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Curation, Spacecraft Recovery and Preliminary Examination for the Stardust Mission: A Perspective From the Curatorial Facility

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Abstract

We briefly describe some of the challenges to the Stardust mission, curation and sample preliminary analysis, from the perspective of the Curation Office at the Johnson Space Center. Our goal is to inform persons planning future sample returns, so that they may learn from both our successes and challenges (and avoid some of our mistakes). The Curation office played a role in the mission from its inception, most critically assisting in the design and implementation of the spacecraft contamination control plan, and in planning and documenting the recovery of the spacecraft reentry capsule in Utah. A unique class 100 cleanroom was built to maintain the returned comet and interstellar samples in clean comfort, and to permit dissection and allocation of samples for analysis.

Introduction

With the exception of a few dust-collecting satellites like the Long-Duration Exposure Facility (See et al., 1990; Zolensky and Kinard, 1993), the Stardust mission was the first sample return mission mounted by NASA in almost thirty years, during which time practically everyone involved in the planning of the previous (Apollo) missions had passed on to a more relaxed lifestyle. Thus the mission was briefly hampered by inexperienced scientists and engineers who all had to relearn the skills involved in sample return. We faced many challenges in designing procedures for sample collection, curation, handling and preliminary sample examination. Many papers have touched on the initial results of the Stardust Mission, but in this paper we briefly describe some of the challenges to the mission, curation and data return itself. Our goal is to inform persons planning future sample returns, so that they may learn from both our successes and challenges, and avoid some of our mistakes, or perhaps repeat our mistakes in more original ways.

Mission Planning and Implementation

Communication Between Scientists and Engineers

The science team had to reinvent many processes, all the while skirmishing with mission engineers whose brains were wired very differently from those of scientists. For many of us this was our first involvement in a major spacecraft mission from the ground up, and there was a large learning curve to travel. It took quite some time for these mission scientists to learn how to communicate with the mission engineers, and during this learning period we made some misjudgements, which may have promoted the early retirement of at least one of the spacecraft designers (in the opinion of the first author). At early planning meetings scientists would sometimes briefly promote new and "trivially simple" spacecraft capabilities, that we would learn at the next meeting that spacecraft designers had taken as new hard requirements, with appropriate changes to the design of the spacecraft and debilitating effects on the mission schedule or budget. As the mission design progressed the engineers began to refer to these "minor" changes as "mission creep" or even more pejoratively as "science creep". At other times the spacecraft designers would appear to be promising spacecraft capabilities that later proved illusory; this was always the result of miscommunication, never ill-intentions or misdirection. The absolutely critical lesson here is for mission scientists to quickly learn how to speak clearly to engineers (and to understand when they speak back) which will be a difficult, though ultimately rewarding skill to master. Perhaps planetary scientists should be able to take engineering courses in graduate school in lieu of the foreign language requirement.

Spacecraft Contamination Control

The first author of this paper was given the task of co-writing the Contamination Control Plan for the mission (Zolensky and Girard, 1997). The mission designers faced a similar (though considerably lesser) learning challenge. In previous missions, where no sample was being returned to Earth, witness surfaces exposed during spacecraft hardware manufacture, cleaning, integration and testing were examined and then discarded. For a sample *return* mission it was crucial to preserve all of these surfaces, and indeed to expose many more than was the norm. The crucial individual here was the contamination control lead for the spacecraft construction contractor. We were fortunate in that ours (Tim Girard, Lockheed Martin Aeronautics) was very skilled and diligent, though, like many other talented individuals on the project, working several missions simultaneously. Accordingly, it was essential for the science team to take a very active role in the design and implementation of contamination control requirements.

One big problem we faced, and never adequately solved, was in making the aerogel (Figure 1) (the heart of the mission) as clean and contamination free as possible. While a bake-out of the aerogel successfully removed a considerable amount of the organic contaminants, much remained. In addition, there are considerable inorganic contaminants in the aerogel, which we still do not completely understand. We should have focused more effort on making the aerogel as clean as possible, and then understanding the nature and levels of contamination better. We can and will recover from these problems because we carefully archived all preflight contamination witness surfaces, spare pieces of all pertinent spacecraft hardware, and many flight-quality unflown aerogel samples. However, a better understanding of the level and nature of indigenous contamination in the capture media would have simplified preliminary sample examination. For example early reports of small carbonate grains in the aerogel were fraught by ambiguity – could these be contaminants in the aerogel? It was critical to settle this matter, for many reasons. This issue is discussed below.

One major blunder we made was to fly too few flight witness surfaces. Flight witness surfaces are the primary way to learn what goop is being provided to the capture media from the spacecraft itself, principally organics outgassing from spacecraft lubricants. Of course we took the precaution of only opening the collection mechanism after several months of flight, to minimize contamination from spacecraft outgassing, but even the smallest amount of such contamination has to be properly accounted for. However, we flew a single piece of cometary aerogel, and one disk each of polished aluminum and sapphire as witnesses to the contamination during flight from spacecraft outgassing and activities. This single point failure mode came back to bite us after Earth return when a lab worker accidentally pushed a screwdriver into the sole aerogel flight witness sample in the course of removing it from the sample return capsule. Fortunately it was a very clean screwdriver, and damage was limited to one quarter of the cell. Nevertheless this incident underscores

the possibilities for single point failures when only one of anything is used for such a critical purpose. Admittedly, we never believed that contamination of the aerogel induced during manufacture would significantly compromise the data return. In particular return of cometary organics was never a major mission goal, because of the expected heating of particles during capture in aerogel at 6.2 km/sec. However, we have subsequently learned that the mineralogy of the comet coma particles is *far* more varied than anyone had guessed prior to the mission (Zolensky et al., 2006; Leroux et al, submitted a, b; Mikouchi et al., 2007; Wirick et al. 2007; Chi et al., 2007; Thomas et al., 2007; see also other papers in this volume), and so the boundary between indigenous cometary inorganic compounds and inorganic contamination in the aerogel is in some critical respects uncertain (and therefore the subject of considerable current research). For example, we see rare Ca-carbonates in at least three particle tracks so far (Wirick, et al., 2007; Leroux et al., submitted b; Mikouchi et al., 2007). Is this carbonate of cometary origin, or merely a trace manufacture contaminant, or possibly have been formed by heating, during capture of a cometary particle, of bystander contaminant grains in the aerogel? There appear to be truly cometary Fe-Mg carbonates in captured particles (Mikouchi et al., 2007), but which, if any, of the Ca-carbonates are cometary? When we better understand the contamination situation of the aerogel we will be able to solve this and similar problems.

Stardust fortuitously returned relatively unaltered organics in at least some tracks (Sandford et al., 2006; Cody et al., 2007), and so learning the nature and extent of organic contamination has taken on even greater importance than we had anticipated. Some of the organic materials are so unlike the known organic contaminants in the aerogel that their recognition is straightforward. But in other cases we simply do not yet know. The lesson here is to prepare for the mission to be more successful than you planned, and for nature to be more complicated than you imagined.

Stardust Curation Facility Planning and Construction

In the years prior to sample recovery, the Stardust Curation team at NASA's Johnson Space Center (JSC) constructed a receiving and curation laboratory. Before sample receipt, the steps required to build these laboratories included (1) developing design requirements based on needs from the scientific community, (2) designing the laboratories, (3) overseeing construction and equipment installation, (4) cleaning and certifying, (5) passing an Operational Readiness Review (ORR), and (6) learning how to use the lab and its special facilities. We benefited from the recent experience of constructing a curation facility for the Genesis Mission; although Stardust actually launched before Genesis, that latter mission was slated to return earlier.

The Stardust Science Team required a Class 100 (ISO Class 5) cleanroom for preliminary examination and long-term curation of the returned samples as well as two Class

10,000 (ISO Class 7) modular cleanrooms for initial sample receipt (one at the recovery site in Utah and one at JSC). In 2003 (three years before sample return), an architecture and engineering firm designed the curation laboratory utilizing three existing rooms within Building 31 at JSC. In 2003 (three years before sample return), an architecture and engineering firm designed this laboratory utilizing three existing rooms within Building 31 at JSC. The design included retrofitting an existing air handling system by re-routing ductwork and adding filtration and demolishing the existing three rooms to create one large room that would be acceptable to receive the new Class 100 modular cleanroom. The final design was completed and approved by the Stardust Science Team later that year. After the successful rendezvous with Comet Wild 2 in January, 2004, JSC awarded the contract for laboratory construction (this timing was not deliberate, believe it or not). The cleanroom was certified and accepted in the summer of 2004, and outfitting of the lab with required equipment to support preliminary examination began. By January 2005 (one year prior to sample return), the lab was complete. This allowed the JSC Curation team one year to train personnel, create and practice preliminary examination techniques, and write and refine their many procedures.

