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Report

Rapid extraction of dust impact tracks from silica aerogel by ultrasonic microblades

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Abstract–In January 2006, NASA's Stardust mission will return with its valuable cargo of the first cometary dust particles captured at hypervelocity speeds in silica aerogel collectors and brought back to Earth. Aerogel, a proven capture medium, is also a candidate for future sample return missions and low-Earth orbit (LEO) deployments. Critical to the science return of Stardust as well as future missions that will use aerogel is the ability to efficiently extract impacted particles from collector tiles. Researchers will be eager to obtain Stardust samples as quickly as possible; tools for the rapid extraction of particle impact tracks that require little construction, training, or investment would be an attractive asset. To this end, we have experimented with diamond and steel microblades. Applying ultrasonic frequency oscillations to these microblades via a piezo-driven holder produces rapid, clean cuts in the aerogel with minimal damage to the surrounding collector tile. With this approach, intact impact tracks and associated particles in aerogel fragments with low-roughness cut surfaces have been extracted from aerogel tiles flown on NASA's Orbital Debris Collector (ODC) experiment. The smooth surfaces produced during cutting reduce imaging artifacts during analysis by scanning electron microscopy (SEM). Some tracks have been dissected to expose the main cavity for eventual isolation of individual impact debris particles and further analysis using techniques such as transmission electron microscopy (TEM) and nano-secondary ion mass spectrometry (nanoSIMS).

INTRODUCTION

A means of efficient extraction of particles from silica aerogel collectors is necessary for the science return of Stardust. This NASA sample return mission, the first from a comet, returns to Earth in early 2006 with its valuable cargo of comet dust that was captured at hypervelocity speeds in aerogel during passage through the coma of Comet Wild-2 (Brownlee et al. 2003; Tuzzolino et al. 2004). In addition to Stardust, aerogel is a likely collector for future sample return missions and is used for capture of hypervelocity ejecta in high-power laser experiments of interest to the Lawrence Livermore National Laboratory (Tobin et al. 2003). Researchers will be eager to obtain Stardust and other aerogel-captured samples for study as quickly as possible, so simple, rapid extraction tools that require little training and have low construction costs would be an attractive asset. Since particles at the termini of impact tracks are expected to be the least altered by impact processes, there is considerable interest in means of extracting individual particles for detailed analysis. In addition, it is important to study intact impact tracks together with their accompanying terminal particles to address the questions of how material is distributed along the impact track and how representative the terminal particle is of the original particle prior to hypervelocity impact. One method for extracting intact cosmic dust impact tracks from silica aerogel tiles is a mature procedure involving sequential perforation of the aerogel with glass needles on computer-controlled micromanipulators (Westphal et al. 2002, 2004). This method is highly successful at removing well-defined aerogel fragments of reasonable optical clarity without causing damage to the surrounding aerogel collector tile. In an attempt to speed up and simplify the process of impact track extraction and eventual isolation of terminal particles for Stardust, we have experimented with microblades. Our ultimate goal is a rapid extraction system in a clean electron-beam environment, such as a scanning electron microscope (SEM) or dual-beam focused ion beam system (FIB), for in situ sample preparation, mounting, and analysis.

We have found that piezo-driven ultrasonic frequency (U/S) oscillations applied to very sharp, thin blades generate rapid cuts of unprecedented smoothness with minimal damage to surrounding aerogel. With this ultrasonic microblade extraction technique, still under development, we have extracted several impact tracks from aerogel tiles from NASA's Orbital Debris Collector (ODC) experiment (Hörz et al. 2000) exposed on the Mir Space Station to study the particle environment in low-Earth orbit. Additional tracks were dissected into two halves exposing the main track cavity. Complete or dissected tracks can be extracted in less than an hour. Due to the smooth cut surfaces, impact debris can be readily located and analyzed in situ in the extracted aerogel fragment by SEM. Particles can then be isolated for in-depth study by techniques not amenable to in situ analysis in aerogel, such as transmission electron microscopy (TEM) and secondary ion mass spectrometry (SIMS).

