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Small hypervelocity particles captured in aerogel collectors: Location, extraction, handling and storage

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Abstract–It has now been about a decade since the first demonstrations that hypervelocity particles could be captured, partially intact, in aerogel collectors. But the initial promise of a bonanza of partially-intact extraterrestrial particles, collected in space, has yet to materialize. One of the difficulties that investigators have encountered is that the location, extraction, handling and analysis of very small (10 μm and less) grains, which constitute the vast majority of the captured particles, is challenging and burdensome. Furthermore, current extraction techniques tend to be destructive over large areas of the collectors. Here we describe our efforts to alleviate some of these difficulties. We have learned how to rapidly and efficiently locate captured particles in aerogel collectors, using an automated microscopic scanning system originally developed for experimental nuclear astrophysics. We have learned how to precisely excavate small access tunnels and trenches using an automated micromanipulator and glass microneedles as tools. These excavations are only destructive to the collector in a very small area—this feature may be particularly important for excavations in the precious Stardust collectors. Using actuatable silicon microtweezers, we have learned how to extract and store "naked" particles—essentially free of aerogel—as small as 3 μm in size. We have also developed a technique for extracting particles, along with their terminal tracks, still embedded in small cubical aerogel blocks. We have developed a novel method for storing very small particles in etched nuclear tracks. We have applied these techniques to the extraction and storage of grains captured in aerogel collectors (Particle Impact Experiment, Orbital Debris Collector Experiment, Comet-99) in low Earth orbit.

INTRODUCTION

About a decade ago, Peter Tsou demonstrated that hypervelocity particles could be captured, partially intact, in ultra-low density aerogel collectors (Tsou, 1990). This was an exciting development, since it constituted a new tool for collection of extraterrestrial particles, either in low Earth orbit (LEO) or in a (then potential) interplanetary sample-return mission. Previously, hypervelocity particle collectors in LEO consisted of ultraclean targets of ordinary density—principally metals and glasses—in which impacts resulted in craters and the melting or vaporization of the incident particles. Only residues of the particles could be analyzed in the melted rims of the craters. Particles captured in aerogels are relatively undamaged by comparison, so it was thought that a complete characterization—mineralogy, elemental and isotopic composition, *etc.*—might be performed. Also, it was hoped that this technique could provide a sample of extraterrestrial grains which did not suffer from the biases of stratospheric collection, which favors small, fluffy interplanetary dust particles (IDPs), or of Antarctic micrometeorites, which suffer from strong alteration due to atmospheric heating and residence in ice. It became clear rather quickly that this technique has its own bias—the survivability of grains during capture in the low-density aerogel depends in a poorly-understood way on the velocity and composition of the particles—but this bias was at least different than those of other techniques. Because of the potential for major advances in our understanding of interplanetary dust, in characterizing anthropogenic orbital debris, as well as the potential for capturing for the first time fresh interstellar particles, several projects have employed this new technology, including collectors flown on the shuttle (Tsou, pers. comm.), on a free-flying satellite (Brownlee *et al.*, 1994, and on *Mir* (Hörz *et al.*, 2000; Borg *et al.*, 2001; Westphal *et al.*, 1998; Ferrini *et al.*, 2001). The largest and most ambitious of these missions is Stardust (Brownlee *et al.*, 1996). The *Stardust* spacecraft carries two aerogel collectors: one will collect cometary particles during a close encounter with comet Wild-2 in 2004, and the other will collect interstellar particles during the cruise phase of the mission. The *Stardust* collectors will be returned to Earth in 2006.

Particles have been successfully extracted from aerogel collectors (Hörz *et al.*, 2000; Barrett *et al.*, 1992; Stadermann and Floss, 2000), but techniques for extracting captured particles from collectors have been rather crude, are destructive to the aerogel collectors, and are generally limited to the extraction of large (generally >>10 μm) particles. Furthermore, these techniques are not robust: particles may be lost in the process of extraction and subsequent handling, with the probability of loss rapidly increasing with decreasing particle size. Robust, near unit-efficiency extraction of particles smaller than 10 μm would greatly increase the statistics of interplanetary particle collection, and is probably required for analysis of fresh interstellar particles.

In this paper, we summarize our work in developing new techniques for locating, extracting, handling and storing aerogel-captured hypervelocity particles. This paper should be read rather as a progress report than as completed work—as we will describe, there is much yet to be done, particularly before the precious Stardust samples are to be touched.

For this development work we principally used collectors which have been exposed externally on the Russian space station *Mir*. The particle impact experiment (PIE), a NASA experiment, was exposed for 11 months in 1996–1997 on the outside of the Kvant-2 module, and included an aerogel collector with a collecting area of 40.5 cm^2 and density of 56 mg cm^{-3}. The orbital debris collector (ODC), another NASA experiment, was exposed for 18 months in 1996–1997, and included 72 100 cm^2 aerogel collectors with density of 20 mg cm^{-3}. Finally, the Comet-99 collectors were exposed during the Franco-Russian PERSEUS mission in 1998–1999. These four aerogel collectors similar to the PIE collectors were exposed during the Earth encounter of the Leonid meteor stream associated with comet Temple–Tuttle. We also used aerogel collectors, kindly provided by Peter Tsou, which were artificially implanted with particles from the Murchison meteorite grains using the light-gas gun at Ames Research Center.

AUTOMATED LOCATION OF GRAINS

The first aerogel collectors were scanned manually in optical microscopes to locate stopping hypervelocity particles. This process was laborious, and suffered from poor, unknown, and probably variable efficiency. At Berkeley, we have developed an efficient technique for locating hypervelocity particles captured in aerogel collectors. We use the existing Berkeley automated scanning systems, originally developed for rapid analysis of glass track-etch detectors used in nuclear astrophysics research (*e.g.*, Westphal *et al.*, 1991, 1996, 1998; Westphal and He, 1993). We have used the scanning system to locate particles in several aerogel collectors flown in space, including collectors flown on the shuttle (Tsou, pers. comm.), and the ODC, PIE, and Comet-99 collectors on *Mir* (Hörz *et al.*, 2000; Borg *et al.*, 2001; Westphal *et al.*, 1998; Ferrini *et al.*, 2001).

