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RAPID EXTRACTION OF DUST IMPACT TRACKS FROM SILICA AEROGEL BY ULTRASONIC MICRO-BLADES

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Abstract

In January 2006, NASA's Stardust Mission will return with its valuable cargo of cometary dust particles, the first brought back to Earth, captured at hypervelocity speeds in silica aerogel collectors. 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 and future missions using aerogel is the ability to efficiently extract impacted particles from collector tiles. Researchers will be eager to obtain Stardust samples as quickly as possible, and 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 micro-blades. Applying ultrasonic frequency oscillations to these micro-blades via a piezo-driven holder produces rapid, clean cuts in the aerogel with minimal damage to the surrounding collector tile. With this approach, impact tracks in aerogel fragments with low-roughness cut surfaces have been extracted from aerogel tiles flown on NASA's Orbital Debris Collector Experiment. The smooth surfaces produced during cutting reduce imaging artifacts during analysis by SEM. Some tracks have been dissected to expose the main cavity for eventual isolation of individual impact debris particles and further analysis by techniques such as TEM and nanoSIMS.

INTRODUCTION

A means of efficient extraction of particles from silica aerogel collector tiles is necessary for the science return of Stardust. This NASA sample return mission, the first from a comet, returns in early 2006 with its valuable cargo of comet dust 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 requiring little construction cost or training would be an attractive asset. One method for extracting 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 and 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 for Stardust, we have experimented with micro-blades. 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 micro-blade extraction technique, still under development, we have extracted several impact tracks from aerogel tiles flown on NASA's Orbital Debris Collector (ODC) Experiment. Additional tracks were dissected into two halves exposing the main 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) 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 longitudinal oscillations also ideal for aerogel cutting. Motion is primarily along the long axis of the cutting blade over an available frequency range of approximately 25 to 60 kHz with maximum amplitude of 1.5 μm.

Readily available cutting tools such as scalpels and standard razor blades (~200 μm thick) have already been used to extract hundreds of 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 micro-blades 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, OR): a utility knife shaped blade and a chisel shaped blade (Fig. 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 micro-blades with utility-knife shapes were produced from 16 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 micro-blades can be made in the lab at low cost (albeit low uniformity of blade shape) while diamond micro-blades 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 give 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 continual observation of the cutting region with less distortion and blurring, and since identical diamond micro-blades are manufactured, the U/S frequency need not be changed significantly between blades. However, with sustained application of U/S oscillations, diamond micro-blades 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 micro-blades 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 micro-blade 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 micro-blades by the piezo-driven holder break up and compress the aerogel locally creating a narrow, wedge-shaped 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/s are optimal for clean aerogel cutting with both diamond and steel micro-blades. 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 Figure 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 micro-blades have been developed: 1) 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 used twice in this manner to create an undercut in the aerogel below the impact track for the extracted track in Figure 3c as illustrated by cuts 1 and 2 in Figure 3a. The same technique was used for the undercut of the extracted track in Figure 3d. 2) 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. The aerogel is rotated for subsequent cuts. Cuts 3, 4 and 5 in Figure 3b were made in this way. This second approach yields highly smooth cut surfaces.

It should be noted that damage (for example, due to handling) 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 are 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 pristine flown aerogel tiles.

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 Figure 4b, or both halves can be extracted. Because the impact track itself is "damage" in the aerogel, there is some risk of further propagation of existing cracks as seen in Figure 4b, inset. 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 Figure 5a. This results in a less predictable and less flat dissection surface, but the fracture surface is still very smooth.

PRELIMINARY ANALYSIS OF EXTRACTED TRACKS

An end goal for U/S micro-blade 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 micro-blade 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, for microanalysis.

Imaging

Typically, secondary electron imaging offers only topographic information while back-scattered electron (BSE) imaging provides atomic number contrast. In the past, a significant problem with BSE imaging has been that the silica aerogel and silicate-dominated micrometeoroids, despite their substantial differences in density, are difficult to distinguish from one another. This is not due to major differences in back-scatter coefficients although the depth of penetration in aerogel does result in major beam dispersion and electron energy loss. The compositional contrast is largely obscured instead by image artifacts generated by the very rough track surface and compacted aerogel debris that can be produced by the glass needle extraction method. In contrast, the U/S micro-blade extraction method generates very smooth surface cuts that reduce imaging artifacts in both secondary and back-scattered 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 (in Figure 2b). The downward-facing surface shows the smooth cut surface produced by U/S micro-blades 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 acquire data 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 Figure 5b shows elevated levels of Mg and Fe, confirmed by single spot analyses (Fig. 5c), in the region containing the particulates. 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 extraction of hypervelocity impact tracks from silica aerogel tiles. We demonstrated that ultrasonic frequency oscillations applied to thin, sharp micro-blades 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 labs 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 micro-blades. 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 ~15-60 μm thick diamond and steel micro-blades 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.