EXTRACTION EQUIPMENT

We experimented with a range of blades from simple surgical scalpel blades to laser-cut diamond blades. All blades were mounted in a commercially available piezo-driven holder (Eppendorf MicroDissector) on a 3-axis micromanipulator (Eppendorf TransferMan NK2) that was controlled by hand under a Leica MZ16 stereomicroscope with a long working distance objective (Fig. 1a). The MicroDissector's primary applications are intracytoplasmic sperm injection and microdissection of tissue samples, but its piezo-driven holder, controlled by a foot pedal, produces oscillations also ideal for aerogel cutting. Motion is primarily driven along the long axis of the cutting blade over an available frequency range of approximately 25–60 kHz with maximum amplitude of 1.5 µm. This results in excitation of higher amplitude, transverse vibrations at the blade tip.

Readily available cutting tools such as scalpels and thin razor blades (~75 µm thick) have already been used to cleave aerogel to extract hundreds of terminal particles from impact tracks (Hörz et al. 2000). Even these less elegant cutting tools cut flight-grade aerogel cleanly with little to no fracturing when combined with piezo-driven U/S oscillations. They do, however, create wide channels in the aerogel and tend to generate tearing at depths beyond a few hundred microns.

To reach greater depths, ultra-thin microblades of high-carbon steel and diamond were developed. Two basic blade types, laser-cut from diamond to very narrow, sharp cutting edges, were designed in collaboration with Norsam Technologies (Hillsboro, Oregon, USA): a utility knife–shaped blade and a chisel-shaped blade (Figs. 1b–d), both 5 mm long, 25 or 55 µm thick at the start of the blade and with 10° or 20° cutting edges. High-carbon steel microblades with utility-knife shapes were produced from 100 µm thick, breakable razor blades (Electron Microscopy Sciences) by snapping off fragments at LN_2 temperatures to prevent plastic deformation at the tip. Breakable razor blade fragments were attached to rods with epoxy for mounting in the piezo-driven holder.

Both blade materials have advantages and disadvantages: high-carbon steel microblades can be made in the lab at low cost (albeit low uniformity of blade shape), while diamond microblades are expensive to purchase. Ultra-thin diamond blades create slightly thinner channels with less damage due to their narrow cutting angle and very smooth cutting edge that result in a lower risk of initiating tears in the aerogel. This enables cuts to be made nearer to the track of interest, and closely spaced impact tracks can be extracted separately. Diamond is optically transparent, allowing continuous observation of the cutting region with less distortion and blurring, and since identical diamond microblades are manufactured, the U/S frequency need not be changed significantly between blades. However, with sustained application of U/S oscillations, diamond microblades show a buildup of compressed silica on the cutting surface, leading to an effective dulling of the blade and increased aerogel tearing. The majority of this buildup can be removed by drawing the blade carefully through styrofoam. Any remaining film can presumably be removed in a dilute HF solution or HF vapor that will not attack the diamond. It is unclear why the high-carbon steel microblades have yet to show this problem. Since diamond is brittle, the fine cutting edges and tip also tend to chip, but with care, diamond blades retain their sharp cutting edges longer than steel blades. The microblade extractions presented here have been made with diamond blades for narrow cutting channels and the cleanest cut surfaces.

AEROGEL CUTTING

Silica aerogel is a highly porous, amorphous glass formed by sol-gel processing followed by supercritical drying. Despite its high strength-to-weight ratio and compressibility, aerogel exhibits glass-like mechanical behavior in tension including spalling and brittle fracture (see, for example, Woignier et al. 2000). These mechanical properties make it challenging to handle and cut without catastrophic failure.

The application of vibrations to blades to facilitate cutting is well-established in nature: leaf-cutting ants, anchored on their hind legs, vibrate their mandibles at kHz frequencies during leaf cutting (Tautz et al. 1995). Analogous vibrating blade technology (Woods et al. 1994) is used for microtoming tissue sections without freezing or embedding. The U/S oscillations applied to the microblades by the piezo-driven holder break up and compress the aerogel locally