The database for the returned samples was very difficult to design and implement. The principal problem was to both (1) adequately name and track aerogel cells which were dissected to yield tracks, which were in turn dissected to yield grains, which were themselves dissected into many forms of slices, fragments, etc., and (2) use a sample numbering system which scientists would actually use. We were not totally successful in this measure, resulting in early confusion regarding some samples, and even today we are not happy with the result. Adding to the problems was the fact that our single database manager resigned (to take a better and less stressful position) in the midst of sample PET. We urge future missions to spend considerable time thinking through *all* of the possible sub-sampling and analytical activities before settling on a database design.

The Stardust spacecraft was initially received in a Class 10,000 (ISO Class 7) modular cleanroom located in a facility close to the Utah Test and Training Range (UTTR) landing site (Figure 2). The science canister was removed and secured in a clean transport container in this facility. After transport to JSC, the science canister entered another Class 10,000 cleanroom that served as the initial receiving facility in Houston. Following another cleaning step, the final stop was the Class 100 Stardust Curation Facility. Requirements for each of these facilities were derived from the science community and Mission team. The Curation Facility required the most careful planning because it would serve to preserve and protect these samples for generations to come.

The Stardust Curation team is composed of scientists and engineers who are a part of NASA's Astromaterials Curation Office at JSC. This group is familiar with extraterrestrial samples and appreciates the levels of cleanliness and security required to preserve and protect

such a precious resource. However, each astromaterial sample collection presents specific and unique challenges with respect to their curation. Unlike lunar rocks, for example, the Stardust samples are quite small. Thousands of cometary dust grains less than 100 μm in size were embedded in the collector media, silica aerogel cells, after the comet fly-by (Figure 1). Fortunately, we have 25 years of experience collecting, curating and handling interplanetary dust particles collected in Earth's stratosphere (Warren and Zolensky, 1994).

The silica aerogel collector media is another reason why the lab design requirements are so unique. Silica aerogel is extremely porous and acts like a very light sponge. It works extremely well to capture fast moving particles in space with minimal damage upon impact; however, because of its sponge-like quality, when exposed to liquid water, it becomes a much heavier solid and is opaque. When immersed in water, silica aerogel absorbs its volume in water, taking on the physical and optical properties of medium-hardness tofu. Given that the samples are imbedded in this medium, wet aerogel would make finding these samples a great challenge. Great care was taken in lab design to minimize the potential for water to encounter the samples and to keep humidity levels well controlled. Although required by JSC Fire Protection, a waiver was granted for the Stardust Curation lab to eliminate wet-pipe sprinklers inside the modular cleanroom. Instead, sprinklers were placed in the outer room (above the modular cleanroom) and a sensitive air sampling fire detection system was installed within the inner cleanroom. As a result we severely limit the presence of flammable materials within the class 100 cleanroom, and this requirement requires considerable constraint on the part of the curation team.

Like most cleanrooms, the Stardust Curation Facility operates at positive pressure, 20°C and 45% relative humidity. The basic design is a room within a room (Figures 3-4). The outer room is a Class 10,000 area served by one air handler with six HEPA fan filter units. The Class 100 modular cleanroom pulls filtered, conditioned air from the outer space with 36 fan filter units mounted on the ceiling. The vertical air flow drives a requirement that nothing be placed above (i.e. upwind) the Stardust aerogel tray. Thus all lab actions involving the tray have to be thought through carefully. Each of the fans can be controlled remotely from the control panel conveniently located within the anteroom. Although designed to be a class 100 facility, in practice the cleanliness is far higher owing to limitations on what can be brought into the lab, what can be done there, and the maximum number of people who can enter at any one time. A laboratory contamination control "officer" controls materials access.

Photo-documentation of the aerogel cells and the dust grains is an ongoing part of Curation. This documentation requires various levels of lighting. For this reason, dimmable lighting exists throughout the facility. These lights can be controlled by the control panel in the change room. Complete darkness is also achievable, which is sometimes helpful during certain types of photography. We do not permit separate camera

lighting instruments to enter the lab (for example the bright lights preferred by film crews), because of their contamination potential. We learned this lesson early on when one such light almost triggered the fire abatement system. Fortunately, this incident occurred before the Wild 2 samples were brought to the lab.

Small samples mandate a unique working environment. One example is static charge that can cause the samples to jump around. An anti-static cleanroom floor helps mitigate this concern, but we recommend that one not install a dark-colored floor (as we did) which shows scuff marks and scratches!. Another facility design consideration when manipulating small samples is minimizing vibration, so vibration dampening pads in the cleanroom framing support system were installed and grain extraction systems were located on vibration isolation tables. We have a cascade antistatic generator over the aerogel scanning platform, but are not convinced it makes a significant difference in sample handling. However, we utilize small, handheld radioactive ^{210}Po sources as local anti-static devices. These work so well we obtained more for the Cosmic Dust Lab, and wonder how we got along for so long without them. These small devices must be rented every year, because of Nuclear Regulatory Commission restrictions, a minor hassle.

Future expansion and flexibility were other things considered during Stardust cleanroom design and construction. A removable section was included in one of the modular cleanroom walls so that large objects could be moved in and out of the lab with ease. This particular wall also contains a large viewing window, which is especially nice for giving tours without requiring visitors to suit up and enter the lab. Make such a window low enough that children and Stephen Hawking can view activities inside the lab. Spare penetrations for future utilities were also specified in the cleanroom design so that additional construction would not be necessary to add future utilities. These wall penetrations came in very handy when JPL and JSC required us to install separate, dedicated video monitoring equipment during initial sample examination.

For ease in future maintenance, not only can the fan speeds be controlled remotely, but the units themselves can be serviced from within the cleanroom ("room side replaceable" units). Given that the modular cleanroom contains 36 fan filter units, multiple circuits were required to control them. This helps with future upkeep of the lab because each circuit only controls a few fans. The lab manager may elect to leave alternating circuits off in order to preserve the life of the filter and fans since all 36 fans do not need to run continuously to maintain the Class 100 environment. It is sometimes necessary to reduce airflow in the lab when certain samples are being manipulated, and this is easily done by turning off banks of fans. As mentioned previously, this cleanroom was designed as a room inside a room. This helps with future maintenance because if the outer room filters are well maintained, then the 36 filters on top of the modular cleanroom will last much longer. Another design requirement was rounded corners where possible. As a general rule, ways to avoid

unnecessary dirt to help with future cleaning were included upfront in the Stardust Lab design.

As with all cleanrooms built for curating NASA's extraterrestrial samples, Stardust cleanroom materials were specified by LASCO, the cleanroom vendor, in advance and samples provided as required. Specifying the materials to be used by the construction contractor and cleanroom vendor was critical. Samples were obtained in advance, which allowed the JSC team to analyze the samples and accept them as being clean and compatible with the future Stardust samples. This prevented any surprises during installation and testing. Particulate and non-volatile residue witness plates were used to monitor the environment in the lab during times of initial processing as well.

Perhaps the greatest lesson learned during cleanroom readiness was the need to be ready early. Having a full year for practice and training was absolutely critical for preparing to receive this precious sample set, and in the end we could have used even more training time.

Curation Planning for Spacecraft Recovery

Given the location and timing of the Stardust spacecraft return (a cold, potentially wet climate in the middle of the night), careful planning for the ground-based recovery operations began several years before return. Approximately 15 months prior to return, the recovery team began a series of field training exercises and simulations for the nominal recovery scenario and several pre-determined contingency scenarios. More than 12 detailed recovery practices were run in the four months prior to recovery – this schedule proved to be too compressed.

A modular Class 10,000 (ISO Class 7) cleanroom was installed in the receiving facility at UTTR not far from the actual field recovery site. Supplies for documenting, encapsulating and transporting the spacecraft from UTTR to the Curation facility at JSC were purchased and shipped to UTTR months before recovery. The UTTR cleanroom and supplies were necessary well before sample return because they were an integral part of the field training exercises.