Summary of Particle Location Technique

Particles are located in the collectors indirectly, by *detection of particle tracks* rather than of the particles themselves. In Fig. 1 we show a schematic of the automated scanning system and the detection technique. The scanning system consists of a Leitz microscope with long working distance objectives, a charged coupled-device (CCD) camera, a real-time image processor, a Sparc computer, and a computer-controlled stage. The aerogel collector is first placed on the stage, and an accurate (~2 μm) map is made of the collector's upper surface. The entire surface of the collector is then scanned in a raster pattern, using the measured surface heights interpolated using a cubic spline to determine the local surface heights. The microscope is locally focused at a fixed distance *below the collector surface*. Any particle track that intersects this focus plane is detected.

The detection method was first developed for the analysis of glass nuclear track-etch detectors; we describe it in detail in Weaver and Westphal (1998). Here we provide a summary. The video signal from the CCD (Fig. 2) is read by the image processor, which computes a gradient image (Fig. 3). The coordinates of all pixels above a certain preset threshold are then sent to the Sparc computer. The subsequent treatment of

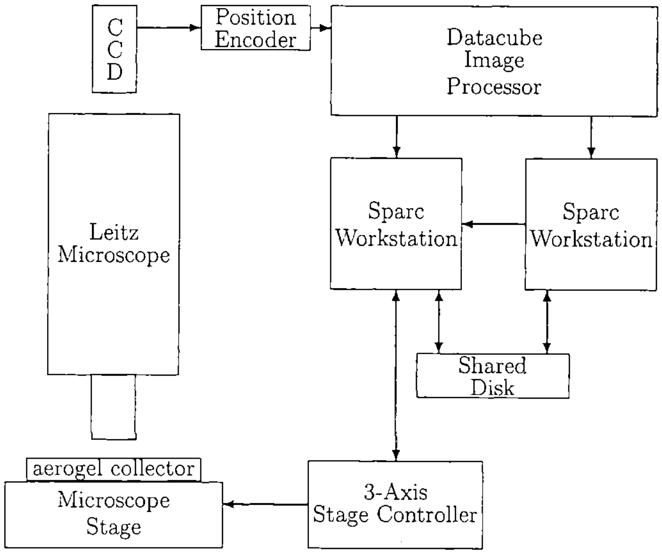

FIG. 1. Schematic layout of the automated scanning system.

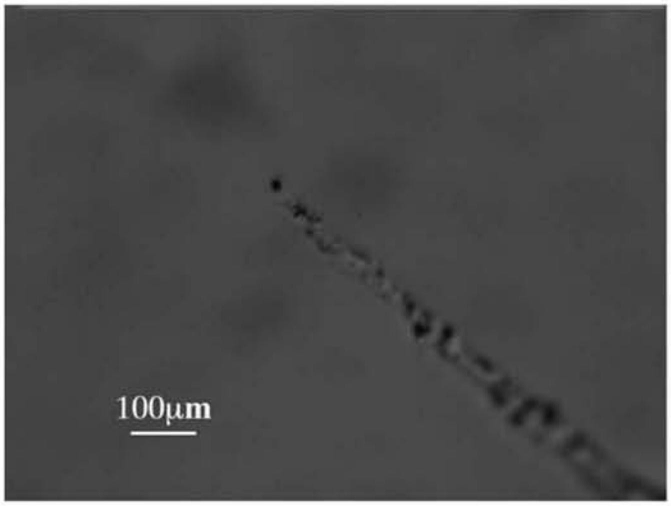

FIG. 2. Unprocessed image of a hypervelocity particle captured in aerogel in orbit, located by the automated microscopic scanning system.

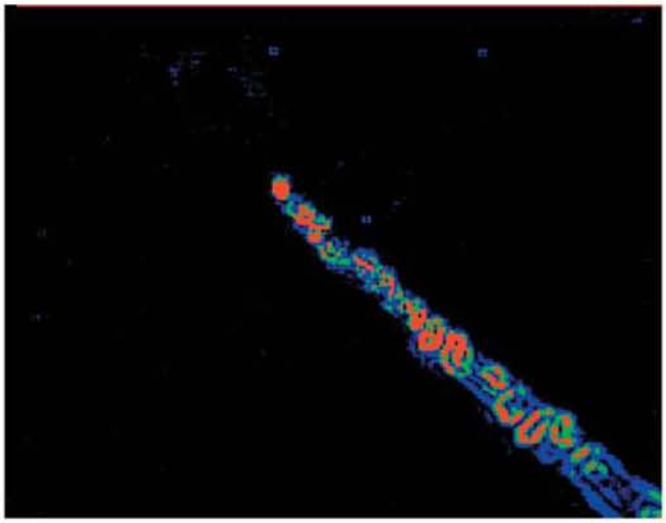

FIG. 3. Real-time gradient image of the track of the same particle. The presence of the track is unambiguous, and is trivial to sense by the scanning software.

the pixel coordinates then depends on the application. For scanning of aerogel collectors, the pixels are sorted into *clumps* of adjacent pixels, using an algorithm developed by Dan Snowden-Ifft, and the sizes and barycenters of the clumps are recorded in a file. This is the level 0 dataset. A typical scan may contain up to ~10^5 recorded clumps, most of which are due to background, as we describe below. Because a track may be recorded as several independent clumps, we further analyze the data—after completion of the scan—by grouping the recorded clumps ("clumping the clumps") using an algorithm based on the minimal spanning tree algorithm. This step is typically performed twice, which reduces the dataset to only a few thousand clumps—this is the level 1 dataset.

In a typical scan, the vast majority of the detected clumps are due to very small—usually unresolvable—inclusions in the aerogel collectors. To go back to examine each of these would be very tedious indeed, so we reduce this background by repeating the original scan, but at a slightly different depth, typically 20 μm above or below the first. Because the depth of field of the scanning system is only a few microns, such inclusions will be detected in only one of the scans, but particle tracks will be detected in both scans as long as the particle stops within the depth-of-field of the lower of the two focus planes. We use a fast matching algorithm to find coincidences between the two processed (level 1) datasets—these are the level 2 positions.

Typically, level 2 data are still dominated by background, consisting principally of cracks, large inclusions (Fig. 4), and diffracted, out-of-focus images of objects on the aerogel surface. These backgrounds cannot be discriminated from hypervelocity particle tracks automatically, but must be examined by an operator. To do this efficiently, the level 2 coordinates are ordered using the minimal spanning tree algorithm to make a nearly minimal total distance tour of the candidates. The scanning system then automatically follows this tour, and records an image of each candidate to disk. After the scan, we rapidly scan the images on a video monitor to identify the tracks of particles, which are easily recognized by eye. We record the coordinates of these candidates (level 3 coordinates), then physically return to these locations to confirm our identification of the candidates as hypervelocity particle captures. Level 4 data consist of the coordinates of actual particle impacts. In Fig. 5, we show examples of particles located in the PIE collector using this technique (Westphal *et al.*, 1997).