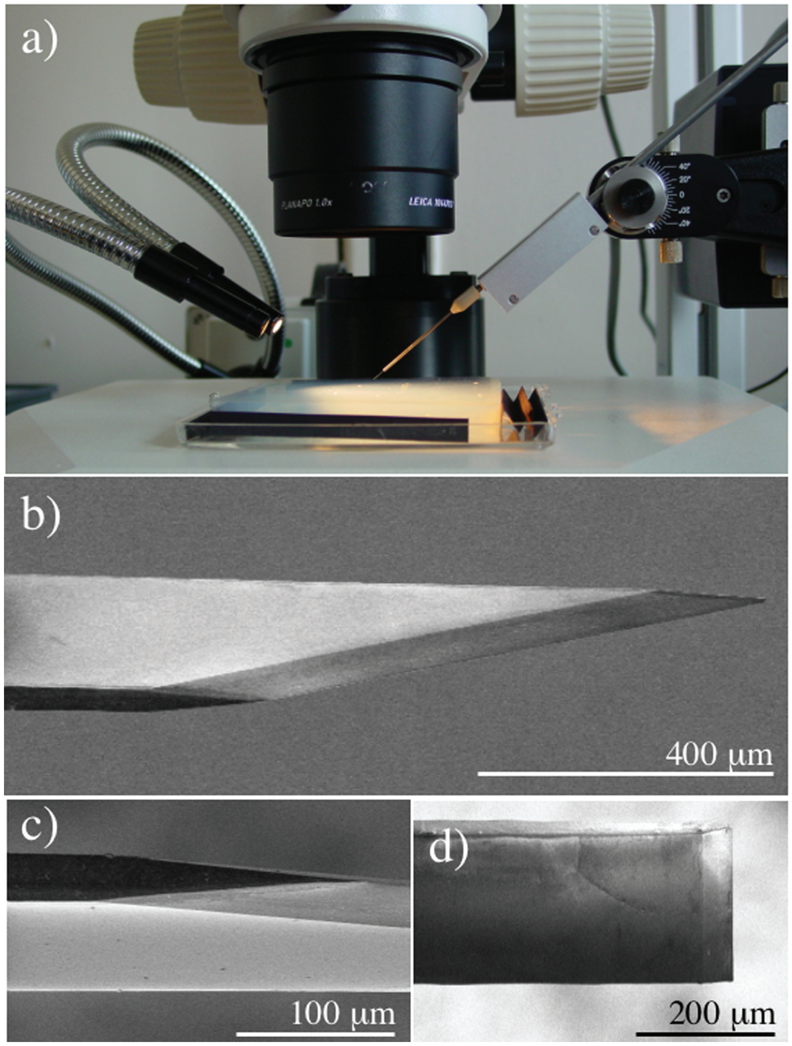

Fig. 1. Equipment for ultrasonic microblade extractions. a) Above an aerogel tile is a diamond microblade mounted in a piezo-driven holder (Eppendorf's MicroDissector) on a micromanipulator under a stereomicroscope with a long working distance objective. b) A secondary electron (SE) image of a diamond utility knife used for vertical cuts. c) An SE image showing the start of the blade's cutting edge. d) An SE image of a diamond chisel used for undercuts (with some artifacts due to charging).

creating a narrow channel that yields a lower friction interaction as the blade passes through. Figure 2a shows cuts made in a flight-grade (20 mg/cm^3) silica aerogel blank with and without the piezo-driven U/S oscillations. We have found that U/S frequencies between 30–45 kHz, amplitudes of ~1.5 µm (100%) and cutting speeds of ~150–200 µm/sec are optimal for clean aerogel cutting with both diamond and steel microblades. In our experience, lower density silica aerogel (10 mg/cc aerogel and Stardust flight spare) can be cut cleanly at higher speeds with less tendency to tear and spall. The U/S frequency is tuned for each blade via test cuts on a pristine aerogel blank. The degree of aerogel damage created by the cuts is highly sensitive to frequency, as illustrated in Fig. 2b. Previous attempts to apply this approach to aerogel have been less successful possibly due to non-optimum frequency or amplitude of U/S oscillations. Blade alignment with the micromanipulator axes of motion is readily achieved by placing a mirror below the blade and adjusting the blade rotation while observing the reflection under a microscope.