When the mission was first planned, the recovery site, UTTR, had been a very dry locale for many years. However, beginning approximately 3 years before Stardust recovery the central Utah region began to see increased rainfall, resulting in large areas of very sticky mud and, in limited areas, standing water in the recovery area. Recognizing this situation, we ran a series of recovery exercises in mud, and obtained special equipment for operations in mud. The landing ellipse was moved slightly northward, to higher and potentially dryer ground. The importance of thinking through multiple contingency scenarios, practicing field recovery for these potential circumstances, and having the contingency supplies on-hand was critical despite the fact that they were unnecessary given the fortunate, nominal landing

of the Stardust spacecraft. This extra preparedness aided in team morale and confidence which ultimately allowed for smooth operations between recovery at UTTR and receiving at JSC. Still, the evening of the sample return capsule (SRC) recovery was very cloudy, and the clouds broke only just prior to SRC reentry. Immediately following recovery of the SRC a blizzard hit the landing area. It is clear that luck played a large role in the ultimately simple recovery operations.

After securing the sample container in a clean container at the UTTR facility, the samples traveled by a chartered C130 aircraft to sunny, warm Houston, reminding us of the flights out of the Antarctic. Logistics associated with receiving these samples required careful planning and coordination with JSC Receiving, Security, Safety, Quality Assurance, Photography and Curation. The samples received a police escort from Ellington Airport to the Curation facility at JSC. We timed the arrival to avoid Houston rush hour traffic.

After a successful receipt at JSC, the Stardust sample canister was handed off to the Science Team for preliminary examination (PE) in the Class 100 Curation Facility. It was difficult to plan for the initial excitement surrounding arrival of these samples. With this excitement brings a desire to work quickly. Good management skills by the Curator, and adequate lab staffing, and numerous dry run practice sessions were critical to keeping the team organized and samples well documented. Flexibility and patience among the PE team were key to staying in control during such a thrilling time. As it was we labored long into the night to open the SRC, remove the aerogel trays from its enclosing canister, and secure them in specially-built holders. We did not get to the celebration at the bar until after 10 pm.

During Stardust PE, a small, representative collection of the samples were selected to be stored at the remote curation facility for JSC located within the White Sands Reservation in New Mexico. Remote storage of such valuable samples is an important detail, and has been the norm for lunar and the most valuable meteorite samples for many years. At this facility selected samples are stored in sealed steel cans, within nitrogen-flooded steel cabinets.

Wild 2 Grain Extraction and Sample Preparation

Cometary feature extraction from the aerogel collector

After extensive photo documentation, the cometary material can finally be extracted for analysis. Because the samples are both microscopic and fragile, and distributed along impact tracks ranging from tens of micrometers to millimeters in size, it has proven to be a significant challenge to reliably and safely remove these samples without incurring significant damage to surrounding aerogel. Three extraction systems have been developed and installed in the Stardust processing cleanroom at NASA Johnson Space Center; one is use of razor blades – a tried and true approach and the technique suited to removal of unusual

aerogel samples. The second approach uses the "keystone" system (Westphal et al. 2004) (Figure 5), and the third is an ultrasonic vibration microblade, the so-called the "quikstone" system (Ishii et al. 2005, 2006) (Figure 6). The latter two systems reliably produce precision extraction and subdivision of aerogel-embedded samples and help to maximize the science return from these extremely valuable materials (Ishii et al. 2006), as described below.

Keystone System

This robotically controlled system is designed to extract a small volume of aerogel ("keystone") that contains an entire cometary particle impact track ranging from tens of microns to millimeters in length (Westphal et al. 2004). The cutting action consists of repeated small axial poking motions of the aerogel by two glass microneedles that are mounted on Sutter MP285 3-axis micromanipulators. The microneedles are oriented relative to the target track with the use of a high-power compound microscope (Figure 5a) that is equipped with a video camera for continuous monitoring of the extraction. The entire system is mounted on a vibration isolation table covered with a mirror that is useful for locating small pieces of loose aerogel. The sequence of poking creates a wedge of aerogel containing a target track that can be removed from a collector cell with negligible damage to nearby material. The entire process is controlled by custom written software (Westphal et al. 2004). The approximate time required to extract one keystone varies from 8 hours to 36 hours depending upon the size and depth of the tracks included in the keystone and other adjustable parameters of the moves such as poking speed and spacing between pokes. The optimal parameters vary from tile to tile because the mechanical properties of the aerogel vary between different batches. Although originating from the same manufactured batches, the flight spare aerogel tiles and actual flight aerogel tiles behave somewhat differently since the latter has experienced seven years in the vacuum of space. We make empty keystones (containing no cometary material feature) on each aerogel tile to learn and optimize the parameters so that we can generate keystones with smooth surfaces and minimize the damage to the surrounding aerogel (Figure 5b). The generated keystones can be mounted on custom-designed silicon fixtures, so-called microforklifts (Figure 5c).

As of this writing, more than 90 cometary features in keystones mounted on microforklifts have been allocated to stardust investigators all over the world. The analytical methods applied to the keystones are mainly synchrotron-based and other X-ray utilized techniques. The largest keystone generated so far is an 8.5 mm long wedge containing two cometary tracks (Figure 5d). We also successfully dissected a 10mm x 2mm x 1cm elliptical cylinder shape aerogel from the aerogel witness coupon using the keystone system with a thicker glass microneedle.

Quikstone system

An alternative method to rapidly extract and subdivide aerogel is the 'quikstone' system developed by Ishii et al. (2005, 2006). This system works by applying ultrasonic frequency oscillations to microblades (either diamond or steel blade) via a piezo-driven holder mounted on a micromanipulator. The oscillation frequency and cutting speed are carefully controlled to rapidly produce clean cuts in the areogel, making it possible to extract cometary dust impact tracks with minimal damage to the surrounding tile. In the JSC Stardust clean lab, the quikstone system is attached to a 3-axis micromanipulator (Sutter MP285), and to a long working distance stereomicroscope, Nikon SMZ1500. As with the keystone system described above, the quikstone extraction system is mounted on a vibration isolation table covered with a mirror that aids in locating loose aerogel fragments. The stage of the stereomicrosope is also covered with small mirror for the same reason, and a central hole in the mirror reflective coating permits transmitted light illumination of the operation.

The quikstone system generates larger-scale cuts in the aerogel tile compared to the keystone system, reaching several cm in length. We have been applying the quikstone system to split aerogel tiles, to sliver a mm-think layers from aerogel tiles, to extract rather large tracks (more than 1cm long) and conduct micro-surgery on aerogel pieces. For example, Figure 6a shows a dissected cometary track from a ~15x10x5mm aerogel chip found on the surface of the canister upon opening (this chip has not been identified with its parent aerogel tile yet). The aerogel chip was fixed onto a clean glass slide using teflon tape. A 5x5x1.5mm square of aerogel including the cometary feature was extracted using the quikstone system with a steel microblade. After synchrotron X-ray-based tomography study (Ishii et al. 2007), this quikstone was subdivided again using an ultrasonic diamond microblade into three blocks (bulb+stylus+terminal) for more detailed study. Figure 6b shows an example of microsurgery on a track using the quikstone system. A 1mm^3 sized quikstone including a part of a 4.7mm-long bulb track was dissected using an ultrasonic diamond microblade attached to the quikstone system.

Cometary grain extraction from dissected aerogel

All samples are extensively photo-documented at every step of the extraction and subdivision processes. In many cases, the extraction and subdivision processes are also recorded by CCD cameras attached to the microscopes. This level of photo-documentation identifies micro-scale tracks and grain features in greatest detail.

For example, in Figure 7 we show a series of images acquired after extracting the keystone produced for cometary track 48 (from aerogel tile C2027; a 1 mm-long track). Figure 7a is a photo mosaic of the entire keystone on a microforklift viewed by transmitted light with a Nikon compound microscope with a 10X objective lens. Individual cometary grains along the track are not recognizable at this scale. Figure 7b is a higher magnification mosaic image of the track 48 showing more details of the feature. For this mosaic, 13

individual images were taken under both transmitted and reflected light using a 20X objective lens with variable focus points, and compressed as a 3D image using computer image processing. We also acquired pictures from different rotation angles under the same conditions so that large volume (bulbous) tracks are completely documented at high spatial resolution. Figures 7c and 7d show the boxed area of the track 48 in Figure 7b including the terminal grain in a bright field view with transmitted/reflected combined light (Fig. 7c) and in a dark-field view with a reflected cross-polarized light (Figure 7d), both taken with a 50X objective lens. Careful examination of grains with crossed-polarized light has been effective for distinguishing amorphous from crystalline grains at this level of magnification.

Once a target grain is identified, the dissected aerogel piece with a cometary track is placed under a long working distance stereomicroscope (Leica MZ10 F), and the stage is covered with a mirror plate. Dissected aerogel pieces, keystones or quikstones are typically from 100 μm^3 to several mm^3 in size and extremely light. It is essential to secure these samples in place, because they are easily lost by light air movement or static charges. We use a borosilicate glass microneedle angled parallel to the stage to hold the aerogel in place. The glass microneedle is held with a 3-axis micromanipulator (Sutter MP285), and gently lowered onto the aerogel piece.

