on the surface; rather, the aerogel is so thin that it is effectively transparent to the electron beam. The track has approximately the length and geometry of that expected from an interstellar dust particle captured in the Stardust interstellar collector.

Extraction of Particle Impacts from Crowded Fields

It is possible that the number of particles collected by Stardust will greatly exceed the Stardust goal and that, if the collectors survive, we will be faced with the delightful challenge of harvesting a bonanza of captured cometary material. In this case, the collectors may be optically opaque near the surface, preventing the initial imaging and targeting of individual grains. A possible approach to the extraction of these particles would be to extract particles in a series of layered keystones. First, a small keystone would be extracted, which would contain small particle residues and only portions of the hypervelocity sections of tracks of larger particles. If this extraction removes the opaque surface layer, deeper particles might then be imaged and targeted for individual extraction. If not, successively deeper and deeper keystones could be extracted below the first, each containing the terminal particles and residues of larger and larger impacting particles.

CURRENT DEVELOPMENT EFFORTS

Here, we have described only some first steps in the development of sample preparation techniques for the analysis of the precious returned samples from Stardust. We are refining our extraction techniques to improve the accuracy and speed and are currently developing techniques for embedding particles using encoded, microtomable microtweezer tips so that particles can be readily located inside opaque embedding media. In this development effort, we have been guided by the broad scientific questions discussed in the introduction. Future

directions in sample preparation should be defined by the requirements of those analytical techniques that are required to address these questions.

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Editorial Handling—Dr. Scott Sandford

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