Two techniques for U/S piezo-assisted cutting of aerogel with microblades have been developed. The first technique involves slowly pushing a vibrating blade directly into the aerogel to the desired depth. By rotating the micromanipulator on its stand, the chisel-style diamond blade has been twice used in this manner to create an undercut in the aerogel below the impact track for the extracted track in Fig. 3c as illustrated by cuts 1 and 2 in Fig. 3a. The same technique was used for the undercut of the extracted track in Fig. 3d.

The second technique is for vertical cuts, The utility knife–style blades (diamond or steel) are drawn with slow motions parallel to the surface. After each cut, the blade is lifted above the aerogel surface to return to the starting position, and the next cutting pass is made ~50 µm deeper.

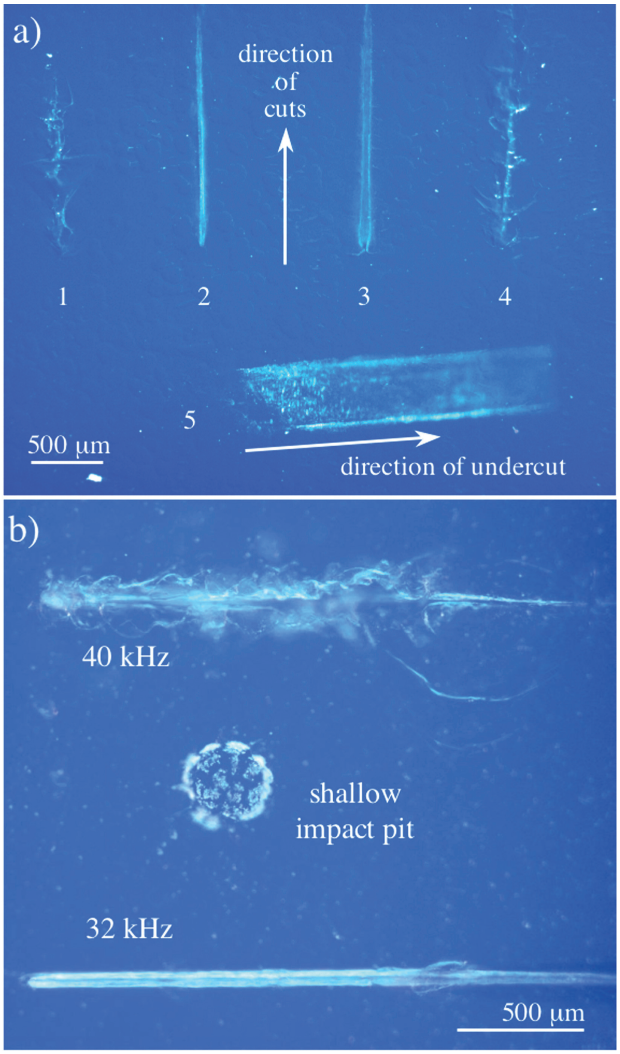

Fig. 2. A demonstration of the effectiveness of aerogel cutting with ultrasonic (U/S) microblades. a) An optical image of test cuts on flight grade aerogel (20 mg/cm^3). Cuts 1 and 4 (200 µm deep) were made without piezo-driven U/S oscillations, and cuts 2, 3, and 5 were made with U/S oscillations. Cuts 1 and 2 were made by a diamond utility knife, cuts 3 and 4 were made by the steel blade, and the undercut, cut 5, was made by a diamond chisel at an angle of 25° to the aerogel surface. b) The frequency sensitivity of microblade cutting. Cuts on either side of a shallow impact pit feature in ODC tile 2D04 were made with U/S frequencies of 40 kHz (upper cut) and 32 kHz (lower cut). Both were made using an ultra-thin diamond utility knife to a depth of 1.4 mm.