All of the grain extractions from the aerogel collector have been performed not by a robotically controlled micromanipulator, but using stable human hands. We have 25 years of experience of this method for handling interplanetary dust particles (IDPs) which are <100 μm in size extraterrestrial dust samples collected in the stratosphere by NASA high altitude airplanes (Warren and Zolensky, 1994). In this program IDPs have been captured using high-viscosity silicone oil that facilitates handling the captured grains minimizing the static charge effect. Extracting submicron-size grains from the Stardust aerogel collector is more difficult since the aerogel is dry and is highly susceptible to static charge. As mentioned earlier we use a ^{210}Po source as a spot ionizer that effectively eliminates static charge accumulation over a several cm area while working with these samples.

The target grain is gradually exposed by carefully removing the surrounding aerogel with a glass needle. Figure 7f shows the end result of this operation, the target grain removed from the keystone. The grain is then temporary stored between two dimpled glass slides for further photo documentation. Proper lighting helps identifying the crystalline grains, and distinguishes the remaining, surrounding, compressed aerogel from cometary material. Figures 7f and 7g show the extracted terminal grain of Track 48; the pictures were taken by a compound microscope with a 50X objective lens. In the dark field image (Fig.7f), only the grain (crystalline) is visible. In the bright field image under transmitted/reflected light, the extracted grain including the surrounding compressed aerogel is visible. The compressed aerogel - partially melted and sintered onto the grain during capture - is impossible to completely remove at this scale.

The grain extraction procedure has been applied to both dissected aerogel pieces and compressed tracks as described in Matrajt and Brownlee (2006). More than 250 grains (1~40 μm in size, average 5μm) have been extracted by this method and studied by infrared and Raman microspectroscopies, synchrotron X-ray diffraction and electron backscattered diffraction.

Cometary material thinning for submicron-nano scale analysis

Some spectroscopic analyses and secondary ion mass spectrometry measurements are influenced by topography of sample surfaces. For such measurements, samples are flattened mechanically before the analysis. Other analytical methods require thinning of specimens so that they are electron or light transparent. Here we describe techniques used to flattening and thin submicron-size cometary grains.

Grain flattening by a micropresser

Sample pressing is a conventional sample preparation method that has been used for infrared (Sandford & Walker, 1985) and Raman microspectroscopy (Wopenka 1987 EPSL) and ion microprobe isotopic measurements (McKeegan 1987 Science) on individual IDPs. For the pressing of submicron-size dust samples, it is critical to keep track of the location and orientation of the sample during the pressing. We are using basically the same micro-sample pressing technique/device that these pioneers of the IDP studies had designed back in mid 1980s'. The press consists of two-piece large brass or stainless steel disks that can be brought together in a controlled fashion (pressed) with micrometer-scale vertical positioning. The samples are pressed with a spectroscopic grade quartz or sapphire disk that is mechanically fixed to the lower surface of the upper portion of the press. These materials have the necessary properties (strong, clean, flat, and transparent) to enable the pressing procedure to be viewed (through a central hole in the upper press) in real time.

The procedure is quite simple. A grain is placed onto a clean substrate with a smooth surface appropriate for the particular analytical technique. We typically use Au (Figure 8a) as a substrate for Raman spectroscopy and ion microprobe isotopic measurements, KBr (Figure 8b) for infrared spectroscopy (Rotundi et al.), and Indium foil (Herzog and Taylor, 2007) for Nuclear Reaction Analysis. The sample mount is placed on the microprocessor base and placed on the stage of a wide working distance stereomicroscope. While viewing the sample with the microscope, the press is gradually lowered toward the sample. At the points where the sapphire or quartz window contacts the sample, the image becomes clearer and nearby areas are marked by Newton's rings. Care must be taken to ensure most of the sample remains on the surface and not the pressing window. The pressed samples are very flat and the surface area becomes larger (Figures 8c and d).

A disadvantage of this sample preparation method is that we lose textural properties of the grains due to the pressing. Since spectroscopic techniques are mostly non-destructive, the pressed samples can be extracted by ultramicrotomy or focused ion beam liftout after the

measurement. During the stardust preliminary examination period, all the grain samples extracted from the aerogel collector were first ultramicrotomed to produce thin sections, and leftover samples in potted butts were extracted from the embedding medium, and pressed using the procedure described above for isotopic measurements by ion microprobes. This type of sample processing enables coordination of analytical studies by many techniques, maximizing the science return from a single grain.

Ultramicrotomy: Embedding

Ultramicrotomy produces continuous thin sections of 50~200 nm thickness from a submicron size dust sample. Ultramicrotomy is widely used for sectioning biological materials, and the same procedure has been applied for sectioning IDPs for transmission electron microscopy studies (Bradley, 1988). Recent advances now enable analysis of ultramicrotomed thin sections by a variety of analytical techniques, including TEM, XANES, FTIR, and NanoSIMS. With care and careful planning, it is generally possible to perform several of these analyses on the same thin section.

The embedding media for Wild 2 grains were EMBED-812 low-viscosity epoxy (Figure 9), sulfur (Figure 10), cyanoacrylate, and Weld-on 40 acrylic (Matrajt & Brownlee, 2006). With the exception of epoxy-embedded samples, grains can be readily removed from the embedding media. Acrylic and cyanoacrylate can be removed with common organic solvents, such as acetone and chloroform, permitting subsequent isotopic or bulk compositional analyses. Sulfur is easily removed by mild vacuum heating (70ºC). We embed pieces of aerogel in EMBED-812 epoxy, during which the aerogel became completely invisible (Figure 9b), revealing all of the grains in a track in the most complete manner (Barrett 1992). When it was desirable to make superior organic analyses of grains following ultramicrotomy we used high-purity sulfur as the embedding medium, as has been the standard practice for IDPs and fine-grained chondritic meteorites. When using sulfur as an embedding medium, S was sublimed prior to analysis of organic matter in the sample such as C- and N-XANES, FTIR and light-element isotopic analysis in NanoSIMS. Sulfur was chosen as an embedding medium to avoid contamination of the samples with low-viscosity resin (epoxy) normally used for ultramicrotomy. Sulfur beads containing the samples were attached to a sample holding an epoxy bullet using a cyanoacrylate adhesive. To evaluate the potential glue contribution to the sample analysis, sulfur beads devoid of sample were prepared in the same manner. We did not see any evidence that cyanoacrylate penetrated the S bead during subsequent TEM investigation of the sample-free S slices. Electron energy-loss spectroscopy (EELS) spectra acquired from the S test slices also did not show evidence of the pronounced CN peak characteristic of cyanoacrylate.

Ultramicrotomy: Slicing and mounting

After the embedding media has cured, Comet Wild 2 grains were sliced into 50~ 300 nm-thick sections with an ultramicrotome (Leica EM UC6) equipped with a diamond knife

(Diatome ultra35 degree). The sections were floated onto ultra-pure water and transferred to transmission electron microscopy (TEM) grids or special sample mounts, depending upon their intended use. The thickness of the sections can be accurately controlled during the sectioning process. The color of a thin section in reflected light gives an indication of its thickness (Peachey, 1958); the reproducibility of the thickness control is excellent on this ultramicrotome (Figure 11).

We use standard 3 mm diameter TEM grids, made of either pure Cu or pure Au. Be grids are also available by special request. These grids are covered with supporting films directly deposited onto the grids. Material of the supporting films is either amorphous carbon, Quantifoil® holey carbon, pure silicon monoxide, or ultrathin carbon film, depending on the intended analytical methods. Silicon monoxide (15-30 nm thickness) has low background contrast, and is stable under the electron beam. TEM grids supported by silicon monoxide are mainly used for synchrotron XANES analysis. Ultrathin carbon film is thinner (3-4 nm thickness) than normal amorphous carbon film (20-30 nm thickness) and is mounted on a carbon holey film. This is particularly useful for high-resolution microscopy of low-contrast grains.

Some ultramicrotomed thin sections have also been mounted on silicon nitride membrane windows (window size: 3 mm on 10x10mm silicon wafer) for synchrotron X-ray microscopy, and both silicon wafers (5x 5 mm) and custom made Au mounts for isotopic measurements.

1~3 thin sections are typically mounted on a grid. We usually mount thin sections from each Wild 2 grain on 8 Cu or Au TEM grids with amorphous carbon supporting films for general mineralogical/crystallographic study and isotopic analysis by NanoSIMS, and one Cu grid with SiO for X-ray microscopic analysis.