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 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 micro-blades have been designed and will be tested.

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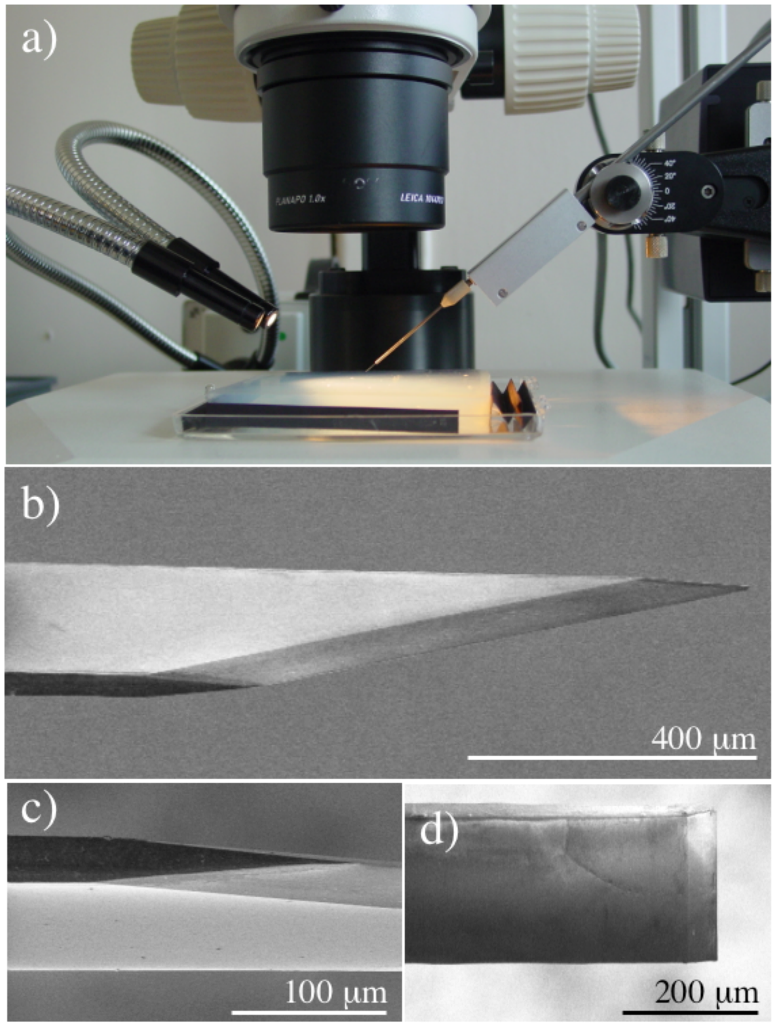

Fig. 1: Equipment for ultrasonic micro-blade extractions. a) Above an aerogel tile is a diamond micro-blade mounted in a piezo-driven holder (Eppendorf's MicroDissector) on a micromanipulator under a stereomicroscope with a long working distance objective. b) Secondary electron (SE) image of a diamond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