Sensitivity

To estimate the sensitivity of the scanning system, we have examined the tracks of submicron (~0.2 μm) carbonyl-iron particles implanted in aerogel by Ralf Srama using the Tandem Van de Graaf accelerator at Heidelberg. We chose a subset of the implanted particles whose tracks were smooth and had diameters too small to be resolved in the microscope. This is a distinct, probably low-velocity, population which did not produce the flared "carrot" tracks characteristic of hypervelocity particles. In Fig. 6, we show the raw and processed image of a typical track. The scanning system appears to be sensitive to the tracks of even submicron particles, but we do not currently have an aerogel sample implanted with carefully calibrated particles of known size and velocity, which would allow us to reliably determine a lower detection threshold.

AEROGEL EXCAVATION

A captured particle must be at least partially exposed before it can be extracted from an aerogel collector. Using a high-

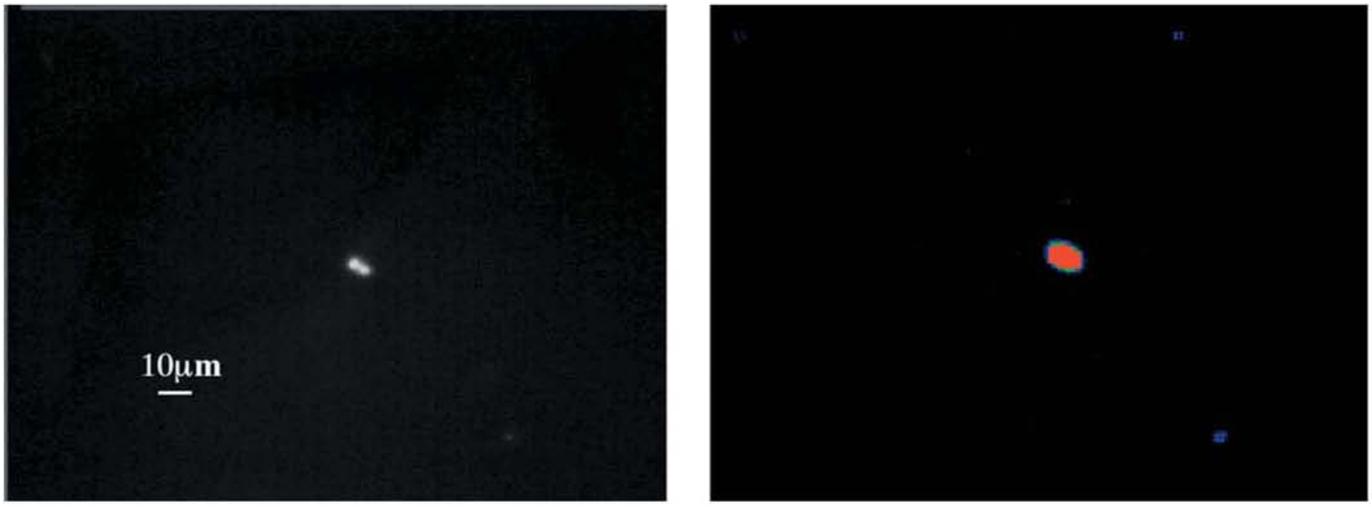

FIG. 4. Unprocessed image (left) and gradient image (right) of a large inclusion in aerogel.