Ultramicrotomy: Potted butts

The advantage of ultramicrotome thin sectioning is that we can preserve overall sample structure at the nano-scale and generate dozens of slices from a single grain. The principal disadvantage of ultramicrotoming is structural damage by chattering (Reid, 1975). This problem is pronounced with large (>1μm) crystal grains that are hard and brittle, which tend to fracture during sectioning by a diamond knife (Figure 12). If not recognized, this chattering artifact might be interpreted as structural feature indigenous to the sample. For example, Figure 12b is a bright field TEM image of an ultramicrotomed thin section of a track 32 terminal grain. This grain is dominated by enstatite surrounded by fine-grained chondritic material. The parallel lines in the grain (weak contrast) are twinning of enstatite, which is an indigenous crystallographic feature. However, the vertical lines which forms the platy structure are s a sectioning artifact (chattering).

Potted butts are good for SEM, microprobe analysis and EBSD analysis. However, one problem we encountered was that embedding medium, especially cyanoacrylate,

polymerizes in an electron beam, making subsequent grain removal difficult.

Removal of Foils Covering the Tray Frame

The frame bars of the aerogel tray were wrapped with small pieces of aluminum foil which had two main purposes, the most important of which was to permit the removal of the aerogel cells with minimal disruption. The aerogel cells were pressure fit into the tray, which made cell removal difficult. The foils provided a handle permitting workers to pull the cells from the tray without actually touching the delicate aerogel. In practice this system works reasonably well, although in any future mission we would not recommend pressure-fitting the aerogel in a sample tray, as the stressed introduced into the aerogel made some cells crack, and at least one virtually disintegrated upon removal.

The second reason for using aluminum foil to surround the tray was to provide a useful surface for survey and analysis of the smallest coma grains – grains so small that their presence in the aerogel cells might be difficult to document. Based upon our experience with the Long Duration Exposure Facility (LDEF), we expected that very few (1-5%) of small craters in aluminum would contain analyzable impactor residue (Bernhard et al., 1992; Amari et al., 1992; Zolensky et al., 1994). To our surprise, many of the craters in the foils contain analyzable residue materials. In fact, the first true presolar grain found amongst the samples was within a penetration hole in aluminum foil (McKeegan et al., 2006). Thus, the cleanliness of the aluminum foil has become a major issue in sample analysis, and we regret that we did not expend more resources doing a better job of cleaning up this foil before flight. This is another example of our new maxim that you should prepare to be more successful than you imagined.

Preliminary Examination of the Samples

A major issue that we managed to successfully address was the magnitude and manner of preliminary examination (PET) of the returned samples (Brownlee et al., 2006). Not since Apollo and Luna days had anyone faced this issue, and the lessons of Apollo PET were not extremely useful because of the very different sample masses in this case, and the incredible advances in analytical capabilities since the 1960s. Everyone agreed that there needed to be some determination of the state and quantity of the returned samples, to provide a necessary guide to both samples requesters and the inevitable oversight committee tasked with sample curation oversight. The heart of our controversy was just how far the preliminary characterization of the samples should proceed. Opinions varied from “do nothing else” to “do all that can reasonably and reliably be done in a short period of time”. Another issue was just who would be permitted to make the analyses, and what the ground rules for participation would be. After *considerable* discussion with the Curation and Analysis Planning Team for Extraterrestrial Materials (CAPTEM) we finally all agreed that

we would make the preliminary examination as comprehensive as possible, and to make this action fair, to also be as inclusive as reasonable with respect to the PET team. We divided the PET effort into six parallel and interrelated efforts, with a science team member at the head of each group. We added two members to the science team to fill all the leader slots, since the science team staffing during the mission itself was limited. These efforts were (1) Bulk Composition, (2) Mineralogy and Petrology, (3) Organics, (4) Optical Properties, (5) Isotopes, and (6) Small Craters in Aluminum. All qualified scientists were invited to join any number of these groups, provided they met some minimal background requirements and agreed to publish all results during the PET effort as groups. All of the initial results were reported together in Science magazine (Brownlee et al., 2006; Hörz et al., 2006; Sandford et al., 2006; McKeegan et al., 2006; Keller et al., 2006; Flynn et al., 2006; Zolensky et al., 2006).

Initially we limited PET participation to PhDs with prior experience with analysis of fine-grained materials. As the effort progressed these rules were relaxed to permit new techniques to be employed and new expertise to be involved. There were no major problems during PET that could not be resolved amicably. An attractive result of this exercise was the entry of numerous new groups into the astromaterials field and the formation of very powerful new collaborations, some of which have lasted to the present and will facilitate the eventual PET for the Stardust interstellar tray (Westphal et al., 2008). Thus, the entire field of planetary materials benefited from the Stardust PET effort.

The PET was designed to proceed from the least invasive analyses through marginally destructive ones, and finally to some completely destructive procedures, to maximize the data harvest from minimal sample mass (Zolensky et al., 2000). Thus we began many analysis trees using synchrotron X-ray fluorescence (SXRF), synchrotron tomography (SCT), and/or scanning transmission X-ray microscopy (STXM) of entire keystoned tracks, before actually removing individual grains from the tracks for analysis. These analyses enabled us to focus later characterization efforts on the most interesting captured grains, that would then be removed from the aerogel. Of course we did not always have the time to follow this incremental analytical protocol during PET, but it was a model we followed whenever possible. For these separated grains we usually performed Vis-IR spectroscopy before proceeding to ultramicrotomy, isotopic, mineralogic or organic analyses of sections of grains. Table 1 lists the most commonly applied analytical techniques for nanogram-sized astromaterials, along with their relative, rough level of sample destructiveness (modified after Zolensky et al., 2000). The techniques actually applied to Stardust samples during PET are underlined. Considering the short time (9 months) available for sample PET the range of applied analyses is remarkable, reflecting the value of the returned samples and the depth and dedication of the sample community. When we began to test silica aerogel as a potential capture media for cometary coma grains, in the mid-1980s, the list of available analytical

techniques was far shorter than what it is today, and the roster of nanogram-able sample analysts in the astromaterials community was far smaller. A principal value of a returned sample over what may be accomplished remotely is that the samples can be reanalyzed as new techniques are developed and new ideas and hypotheses are proposed. As long as we continue to take good care of the Wild 2 samples, we can expect far more and improved analyses to be made of them in the coming decades.

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Because this paper was written from a purely curatorial perspective, many persons who contributed mightily to the Stardust Mission are not co-authors, despite the fact that they deserve complete credit for much of the critical work described herein. Andrew Westphal and Christopher Snead originated and developed the keystone system, which has been so integral to the success of the sample analysis; Hope Ishii and John Bradley developed the quikstone technique, and Hope, Andrew and Christopher spent many hours at JSC performing critical shakedown duty with the freshly-returned samples. David Blake, Kathie Thomas-Keprta and John Bradley developed the ultramicrotomy techniques so integral to astromaterials characterization – many people are unaware of David's contribution because he was the first person in our community to use this technique twenty years ago. The actual recovery of the Stardust spacecraft was largely planned and performed by Lockheed Martin troops led by Mike McGee with the able assistance of Scott Sandford and Karen McNamara, all of whom spent literally months performing recovery rehearsals, living in run-down gambling casinos and subsisting on submarine sandwiches. Reviews of the original version of this paper by Scott Sandford, Steve Sutton and an anonymous reviewer greatly improved the manuscript. The Stardust Mission owes it existence to Don Brownlee, Peter Tsou and Ben Clark, who dreamed it up, Joe Velinga, Ken Atkins and Tom Duxbury who managed it, and Chen Wan Yen, who discovered the spectacular option of a comet sampling encounter (the original goal of the mission was solely the interstellar dust collection). CAPTEM demanded, and NASA Headquarters, principally in the persons of Marilyn and David Lindstrom and Tom Morgan, happily "found" the funding necessary for recovery support, the curation lab and facilities, and PET support – the original Discovery AO made no mention of sample curation as a necessary budget consideration. Carl Allen (Head of NASA's Curation Office), Eileen Stansberry and Steve Hawley smoothed the waters at JSC and killed many sharks. Fred Hörz, Gerald Haynes, Frank Cardenas and Peter Tsou designed and built and endlessly all of the tray and aerogel deintegration hardware, which played the key role in the smooth SRC deintegration. Almost two hundred scientists worldwide found the time, support and patience to work together on the preliminary analysis of the first comet samples returned to Earth – in some ways this was the most amazing event of all.