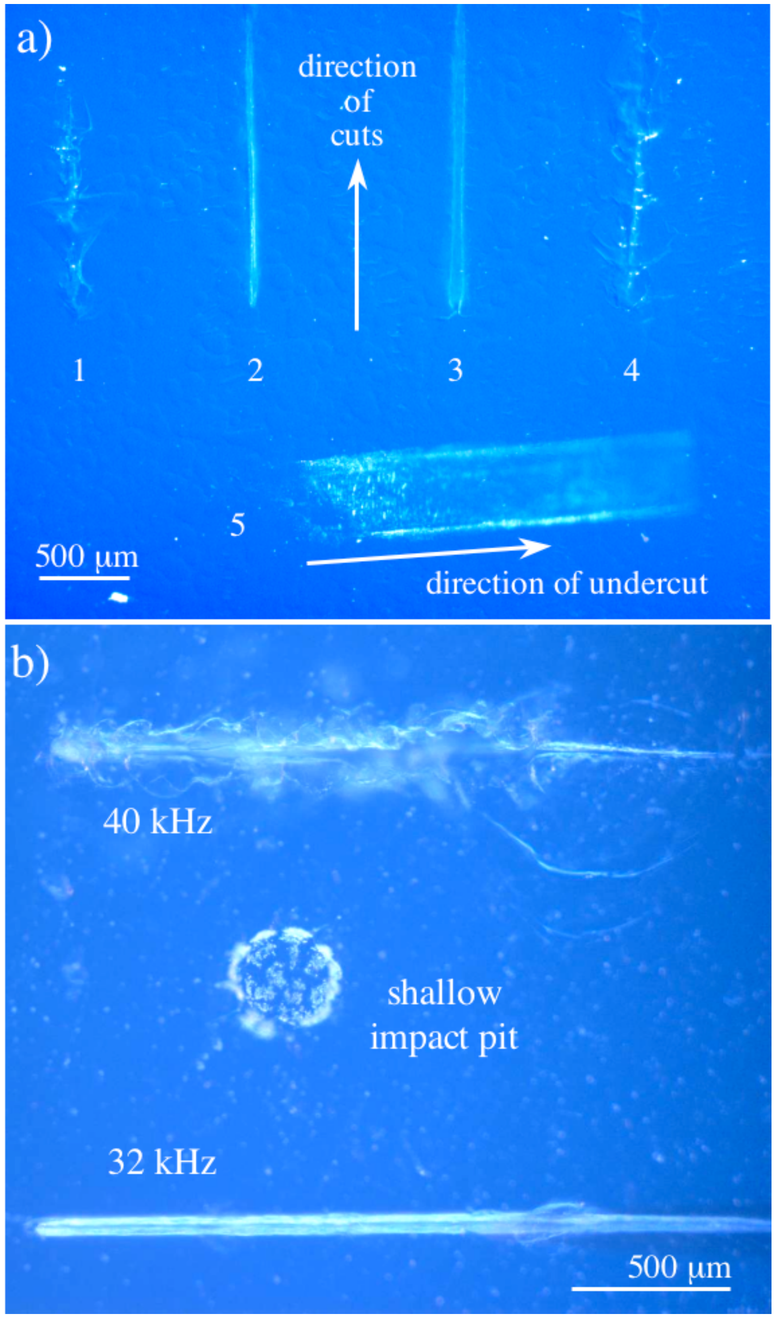

Fig. 2: Demonstration of the effectiveness of aerogel cutting with ultrasonic (U/S) micro-blades. a) Optical image of test cuts on flight grade aerogel (20 mg/cm^3): Cuts 1 and 4 (200 μm deep) were made without piezo-driven U/S oscillations, and cuts 2, 3 and 5 were made with U/S

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) Frequency sensitivity of micro-blade 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.