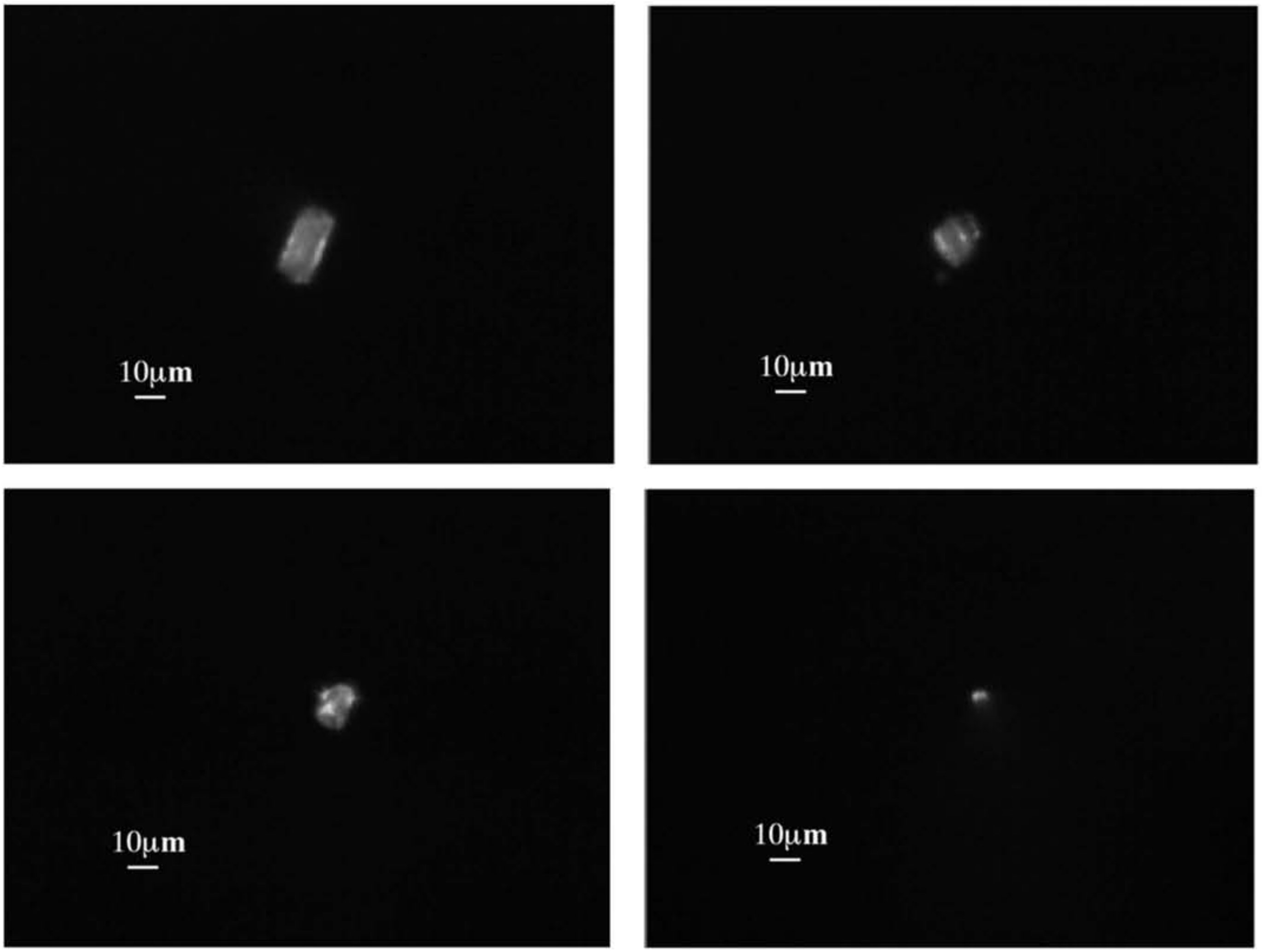

FIG. 5. Hypervelocity particles captured in aerogel onboard *Mir*, ranging in size from 10 to <2 μm in major dimension.

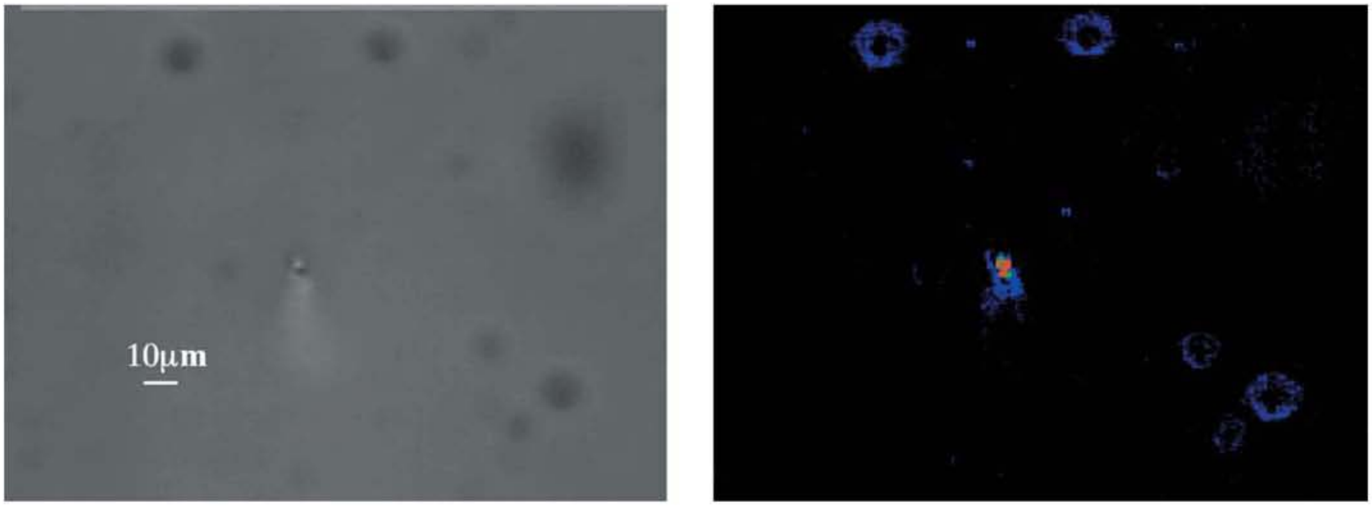

FIG. 6. Unprocessed image (left) and gradient image (right) of the track of an apparent low-velocity, submicron carbonyl-iron grain.

precision (200 nm) three-axis micromanipulator and pulled glass microneedles, we have learned how to excavate rectangular mineshafts and trenches in aerogel collectors to gain access to captured particles. The excavation is accomplished using repetitive computer-controlled robotic motion of the micromanipulator. The microneedle penetrates the aerogel by repetitive axial motions at a shallow angle. Penetration positions are located on a grid, with penetration points very finely spaced near the central axis of the mineshaft and successively coarser towards the walls. Excavation of a tunnel or a trench—a tunnel with no roof—is accomplished by repeating the grid pattern at successively deeper and deeper levels. The repetitive penetration by the mining tool breaks the aerogel up into a very fine fluffy material, which is removed from the excavated volume by automated, transverse sweeping motions of the excavation tool and by gently blowing with compressed air.

The next steps depend on the type of analysis planned for the particle. We have developed two techniques for extraction of particles: either the particles can be extracted "naked", that is with a minimum of surrounding or attached aerogel, or they can be excavated, along with a terminal segment of the particle's track, in a small (typically 100 μm) aerogel block.

Technique for Extraction of Particles and Tracks in Small Aerogel Blocks

If the particle is to be extracted within an aerogel block, a vertical wall is created 50 μm from the particle, and auxiliary shafts are dug on either side, leaving a column of aerogel containing the particle. The collector is rotated 90°, and the base of the column is cut using a narrow trench. The block, containing the particle and the terminal track, can then be extracted using microtweezers (see next section). One such mineshaft is shown in Fig. 7. This approach minimizes damage to the aerogel collectors: aerogel adjacent to the excavated mineshaft is undisturbed. In this example, the mineshaft is sufficiently wide that the microtweezer can open to its full extent without touching the walls, but mineshafts could be made narrower in precious samples to minimize damage to the collector. Excavation is accomplished in a few hours of mining, most of which is automated so does not need the attention of an operator.

Technique for Extraction of "Naked" Particles

If the particle is to be extracted "naked", a trench is dug with the floor ~10 μm above the particle. The optical quality of the trench floor is typically nearly identical with the original aerogel surface, so the particle can be easily imaged after the trench is made. The entire collector is then rotated, and the remaining few microns of aerogel is pushed aside by the tool using manual control.