References

Amari S., Foote J., Swan P., Walker R.M., Zinner E., Lange G. (1992) SIMS chemical analysis of extended impacts on the leading and trailing edges of LDEF Experiment AO187-2. *LDEF-69 Months in Space.* Second Post-Retrieval Symposium, NASA Conference Publication 3194, 513-528.

Bernhard R.P., Durin C. and Zolensky M.E. (1993) Scanning electron microscope/energy dispersive X-ray analysis of impact residues in LDEF tray clamps. *LDEF-69 Months in Space.* Second Post-Retrieval Symposium, NASA Conference Publication 3194, 541-549.

Bradley J.P. (1988) Analysis of chondritic interplanetary dust thin-sections. *Geochimica et Cosmochimica Acta* **52**, 889-900.

Brownlee D., Tsou P., Aléon J., Alexander C.M.O'D., Araki T., Bajt S., Baratta G.A., Bastien R., Bland P., Bleuet P., Borg J., Bradley J.P., Brearley A., Brenker F., Brennan S., Bridges J.C. Browning N., Brucato J.R., Brucato H., Bullock E., Burchell M.J., Busemann H., Butterworth A., Chaussidon M., Cheuvront A., Chi M., Cintala M.J, Clark B.C., Clemett S.J., Cody G., Colangeli L., Cooper G., Cordier P.G., Daghlian, C., Dai Z., D'Hendecourt L., Djouadi Z., Dominguez G., Duxbury T., Dworkin J.P., Ebel D., Economou T.E., Fairey S.A. J., Fallon S., Ferrini G., Ferroir T. Fleckenstein H., Floss C., Flynn G., Franchi I. A., Fries M., Gainsforth Z., Gallien J.- P., Genge M., Gilles M.K., Gillet P., Gilmour J., Glavin D.P., Gounelle M., Grady M.M., Graham G.A., Grant P.G., Green S.F., Grossemy F., Grossman L., Grossman J., Guan Y., Hagiya K., Harvey R., Heck P., Herzog G.F., Hoppe P., Hörz F., Huth J., Hutcheon I.D., Ishii H., Ito M., Jacob D., Jacobsen C., Jacobsen S., Joswiak D., Kearsley A.T., Keller L., Khodja H., Kilcoyne A.L.D., Kissel J., Krot A., Langenhorst F., Lanzirotti A., Le L., Leshin L., Leitner J., Lemelle L., Leroux H., Liu M.-C., Luening K., Lyon I., MacPherson G., Marcus M.A., Marhas K., Matrajt G., Meibom A., Mennella V., Messenger K., Mikouchi T., Mostefaoui S., Nakamura T., Nakano T., Newville M., Nittler L. R., Ohnishi I., Ohsumi K., Okudaira K., Papanastassiou D. A., Palma R., Palumbo M. O., Pepin R.E., Perkins D., Perronnet M., Pianetta P., Rao W., Rietmeijer F., Robert F., Rost D., Rotundi A., Ryan R., Sandford S.A., Schwandt C.S., See,T. H., Schlutter D., Sheffield-Parker J.A., Simionovici S., Sitnitsky S.I., Snead C.J., Spencer M.K., Stadermann F.J., Steele A., Stephan T., Stroud R., Susini J.,. Sutton S.R., Taheri M., Taylor S., Teslich N., Tomeoka K., Tomioka N., Toppani A., Trigo-Rodríguez J.M., Troadec D., Tsuchiyama A., Tuzolino A.J., Tyliszczak,T., Uesugi K., Velbel M., Vellenga J., Vicenzi E., Vincze L., Warren J., Weber I., Weisberg M., Westphal A. J., Wirick S., Wooden D., Wopenka B., Wozniakiewicz P.A., Wright I., Yabuta H., Yano H., Young E.D., Zare R.N., Zega T., Ziegler K.,

Zimmerman L., Zinner E. and Zolensky M. (2006) Comet Wild 2 under a microscope. *Science* **314**, 1711-1716.

Chi M., Ishii H., Dai Z.R., Toppani A., Joswiak D.J., Leroux H., Zolensky M., Keller L.P., Browning D., and Bradley J.P. (2007) Does Comet Wild 2 contain GEMS? *LPSC* **38**, 2010.

Cody G.D., Yabuta H., Alexander C.M.O'd., Araki T., Kilcoyne A.L.D., and the Stardust PET (2007) Placing Comet 81p/Wild 2 organic particles into context with chondritic organic solids. *LPSC* **38**, 2286.

Flynn G..J., Bleuet P., Borg J., Bradley J., Brenker F., Brennan S., Bridges J., Brownlee D.E., Bullock E., Clark B.C., Dai Z.R., Daghlian C., Djouadi Z., Fakra S., Ferroir T., Floss C., Franchi I., Gainsforth Z., Gallien J.-P., Gillet Ph., Grant P.G., Graham G.A., Green S., Grossemy F., Heck P., Herzog G.F., Hoppe P., Hörz F. , Huth J., Ignatyev K., Ishii H., Joswiak D., Kearsley A.T., Khodja H., Lanzirotti A., Leitner J., Lemelle L., Leroux H., Luening K., MacPherson G., Marhas K., Marcus M., Matrajt G., Nakamura T., Nakano T., Newville M., Papanastassiou D., Pianetta P., Rao W., Rietmeijer F., D. Rost, Schwandt C., See T., Sheffield-Parker J., Simionovici A., Sitnitsky I., Snead C., Stadermann F., Stephan T., Stroud R., Susini J., Suzuki Y., Sutton S., Taylor S.R., Teslich N., Troadec D., Tsou P., Tsuchiyama A., Uesugi K., Westphal A., Wozniakiewicz P., Vicenzi E., Vincze L., Zolensky M.E. (2006) Elemental Compositions of Comet 81P/Wild 2 Samples Collected by Stardust. *Science* **314**, 1731-1735.

Gallien J.-P., Khodja H., Herzog G.F., Taylor S., Koepsell E., Daghlian C.P., Flynn G..J., Sitnitsky I., Lanzirotti A., Sutton S.R., Keller L.P. (2007) Characterization of Three Carbon- and Nitrogen-rich Particles from Comet 81P/Wild 2. *Lunar and Planetary Science XXXVIII*, LPI, 1605

Hörz F., Bastien R., Borg J., Bradley J.P., Bridges J.C., Brownlee D.E., Burchell M.J., Chi M., Cintala M., Dai Z., Djouadi Z., Dominguez G., Economou T.E., Fairey S., Floss C., Franchi I.A., Graham G.A., Green S.F., Heck P., Hoppe P., Huth J., Hope Ishii [4], Kearsley A.T., Kissel J., Leitner J., Leroux H., Marhas K., Messenger K., Schwandt C.S., See T.H., Snead C., Stadermann F.J., Stephan T., Rhonda Stroud [17], Teslich N., Trigo-Rodríguez J., Tuzzolino A.J., Troadec D., Tsou P., Warren J., Westphal A., Wozniakiewicz P., Wright I., Zinner E. (2006) Impact features on Stardust: Implications for Comet 81P/Wild 2 dust. *Science* **314**, 1716-1719.

Ishii H.A., Graham G.A., Kearsley A.T., Grant P.G., Snead C.J., Bradley J. P. (2005) *Meteoritics & Planetary Science* **40**, 1741-1747

Ishii H.A. and Bradley J.P. (2006) Macroscopic subdivision of silica aerogel collectors for sample return missions. *Meteoritics & Planetary Science* **41**, 233-236

Ishii H.A., Teslich N., Graham G.A., Sheffield-Parker J., Bradley J.P. Stardust in 3-D: X-ray

micro-tomography of comet dust impacts. Submitted to *MAPS*.

Keller L.P., Bajt S., Baratta G.A., Borg J., Busemann H., Brucato J., Burchell M., Colangeli L., d'Hendecourt L., Djouadi Z., Ferrini G., Flynn G., Franchi I., Fries M., Grady M., Graham G.A., Grossemy F., Kearsley A., Matrajt G., Mennella V., Nittler L., Palumbo M., Rotundi A., Sandford S.A., Steele A., Wooden D., Wopenka B., Zolensky M.E. (2006) Infrared, UV/Vis And Raman Spectroscopy Of Comet Wild-2 Samples Returned By The Stardust Mission. *Science* **314,** 1728-1731.

Leroux H., Rietmeijer F. J.M, Velbel M.A., Brearley A.J., Jacob D, Langenhorst F., Bridges J.C., Zega T.J., Stroud R.M., Cordier P., Harvey R.P., Lee M., Gounelle M., and Zolensky M.E. A TEM study of thermally modified Comet 81P/Wild 2 dust particles by interactions with the aerogel matrix during the Stardust capture process. *Meteoritics and Planetary Science,* Submitted a.