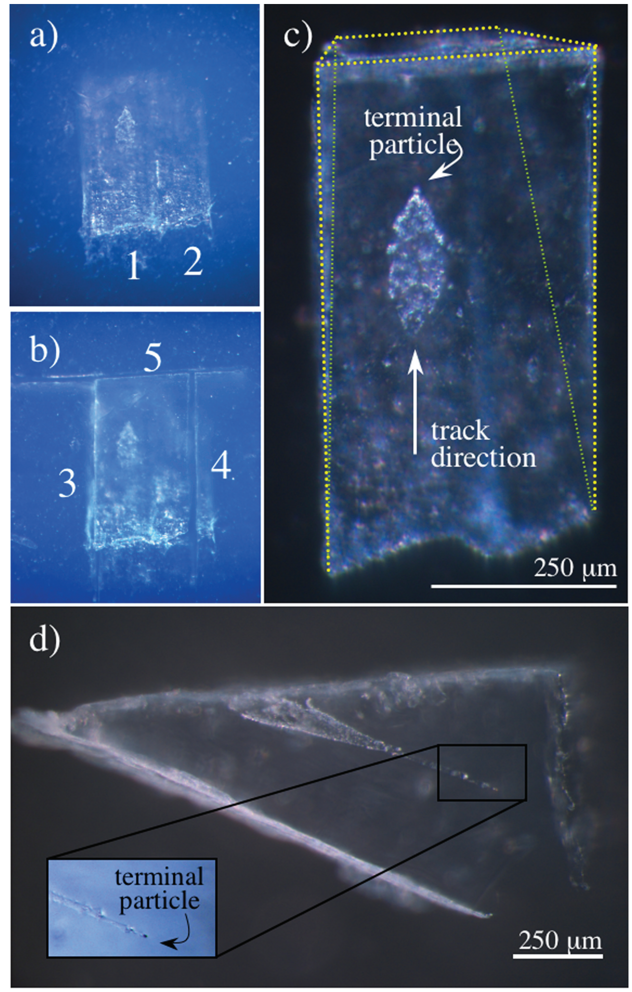

Fig. 3. Optical images of the extraction of ODC impact tracks from tile 2C01 using U/S piezo-driven oscillating diamond microblades. a) The first cuts (1 and 2) are undercuts made with a diamond chisel at an oblique angle to the aerogel surface and extending below the impact track. b) The final three cuts (3, 4, and 5) are vertical cuts made with a diamond utility knife perpendicular to the aerogel surface. c) The aerogel fragment containing the impact track after removal from the aerogel collector tile. The dashed yellow lines indicate the aerogel boundaries. d) A side view of another extracted aerogel wedge containing an impact track from ODC tile 2C01. The top surface shows some pre-existing damage. Inset: The terminal particle at the end of the stylus in transmitted light.

The aerogel is rotated for subsequent cuts. Cuts 3, 4, and 5 in Fig. 3b were made in this way. This second approach yields highly smooth cut surfaces.

It should be noted that damage (due to handling, for example) can create a thin surface crust of higher density aerogel that results in fragmentation of the surface rather than disintegration. This aerogel debris falls into the channel and is tumbled by the blade, creating ragged edges. For cuts sufficiently far from the track (~200–300 µm), the track itself is unharmed. This problem has not been found on aerogel tiles flown as part of the ODC experiment but having little handling damage.

U/S piezo-assisted dissection can be used to cleave the main cavity of large impact tracks in order to expose particulates and ablated material in the track walls. This results in the terminal particles residing closer to the surface simplifying their eventual isolation from the aerogel. These dissections can be carried out in the bulk aerogel collector tile. One half of the impact track can be extracted, leaving the remaining half in the tile (as was done with the extraction shown in Fig. 4b), or both halves can be extracted. Because the impact track itself is "damage" in the aerogel, there is