PARTICLE EXTRACTION

Silicon Actuatable Microtweezers

Christopher Keller, working at the Berkeley Sensor and Actuator Center at Berkeley, has developed actuatable silicon microtweezers, with the principal goal of assembling micromachined components into micromachines (Keller, 1997). Figure 8 shows details of one such pair of tweezers. These microtweezers can be made to be either normally open or normally closed. We have employed Keller's microtweezers, fitted with custom-designed tips, to extract grains from aerogel collectors. In Figs. 9 and 10 we show naked particles and particles still embedded in small, aerogel blocks extracted using the microtweezers. We use normally closed microtweezers to

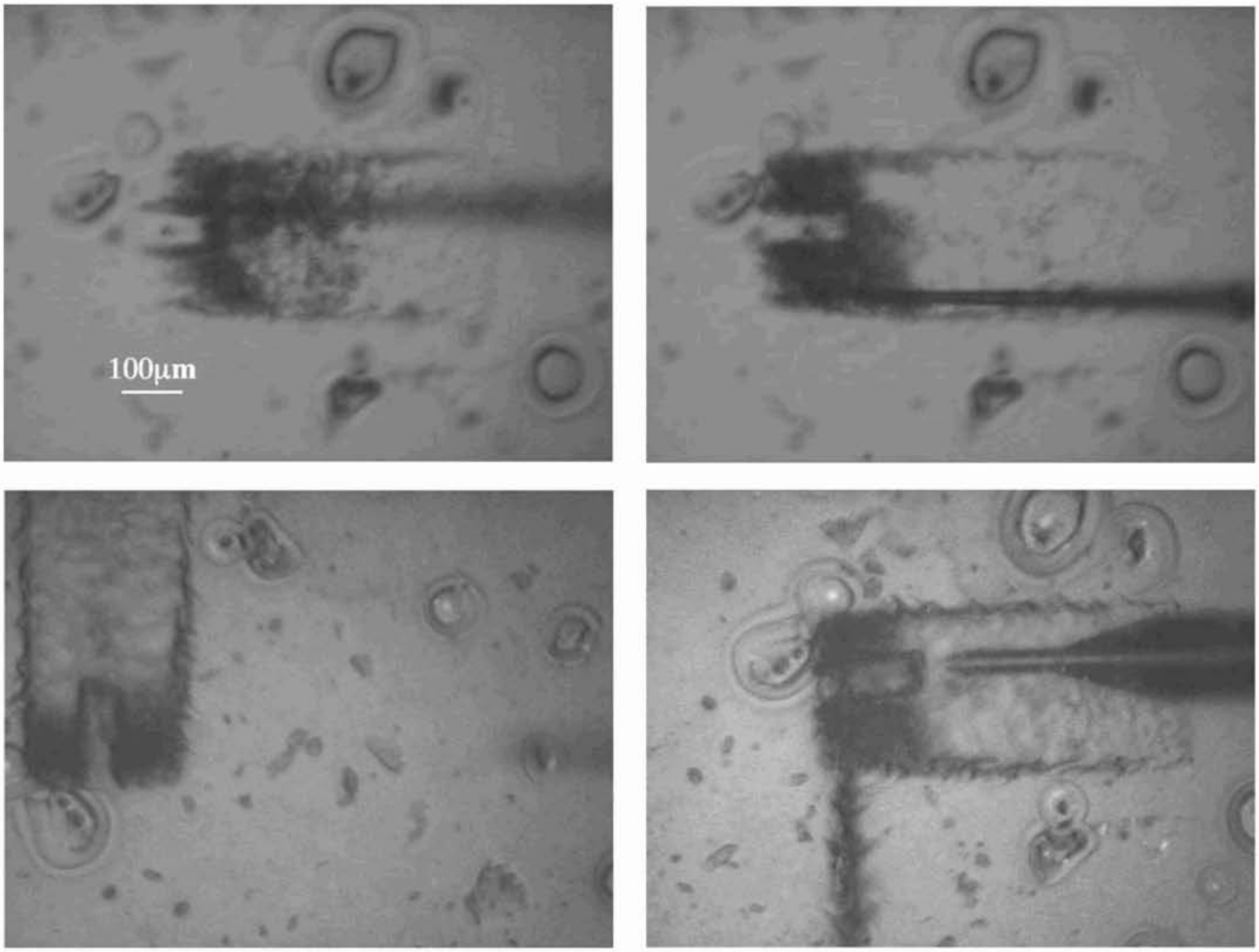

FIG. 7. Excavation of a 200 μm wide access trench in the *Comet-99* collector. The target particle is embedded in the column in the vertical wall on the left. (Upper right) first moves; (upper left) isolation of a small aerogel block; (lower right) preparation for amputation of the aerogel block from the trench wall; (lower left) preparation for extraction of block. Note that aerogel adjacent to the excavated mineshaft is undisturbed.

maintain a secure grip on extracted particles. Since the tweezers are made using standard very large scale integration (VLSI) technology, several hundred could be manufactured on one chip—they would be inexpensive and expendable if made in quantity. Indeed, they would be so inexpensive that one tweezer could be dedicated to each particle. We plan to take advantage of this feature to use tweezers for embedding grains, as we describe later.

Dry transfer techniques are required for most of the analytical techniques that are used in the analysis of small extraterrestrial particles (for a review of techniques, see Zolensky *et al.*, 2000). For micro-infrared and micro-Raman spectroscopy, particles must be transferred to a KBr window, or, for temporary storage and transport, into a well in a glass or quartz slide (see below). These media may also be used for scanning electron microscope (SEM) and energy dispersive x-ray spectroscopy (EDX) analyses (Fig. 11). (Here we point out that it is also possible to do *in situ* micro-Raman spectroscopy on embedded particles as demonstrated by Burchell *et al.*, 2001 and Kearsley *et al.*, 2001.) It is highly desirable that no liquids be used to facilitate transfer from tweezer to window. Since gravitational forces are negligible on this length scale, it is not possible to transfer the particles simply by opening the tweezers and allowing them to drop to the surface. Instead, the particles must be gently but robustly transferred. We have found that micropipettes, mounted in a second micromanipulator, are excellent tools for this task.

PARTICLE STORAGE

Extracted particles must be stored. Particles which are to be microtomed can be conveniently embedded in epoxy or sulfur, but many analysis techniques (*e.g.*, micro-Raman, x-ray

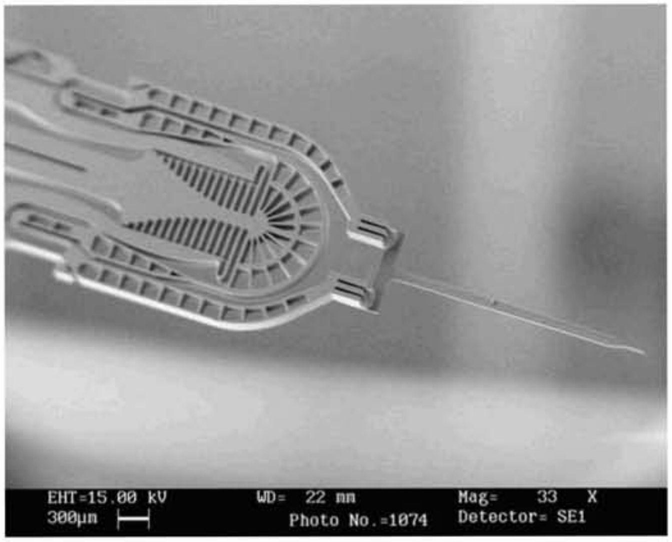

FIG. 8. Silicon actuatable microtweezer used for extraction of particles from aerogel collectors.