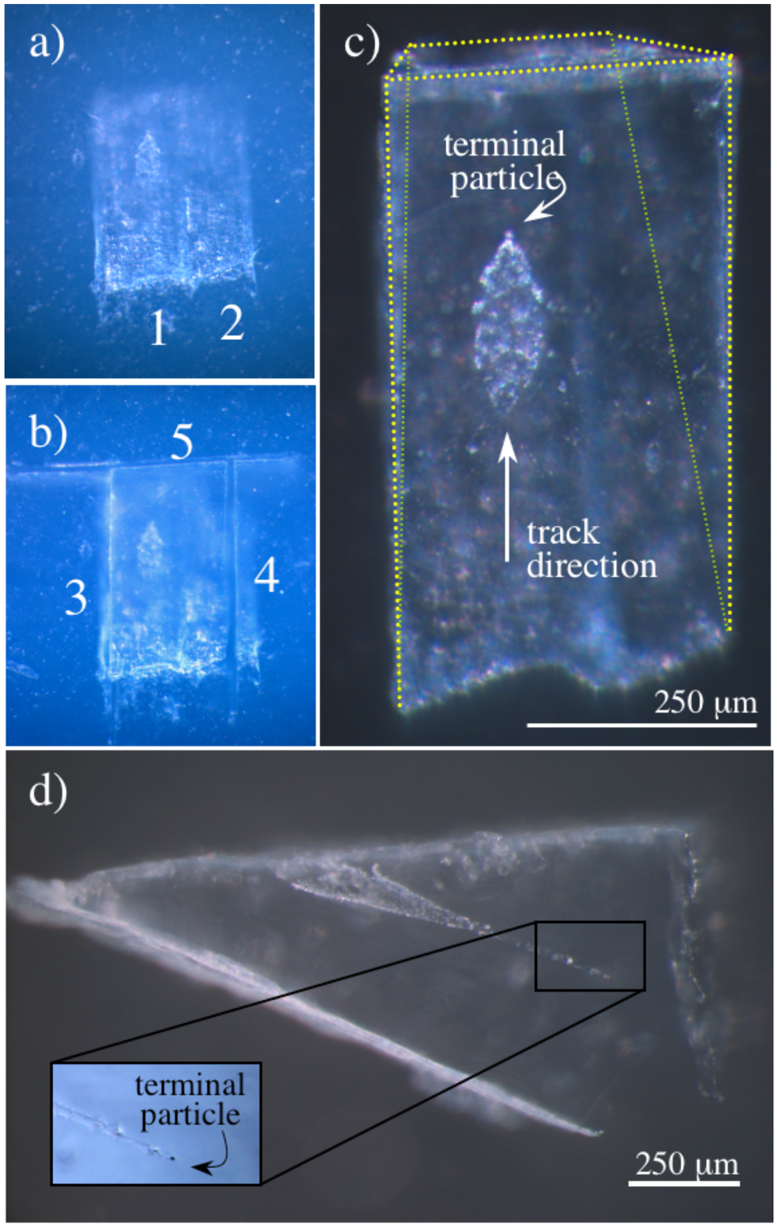

Fig. 3: Optical images of the extraction of ODC impact tracks from tile 2C01 using U/S piezo-driven oscillating diamond micro-blades: a) The first cuts (1 and 2) are undercuts made with a diamond chisel at an oblique angle to the aerogel surface and extending below the impact track. b) The final three 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. (Dashed yellow lines indicate the aerogel boundaries.) d) Side view of another extracted aerogel wedge containing an impact track from ODC tile 2C01. The top surface shows some pre-existing damage. Inset: Terminal particle at the end of the stylus in transmitted light.

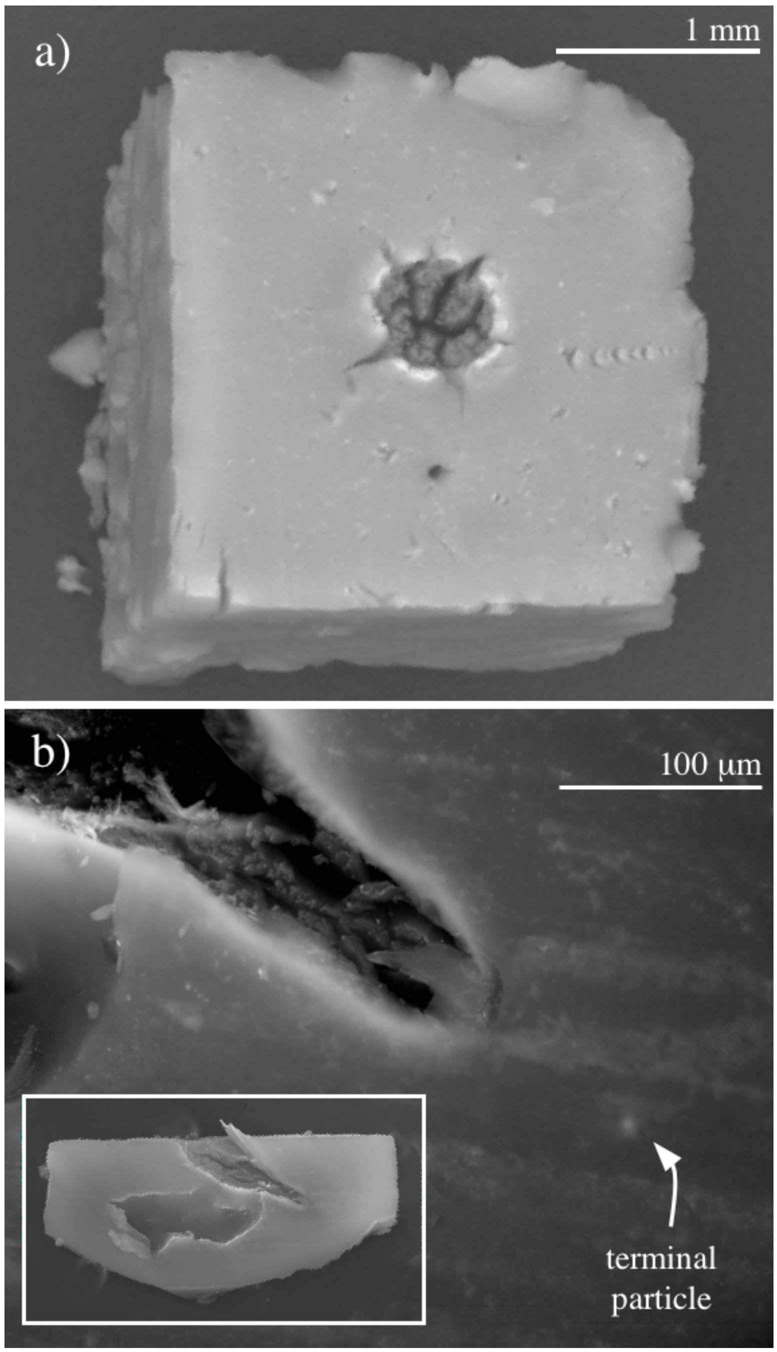

Fig. 4: Smooth cut surfaces produced by U/S micro-blades reduce imaging artifacts and allow quick identification of impact particles. a) Back-scattered electron (BSE) image of the final extracted aerogel cube containing the shallow impact pit feature from Fig. 2b. The downward-facing surface was cut at the optimized U/S frequency for the diamond micro-blade. 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.