Leroux H., Stroud R.M., Dai Z.R., Graham G., Troadec D., Bradley J., Teslich N., Borg J., Kearsley A. and Hörz F. Transmission electron microscopy of cometary residues from micron-sized craters in the Stardust Al-foils. *Meteoritics and Planetary Science,* Submitted b.

Matrajt G. and Brownlee D.E. (2006) Acrylic embedding of Stardust particles encased in aerogel. *Meteoritics & Planetary Sciences* **41**, 1715-1720.

McKeegan K.D. (1987) Oxygen isotopes in refractory stratospheric dust particles - Proof of extraterrestial origin. *Science* **237**, 1468-1471.

McKeegan K.D., Aléon J., Bradley J., Brownlee D., Busemann H., Butterworth A., Chaussidon M., Fallon S., Floss C., Gilmour J., Gounelle M., Graham G., Guan Y., Heck P.R., Hoppe P., Hutcheon I.D., Huth J., Ishii H., Ito M., Jacobsen S.B., Kearsley A., Leshin L.A., Liu M.-C., Lyon I., Marhas K., Marty B., Matrajt G., Meibom A., Messenger S., Mostefaoui S., Mukhopadhyay S., Nakamura-Messenger K., Nittler L., Palma R., Pepin R.O., Papanastassiou D.A., Robert F., Schlutter D., Snead S.J., Stadermann F.J Stroud R., Tsou P., Westphal A., Young E.D., Ziegler K., Zimmermann L., Zinner E. (2006**)** Isotopic Compositions of Cometary Matter Returned by Stardust. *Science* **314,** 1724-1728.

Mikouchi T., Tachikawa K., Hagiya K., Ohsumi K., and Zolensky M. (2007) Mineralogy and crystallography of Comet 81p/Wild 2 particles returned by the Stardust Mission. *Lunar and Planetary Science XXXVIII*, 1946.

Peachey L.D. (1958) Thin sections. I. A study of section thickness and physical distortion produced during microtomy. *J. Biophys. Biochem. Cytol*. **4**, 233-242.

Reid N. 1975. Ultramicrotomy. In *Practical Methods in Electron Microscopy*. Editors Glauert, A.M., Elsevier, 213-353.

Rotundi A. et al. Combined Micro-IR and Micro-Raman Analyses of Comet 81P/Wild 2 Particles Collected by Stardust. Submitted to MAPS

Sandford S.A., Aléon J., Alexander C., Araki T., Bajt S., Baratta G., Borg J., Brucato J., Burchell M., Busemann H., Butterworth A., Clemett S., Cody G., Colangeli L., Cooper G., D'Hendecourt L., Djouadi Z., Dworkin J., Ferrini G., Fleckenstein H., Flynn G., Franchi I., Fries M., Gilles M., Glavin D., Gounelle M., Grossemy F., Jacobsen C., Keller L., Kilcoyne A., Leitner J., Matrajt G., Meibom A., Mennella V., Mostefaoui S., Nittler L., Palumbo M., Robert F., Rotundi A., Snead C., Spencer M., Steele A., Stephan T., Tyliszczak T., Westphal A., Wirick S., Wopenka B., Yabuta H., Zare R., Zolensky M. (2006) Organics Captured from Comet Wild 2 by the Stardust Spacecraft. *Science* **314**, 1720-1724.

Sandford S.A., Walker R.M. 1985. Laboratory infrared transmission spectra of individual interplanetary dust particles from 2.5 to 25 microns. *Astrophysical Journal* **291**, 838-851

See T., Allbrooks M., Atkinson D. and Zolensky M.E. (1990) *Meteoroid and Debris Impact Features on the Long Duration Exposure Facility*. Planetary Science Branch Publication 84, NASA Johnson Space Center, Houston, 586p.

Stephan T., Flynn G.J., Sandford S.A. and Zolensky M.E. (2007) TOF-SIMS analysis of Comet Wild 2 particles extracted from Stardust aerogel. *LPSC* **38**, 1126.

Westphal A.J., Snead C., Butterworth A., Graham G. A., Bradley J. P., Bajt S., Grant P. G., Bench G.. Brennan S., Pianetta P. (2004) Aerogel keystones: Extraction of complete hypervelocity impact events from aerogel collectors. *Meteoritics & Planetary Science* **39**, 1375-1386.

Wopenka B. (1988) Raman observations on individual interplanetary dust particles. *Earth and Planetary Science Letters* **88**, 221-231.

Warren J.L. and Zolensky M.E. (1994) Collection and Curation of Interplanetary Dust Particles Recovered from the Stratosphere by NASA. In *Analysis of Interplanetary Dust*, Edited by M.E. Zolensky, T.L. Wilson, F.J.M. Rietmeijer, and G..J. Flynn., AIP Conference Proceedings Vol. 310, 245.

Wirick S., Leroux H., Tomeoka K., Zolensky M, Flynn G., Tyliszczak T., Butterworth A, Tomioka N., Ohnishi I, Nakamura-Messenger K, Sandford S, Keller L., Jacobsen C. (2007) Carbonates found in Stardust aerogel tracks. *LPSC 38*, 1354.

Zolensky M.E., Zega T., Yano H., Wirick S., Westphal A., Mike K. Iris Weber I., Warren J.L., Velbel M., Tsuchiyama A., Tsou P., Toppani A., Tomioka N., Tomeoka K., Teslich N., Taheri M., Susini J., Stroud R., Stephan T., Stadermann F., Snead C., Simon S., Simionovici A., See T., Robert F., Rietmeijer F., Rao W., Perronnet M., Papanastassiou D., Okudaira K., Ohsumi K., Ohnishi I., Nakamura-Messenger K., Nakamura T., Mostefaoui S., Mikouchi T., Meibom A., Matrajt G., Marcus M., Leroux H., Lemelle L., Le L., Lanzirotti A., Langenhorst F., Krot A., Keller L., Kearsley A., Joswiak D., Jacob D., Ishii H., Harvey R., Hagiya K., Grossman L.,

Grossman J., Graham G., Gounelle M., Gillet P., Genge M., Flynn G., Ferroir T., Fallon S., Ebel D., Dai Z.-R., Cordier P., Clark B., Chi M., Butterworth A., Brownlee D., Bridges J., Brennan S., Brearley A., Bradley J., Bleuet P., Bland P., Bastien R. (2006) Mineralogy and Petrology of Comet Wild 2 Nucleus Samples. *Science* **314**, 1735-1739.

Zolensky M.E. and Girard T. (1997) Stardust Spacecraft Program Contamination Control Plan. JSC Publication 27954, 26p.

Zolensky M.E., Hörz F., See T., Bernhard R.P., Barrett R.A., Mack K., Warren J.L. Kinard W.L. (1994) Meteoroid investigations using the Long Duration Exposure Facility. In Analysis of Interplanetary Dust (Eds. M. Zolensky, T. Wilson, F. Rietmeijer and G. Flynn), American Institute of Physics, New York, pp. 291-304.

Zolensky M.E., Kinard W.H. (1993) Results of the LDEF meteoroid and debris special investigation group. *Advances in Space Research* **13**, (8)75-(8)85.

Zolensky M.E., Pieters C., Clark B. Papike J. (2000) Small is beautiful; Analysis of nanogram-sized astromaterials. *Meteoritics and Planetary Science* **35**, 9-29.