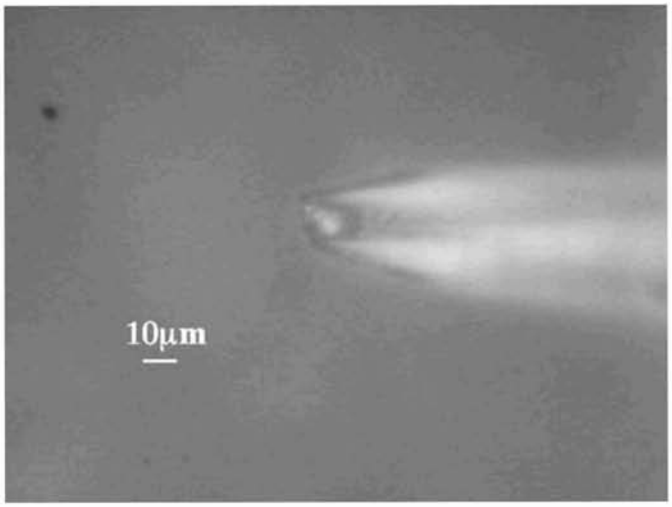

FIG. 9. Extraction of a naked particle from aerogel collector using silicon microtweezers.

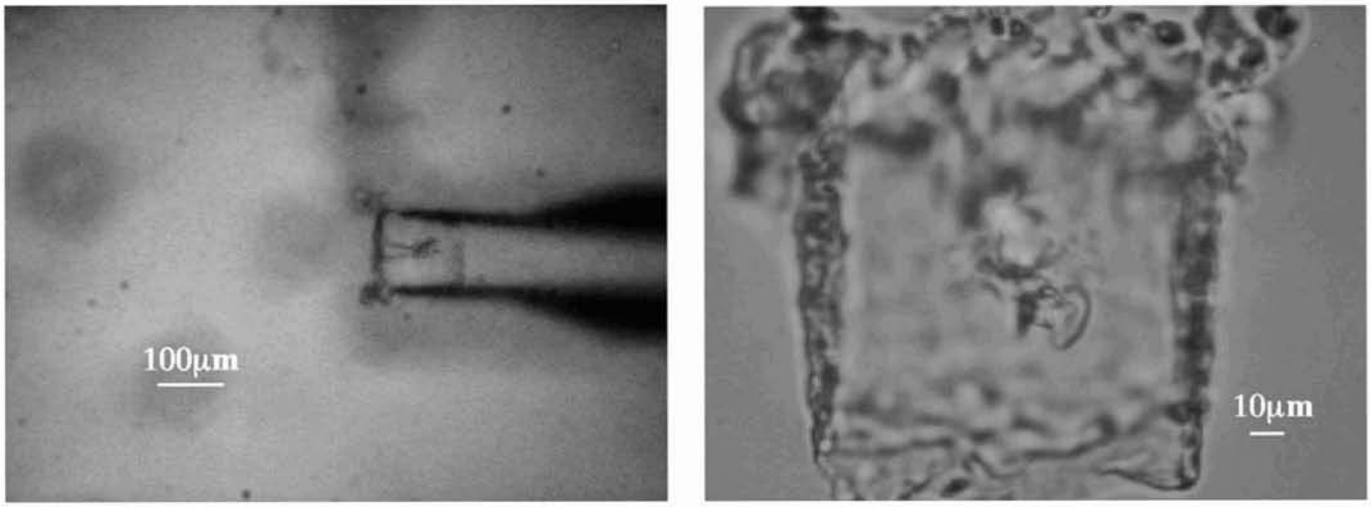

FIG. 10. Exposure and extraction of a particle, along with its track, in a small aerogel block.

fluorescence spectroscopy (XRF), SEM, EDX) require dry, that is, unembedded, particles. Here we describe dry storage methods that we have developed for extracted particles of various size ranges.

Dry Storage of Large Particles and Aerogel Blocks: Kapton Buttons

Large particles ($>10\ \mu$m) and aerogel blocks may be stored in kapton "buttons"—circular kapton disks manufactured as electrical insulators for transistors by Wakefield Engineering. The disks are $120\ \mu$m thick, and each includes four holes $1000\ \mu$m in diameter. The disks are epoxied to a microscope slide. The particle is then placed into one of the holes (Fig. 12), a glass cover slide is placed over the assembly and clamped in place. If the particle moves to the cover slip during transport, it is still easily located after separation of the slides because of its large size.

Small Particles: Etched Nuclear Tracks

Particles smaller than $\sim 10\ \mu$m can be stored in etched nuclear tracks in glass or quartz microscope slides. The etchpits are manufactured by etching the tracks of fission fragments from a ^{252}Cf source. These wells are $\sim 15\ \mu$m deep, and may be made with almost any diameter. The particles shown in Figs. 13 and 14

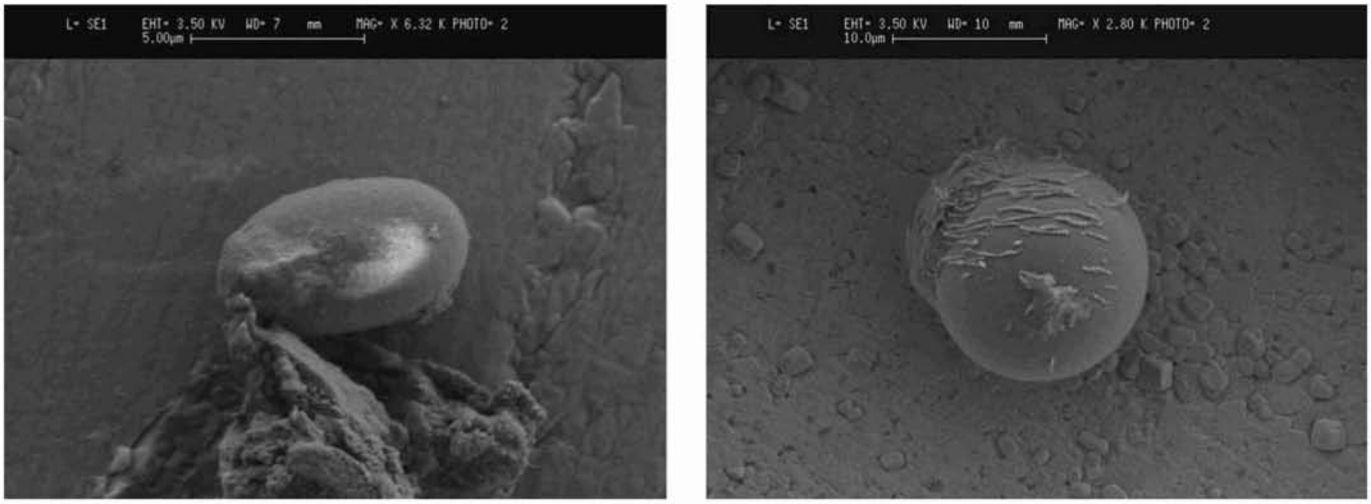

FIG. 11. Two particles extracted from the Napoli *Comet-99* collector, stored on a KBr window.