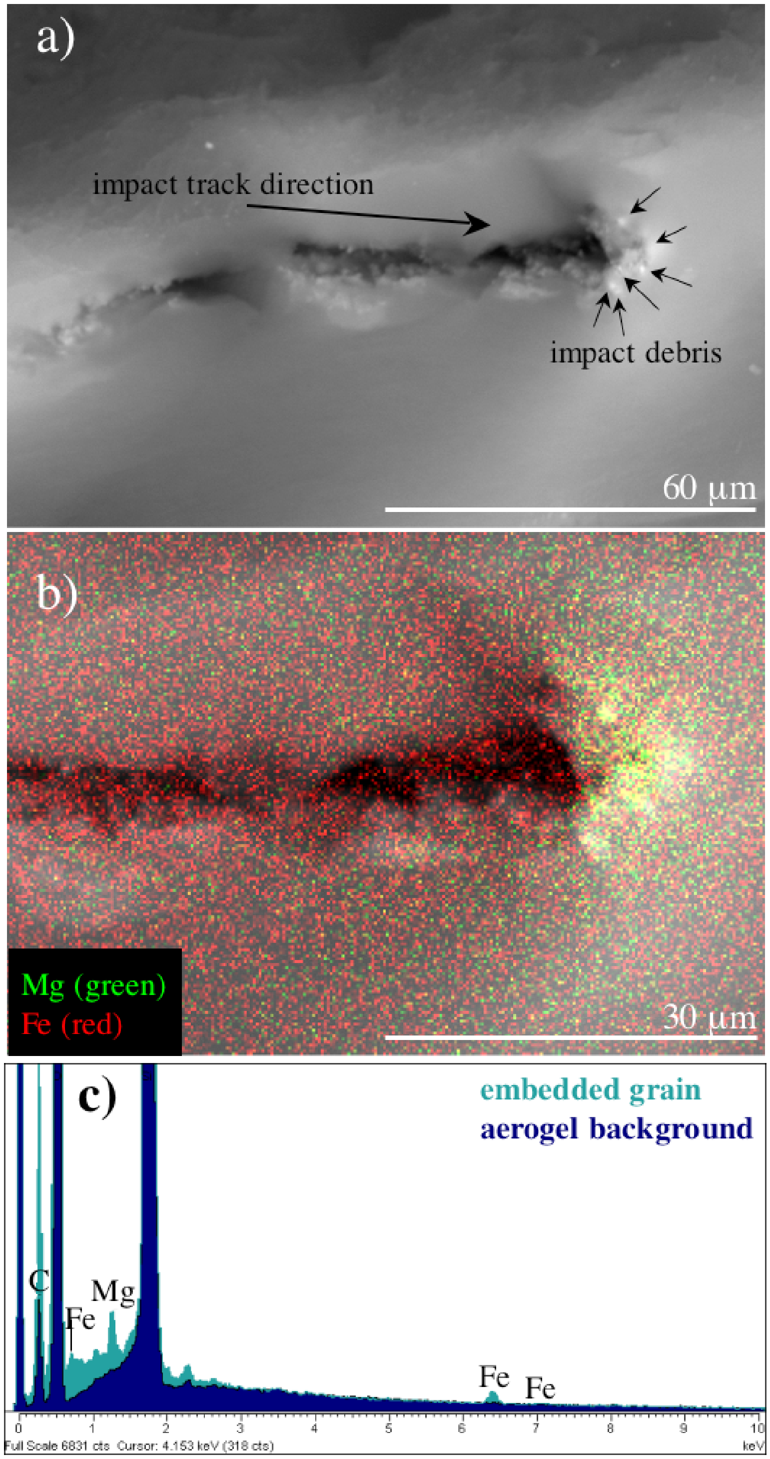

Fig. 5: a) A back-scattered electron (BSE) image of an aerogel fragment extracted from ODC collector tile 2C01. This track was extracted whole from the tile using U/S micro-blades and

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.