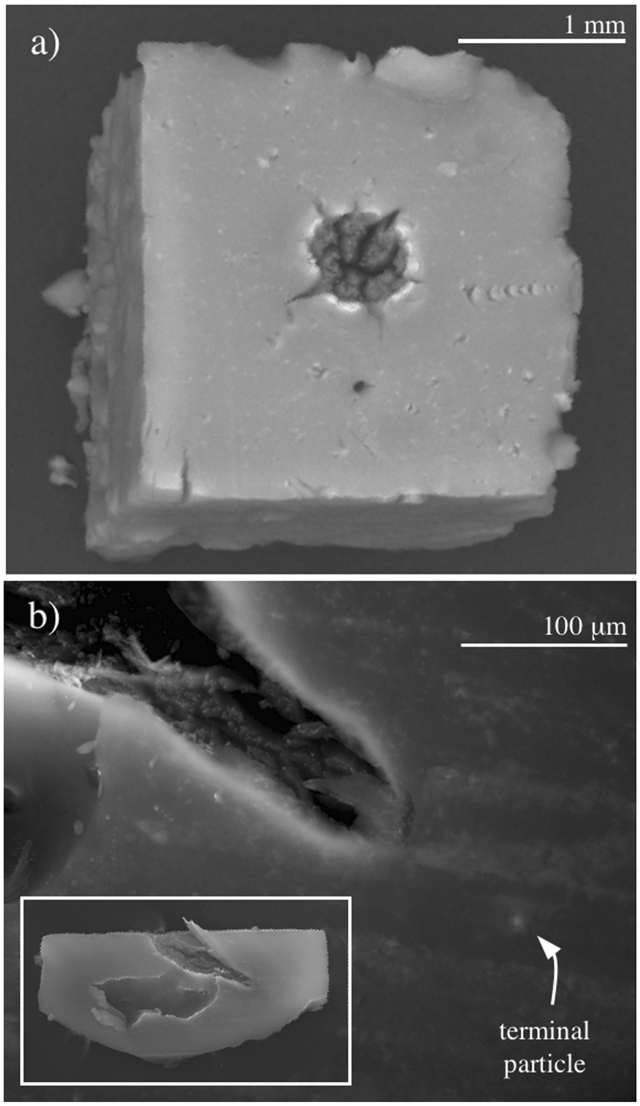

Fig. 4. Smooth cut surfaces produced by U/S microblades reduce imaging artifacts and allow quick identification of impact particles. a) Backscattered electron (BSE) image of the final extracted aerogel cube containing the shallow impact pit feature from Fig. 2b. The downward-facing surface was cut at the optimized U/S frequency for the diamond microblade. b) BSE image of an ODC track (terminal particle visible just sub-surface as a diffuse bright spot) from tile 1F04 dissected using a diamond utility knife with piezo-driven U/S oscillations. Inset: Secondary electron image of the entire dissected aerogel fragment. Tearing below the track propagated from the pre-existing damage at the bottom surface of the impact track.

some risk of further propagation of existing cracks, as seen in the inset of Fig. 4b. These cracks tend to propagate in the direction of blade motion at acute angles. This is especially an issue in large impact tracks; however, slow and controlled blade motion minimizes the extent of aerogel tearing. In addition, there is some compression of the aerogel at the edges of the cut as well as some saw-kerf (material loss due to breakup or sticking on the diamond blade). For small tracks (<50 µm diameter), there is a risk of eroding away much of the material of interest. In this case, tracks can be extracted whole from the aerogel tile with U/S piezo assistance and then cleaved out-of-tile using a sharp blade without U/S piezo oscillations, as in Fig. 5a. This results in a less predictable and less flat dissection surface, but the fracture surface is still very smooth.