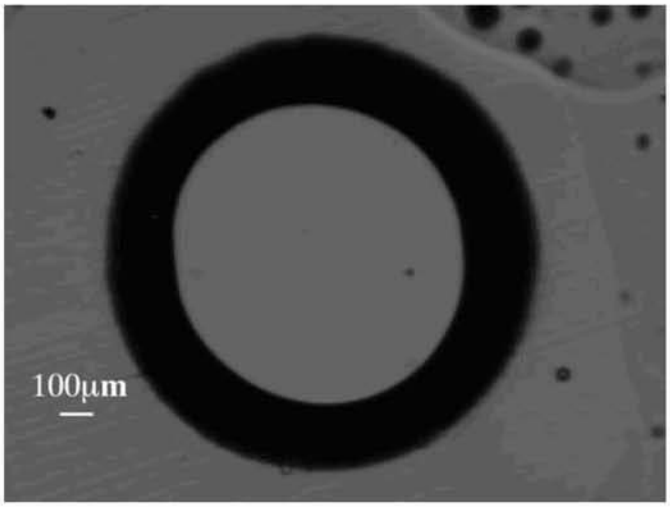

FIG. 12. A large (~25 µm) particle stored in a kapton button on a microscope slide.

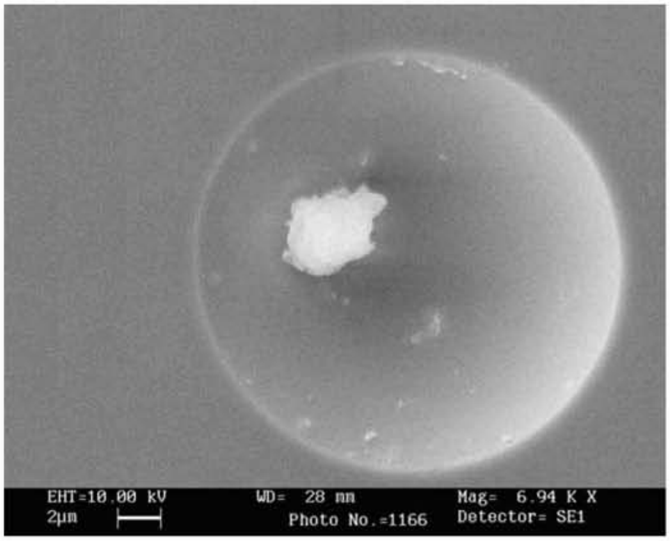

FIG. 13. PIE particle (PIE 003) stored in an etched nuclear track in a glass microscope slide.

are stored in such etchpits. These etchpits are ideal for robustly storing and transporting small particles. We have also demonstrated that such etchpits can be readily produced in quartz slides.

FUTURE DEVELOPMENT

Increasing Scanning Speed

We currently scan at a rate of ~1 $\text{cm}^2\ \text{h}^{-1}$. This speed is not onerous, but it is inconvenient. For scanning glass track-etch detectors, we use a synched stroboscopic illumination system which enables us to scan at ~6 $\text{cm}^2\ \text{h}^{-1}$ at the same magnification, but this strobe is not bright enough to scan aerogel collectors. By purchasing a brighter strobe, and using a larger CCD camera and a modern image processor, we estimate that we should be able to achieve a speed of at least 10 $\text{cm}^2\ \text{h}^{-1}$.

Automated Mining Techniques

We have performed the first demonstrations of precision excavation of mineshafts in aerogel collectors using pulled microneedles. However, the current technique has two principal limitations. First, although the time required to excavate a mineshaft is not unreasonable—less than a day—we expect that the time could be reduced considerably by experimenting with different excavation parameters (grid spacing, depth increment, micromanipulator speed, *etc*.). Second, we have found that during the course of the several thousand repetitive robotic

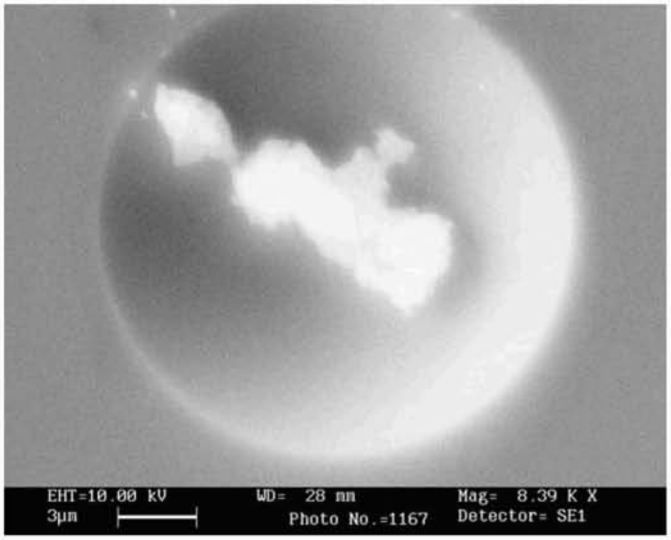

FIG. 14. Particle extracted from the ODCE collector stored in an etched nuclear track in a glass microscope slide.

motions required for an excavation, the micromanipulator drifts by a few microns. This limits the clearance of the floor of the mineshaft with respect to the target particle. As a result, we occasionally extract a small volume of aerogel (a few microns in major dimension) along with the particle. Using the existing image processor, we plan to implement a rezeroing operation which will automatically rezero the micromanipulator every few minutes during excavations. We anticipate that this will allow us to reduce to floor-to-particle clearance distance dramatically, and allow us to extract grains practically aerogel-free.