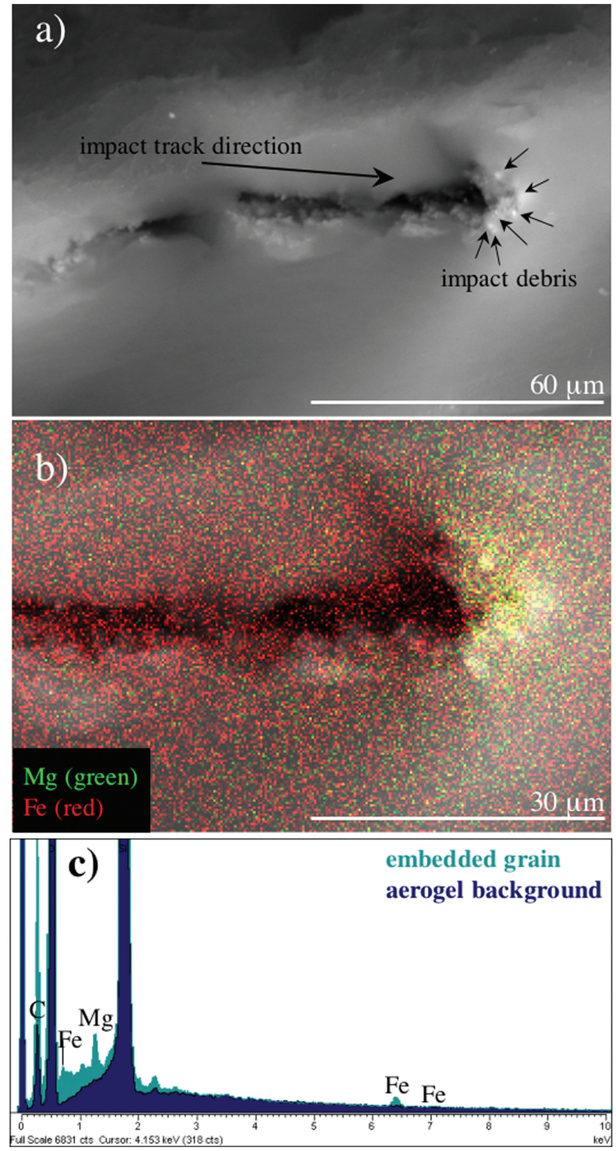

Fig. 5. a) A backscattered electron (BSE) image of an aerogel fragment extracted from ODC collector tile 2C01. This track was extracted whole from the tile using U/S microblades and then dissected out-of-tile by a diamond blade without U/S oscillations to expose a region of the track's main cavity. Several bright impact debris particles at the end of the exposed region are identified by small arrows. The intensity from the particles appears diffuse due to the fact that they are still beneath the surface of the aerogel. b) The high intensity locations (yellow-white) in X-ray energy-dispersive maps for Mg (green) and Fe (red) correlate strongly with the bright particles in the BSE image. c) A typical energy-dispersive spectrum for one of the individual particles is consistent with a silicate composition.

PRELIMINARY ANALYSIS OF EXTRACTED TRACKS

An end goal for U/S microblade development is incorporation of the blades in an SEM or dual-beam FIB environment to enable sample recovery/preparation and preliminary characterization. This goal is motivated by previous analysis of extraterrestrial material preserved in ODC impact tracks that shows a high degree of fragmentation of the original projectile with debris deposited down the length of the track (e.g., Borg et al. 2004). This underscores the need for a means of identifying and removing particles of various sizes.

For the SEM studies, aerogel fragments containing dissected impact tracks and one aerogel fragment containing a shallow impact pit, all extracted using the U/S microblade technique, were mounted on standard SEM mounts with adhesive, conductive carbon pads and examined in a LEO 1455VP SEM fitted with an Oxford INCA Pentafet energy dispersive X-ray spectrometer (EDS). A 15 mm working distance and a 20 kV accelerating voltage were used for both imaging and microanalysis. A 700 pA beam current was used for imaging and a 1.2 nA beam current was used for microanalysis.

Imaging

Typically, secondary electron imaging offers only topographic information while backscattered electron (BSE) imaging provides atomic number contrast. Thus, BSE imaging is an obvious candidate for locating higher density micrometeoroid fragments in dissected impact tracks in low-density silica aerogel. However, there are a number of issues in the track alone: There is major beam dispersion and electron energy loss with depth in the aerogel, and much of the debris is located subsurface. In addition, surface roughness and densified aerogel formed by compression and by melting and recondensation in the track add noise to the image. In the past, it has been difficult to distinguish micrometeoroid fragments from the surrounding aerogel by BSE imaging of tracks extracted using the glass needle extraction method. Compositional contrast has been largely obscured by image artifacts generated by additional high roughness and compacted aerogel debris produced by the glass needle extraction method on the cut surfaces surrounding the track. This roughness and additional debris add more artifacts to the already complex picture in the track cavity. In contrast, the U/S microblade extraction method generates very smooth surface cuts that reduce imaging artifacts in both secondary and backscattered electron imaging. The low-roughness surface removes uncertainties in identifying impact debris so that particles exposed on the surface, or even located subsurface, can be quickly located by BSE imaging. The ease of locating cosmic dust particles within smooth-cut extracted tracks will simplify in situ recovery of isolated particles in a SEM or dual-beam FIB (Graham et al. 2004; Graham et al. 2005). Figure 4a contains a BSE image of the shallow impact pit cut from a bulk ODC aerogel tile (Fig. 2b). The downward-facing surface shows the smooth-cut surface produced by U/S microblades at the optimized frequency. Figure 4b shows the BSE image of an ODC impact track dissected using a diamond utility knife with piezo-driven U/S oscillations. The terminal particle lies just below the aerogel surface and appears as a clearly distinguishable, bright spot in the BSE image. The bright spot has a diffuse appearance due to the subsurface location of the particle.