Tweezer Development

Improved Tweezer Tips for Handling Very Small Particles–Building on our experience in using microtweezers for extraction of particles from the PIE samples, we plan to develop and employ two different styles of tweezers, each designed specifically for the two major analysis techniques expected to be employed for most extraterrestrial particles. We plan to employ tweezer tips for extractions which are as closely matched to the particle size as possible, with tips as small as 1–2 μm.

Passive Gripping Force Limitation–Although particle breakage due to crushing has not, so far, been a problem, we plan to passively limit the gripping force of the tips by incorporating flexible force-limiting segments into the tips. The maximum gripping force may be specified in the design of the tip by specification of the width and length of the flexure. This feature will be incorporated in any microtweezers to be purchased in the future for particle extraction, including the two versions discussed below. We also plan to investigate the feasibility of tweezers with *active* force feedback, which may facilitate the handling of very fragile particles.

Tweezers for Epoxy Embedding–Particles which will be microtomed for transmission electron microscopic (TEM) analysis are first embedded in epoxy or sulfur. For relatively large particles (>10 μm), this is a relatively straightforward procedure, but for small particles, the probability of missing the particle during microtoming becomes substantial. We plan to develop tweezers which can be used to directly implant the extracted particle into embedding epoxy, and to track the particle during microtoming. Since the microtweezer tips can be made in large batches, they will be inexpensive in large quantities, and the tips will be embedded along with the particle that they grasp.

These tweezers will use a tweezer body identical to those in existing models. However, the tweezer tips will have two novel features. First, the tips will be made of SU-8 spin-on epoxy (SOE), so that the embedded tips can be microtomed reliably—polysilicon tips are too brittle to be microtomable. This is a standard technology, and can be implemented in tip manufacture. Second, a pattern of narrow notches will be incorporated into the tips. This pattern encodes position with respect to the tweezer tip, so that any microtomed slice can be read to determine the current distance of a slice from the tweezer tip. (The position of the particle with respect to the tip will be measured microscopically or with an SEM before embedding.) The resolution of the position encoder will be <1 μm. In order to detect a rotation of the tweezer tips with respect to the microtoming plane, position encoders will be built into the top and bottom surfaces of each tweezer tip. Resolution in detecting tilt is expected to be <1°.

We have verified that a SOE tip embedded in EMBED 812, a standard embedding epoxy, is trivially visible in reflected light in a polished sample. We have also recently verified that the SOE tip in EMBED 812 is visible in a microtomed thin section (Fig. 15). We have not yet tested the feasibility of microtoming tweezer tips embedded in sulfur.

In this development effort, we will concentrate on extracting, embedding, and microtoming the smallest particles possible, keeping firmly in mind the upcoming challenge of extracting submicron interstellar particles from the *Stardust* interstellar dust collector. The current state of the art for extraction is limited to particles ~5 μm in diameter or larger: we will attempt to push this limit down by as much as an order of magnitude. We are confident that submicron particles can be extracted, along with a small volume of aerogel, using the excavation and extraction techniques under development. For microtoming such small particles the position-encoding feature of the tweezers will be critical in order to accurately locate the particle in thin sections.

CONCLUSION

Here we have demonstrated that grains as small as 3 μm can be located, extracted and stored robustly in preparation for analysis and characterization. To be sure, a number of difficulties remain to be addressed. The efficiency of this

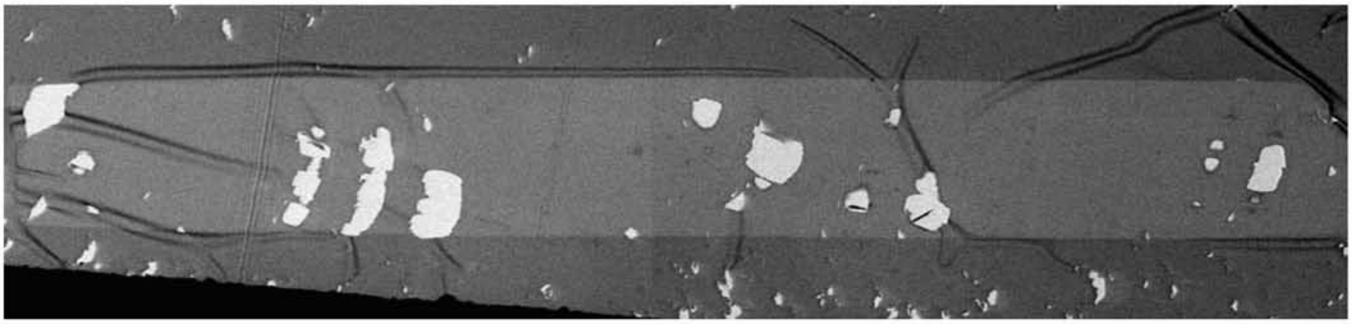

FIG. 15. Microtomed ~90 nm thin section of a 190 × 25 μm SU-8 beam in EMBED 812.

technique for extraction of "naked" particles larger than ~5 μm is near unity, but for smaller grains we do not yet have sufficient statistics to accurately measure the success rate as a function of size. Although we have not yet reached the lower size limit on grains which can be extracted, we suspect that grains much smaller than 1 μm would be impractical to extract "naked" using these techniques. But since current analytical techniques are limited to much larger grains, this challenge may be safely deferred for the future. Among the other improvements that we have already described, we plan to attempt to microtome epoxy-tipped tweezers embedded in sulfur. Since particles cannot be imaged in sulfur during microtoming as they can in epoxy, this technique could lead to substantial improvement in the precision of microtoming.

Here we have focused on the extraction of grains from aerogel collectors. These techniques may be used not only for *Stardust* and the aerogel collectors that we have presented here. Recently, Leshin *et al.* have proposed to collect and return martian dust using aerogel collectors (Leshin *et al.*, 2001). These techniques may also be applied generically to the handling of very small grains in almost any context, such as recovery of small particles from "flying carpet" collector of the proposed Aladdin mission to collect regolith from Phobos and Deimos.

Acknowledgements–In pursuing this work, we have been inspired by a comment made by Robert Walker several years ago in an paper about a potential cometary sample return mission (Walker, 1991):

> "...a certain fraction of the cost of mounting sample return missions should be set aside to stimulate instrument development here on Earth. This would maximize the integrated scientific return from the missions; perhaps more important, such investment would increase the benefits to technology and science as a whole. Unless such a policy is adopted, I am afraid that our community will not play as key a role in the future development of advanced analytical techniques as it could and should."

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