Microanalysis

All of the dust particles captured in aerogel, from either LEO collectors or the Stardust collector, have experienced hypervelocity impact. As a result, they are likely to have undergone some degree of alteration, most likely fragmentation and thermal modification, and therefore, they are not pristine in nature (Anderson and Ahrens 1994; Hörz et al. 2000). To ensure the data acquired is representative of the composition of a cometary grain prior to hypervelocity capture, a number of particulates associated with a single impact should be characterized. While other techniques, such as synchrotron X-ray fluorescence (e.g., Borg et al. 2004), have been shown capable of such studies, EDS mapping and single spot analysis are also well-suited to identifying candidates for isolation as "naked" particles for in-depth TEM or SIMS analysis. Figure 5a is a BSE image of an impact track associated with the chondritic swarm event previously identified in the ODC tiles (Hörz et al. 2000). This track was extracted whole from the bulk aerogel tile and then dissected to expose the main cavity by a diamond blade without applying U/S oscillations. The cleaved surface again has very low roughness, allowing imaging of impact debris that appears as a cluster of bright spots in the BSE image. EDS mapping in Fig. 5b shows elevated levels of Mg and Fe in the region containing the particulates, which is confirmed by single spot analyses (Fig. 5c). While it is not possible to unambiguously identify the mineralogy of the particulates due to the high background from the surrounding aerogel, it is highly probable that they are fragments of an Mg-Fe silicate. For rigorous mineralogical analysis, isolation of the particulates from the silica aerogel fragment is necessary.

SUMMARY AND FUTURE WORK

We have presented a simple, rapid, and accessible method for the extraction of hypervelocity impact tracks from silica aerogel tiles. We demonstrated that ultrasonic frequency oscillations applied to thin, sharp microblades quickly produce clean cuts in aerogel with minimal damage to the surrounding tile. Extraction time depends on the details of track depth and morphology; however, using our current setup under hand control, complete tracks have been extracted in less than an hour. In addition to the optical microscope and micromanipulator normally found in laboratories involved in small particle manipulation, a system for extracting complete or dissected impact tracks from bulk aerogel tiles requires only a piezo-driven holder and microblades. Although piezo-driven holders may be home-built, we have chosen a commercially available piezo-driven holder to generate ultrasonic oscillations of the appropriate frequency, amplitude and direction. For the ~25–100 µm thick diamond and steel microblades used in this work, frequencies between 30 and 40 kHz are optimum. With this equipment, impact tracks have been extracted from aerogel tiles flown on NASA's Orbital Debris Collector experiment. Tracks have also been dissected exposing the main cavity for SEM imaging and elemental analysis. The smoothness of the resulting cut surfaces allows quick identification of impact debris particles for EDS analysis and, if desired, eventual isolation of individual particles for other analyses.

The various methods now available for extracting impact tracks/pits and terminal particles/residues from silica aerogel offer advantages in different conditions. For large tracks or pits that are sufficiently widely spaced, cleaving the aerogel via handheld razor blades may provide the least expensive and quickest method of track and/or particle extraction. The ultrasonic microblades presented here are well-suited to small and intermediate-size features that may be more closely spaced. The computer-controlled glass needle extraction method is best applied to small and very closely spaced tracks since the technique, although slower and more expensive in setup cost, is not limited to straight cuts and causes very little damage outside the extracted region.

Our current micromanipulator has orthogonal motions, one perpendicular to the sample surface. The entire unit can be rotated to make undercuts in aerogel not perpendicular to the sample surface. We are installing a micromanipulator with an additional axis for angled blade motions. This will permit freeing an aerogel fragment completely from a tile without adjusting micromanipulator angle. For tracks that are nearly perpendicular to the surface, such as those expected in the Stardust aerogel, an inverted regular pyramid can be extracted with 3 or 4 angled cuts. For tracks at more oblique angles, one or more of those cuts may be perpendicular to the surface. To assist in such extractions, modified chisel-style diamond microblades have been designed and will be tested.

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