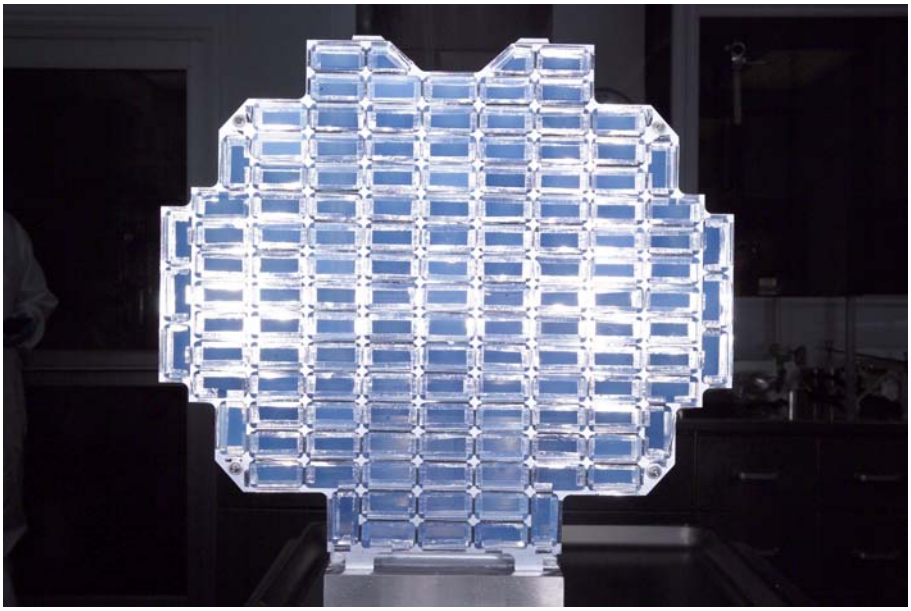
Table 1. Summary of Analytical Techniques Available to Nanogram-sized Samples; Analyses Performed During Stardust PET are Underlined

Technique	Destructiveness
Imaging	
Light-Optical Techniques	non-destructive
Scanning Electron Microscopy/ Energy Dispersive Spectrometry	non-destructive
Synchrotron Tomography	non-destructive
Transmission/Analytical Electron Microscopy	partially
Scanning Transmission X-Ray Microscopy	partially
Atomic Force Microscopy	partially
Force Spectroscopy	partially
Holographic Low-Energy Electron Diffraction	partially
SIMS Ion Imaging	destructive
Bulk and Mineral Compositional Analyses	
Microparticle Instrumental Neutron Activation Analysis	non-destructive
Synchrotron X-ray Fluorescence	non-destructive
XRF Tomography	non-destructive
Scanning Transmission X-ray Microscopy	non-destructive
Micro Raman Spectroscopy	non-destructive
Electron Microprobe Analysis	partially
Protron Induced X-ray Emission	partially
X-ray Spectroscopy	partially
Secondary Ion Mass Spectrometry (incl the Nano persuasion)	destructive
Time-of-Flight Secondary Ion Mass Spectrometry	destructive
Laser Ablation Microprobe- Inductively Coupled Plasma-Mass Spectrometry	destructive
Double Focusing Secondary Ion Mass Spectrometry	destructive
Resonance Ion Mass Spectrometry	destructive
Thermal Ionization Mass Spectrometry	destructive
Organic Analyses	
Micro Raman Spectroscopy	non-destructive
Fluorescence	non-destructive
Electron Energy-Loss Near Edge Structure	partially
Scanning Transmission X-Ray Microscopy	partially
Transmission and Reflectance IR-Vis Spectroscopy	partially
Optically- and Acoustically-Excited Phonon Spectroscopy	partially

Time-of-Flight Secondary Ion Mass Spectrometry	destructive
Chromatography	destructive
Secondary Ion Mass Spectrometry (incl the Nano persuasion)	destructive
Stepped Combustion and Static Mass Spectrometry	destructive
Two-Stage Laser Desorption/Laser Multiphoton Ionization Mass Spectrometry	destructive
Noble Gas and Sample Exposure History	
Solar Flare Track Analysis	partially
Double-Focusing Mass Spectrometer	destructive
Age Dating	
Laser Ablation Mass Spectrometry	destructive
Mineralogy and Atomic Structure	
Synchrotron X-ray Diffraction	non-destructive
X-ray Absorption Spectroscopy	non-destructive
Transmission IR-Vis Spectroscopy	non-destructive
Micro Raman Spectroscopy	non-destructive
Transmission Electron Microscopy	partially
Electron Energy-Loss Near Edge Structure	partially
Atomic Force Microscopy	partially
Electron Energy Loss Spectroscopy	partially
Extended X-ray Absorption Fine Structure	partially
X-ray Absorption Near-edge Structure	partially
IR-Vis Reflectance Spectroscopy	partially
Cathodoluminescence Microscopy and Spectroscopy	partially
Physical Properties	
Density Measurements	non-destructive
Atomic Force Spectroscopy	partially
Magnetic Force Microscopy	partially

Figures

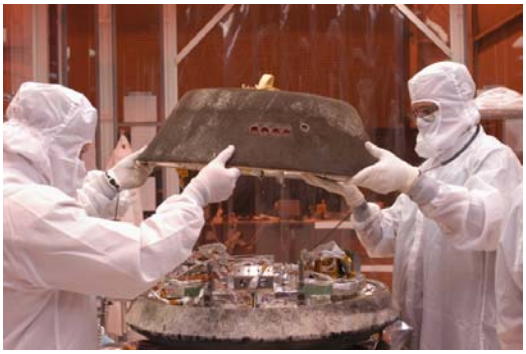
a



b

Figure 1. Stardust aerogel tray. (a) Stardust Science Team's first glance at the sample collector. (b) Back-lit photo of sample collector tray just after first opening in the Stardust Curation Facility.

a



b

Figure 2 Cleanroom at the Utah Test and Training Range (UTTR). (a) The field cleanroom at UTTR. (b) The field recovery team opens Stardust capsule in the UTTR cleanroom hours after recovery.

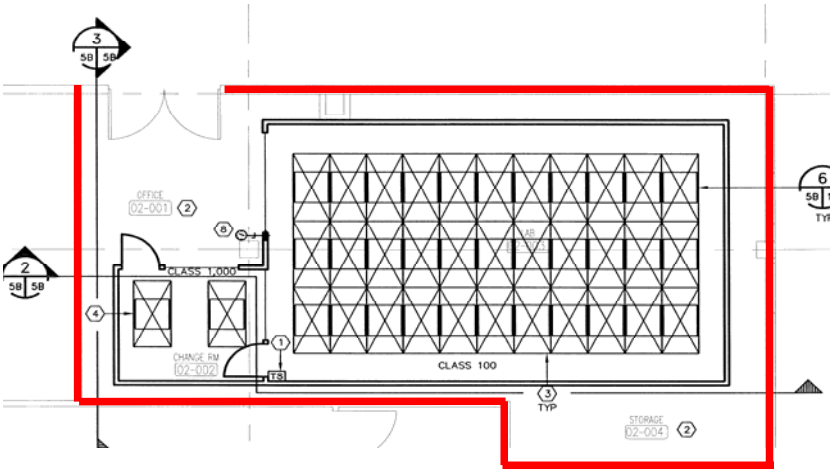


Figure 3. Stardust Curation Facility layout. Outer Class 10,000 cleanroom (red outline) with inner Class 100 modular cleanroom (inside double-black line) and change room (lower left). The large "X" s indicate positions of vertical flow HEPA filters. The full Lab measures 18' by 37'.

a

b

Figure 4: Stardust Curation Lab. (a) View into Stardust Curation class 100 clean room. (b) Entrance to the change room (middle). Clean room is to left.

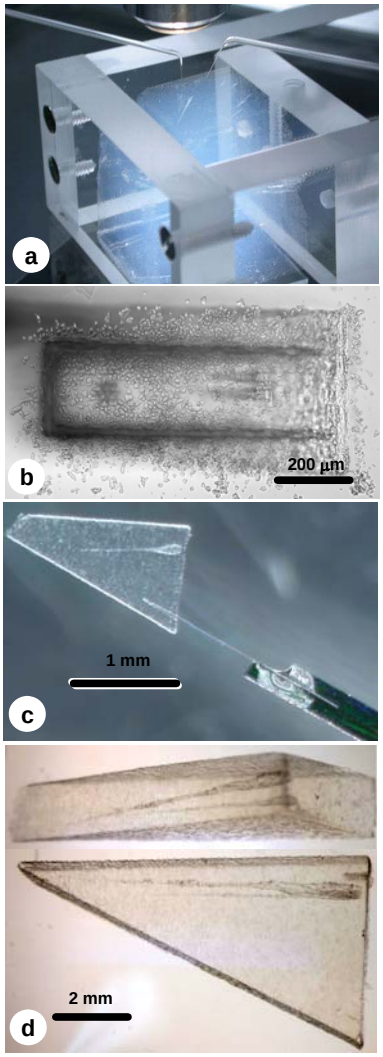


Figure 5. The Keystone System.
(a) Overview of the keystone system. An aerogel collector tile is fixed in a special vise, and is viewed with a compound microscope. Two borosilicate glass microneedles (tip diameter ~ 1µm, length ~1cm) are attached to computer-controlled micromanipulators (not seen in this figure). A wedge-shaped white shadow in the aerogel is the "keystone". (b) Top view of a completed keystone before removal from the aerogel collector tile. A Clamp shape groove is the side cut, and the light contrast vertical line is the entrance of the undercut. A dark round shadow in the middle left inside the keystone is the cometary feature (track #94). Two horizontal lines are holes for supporting microforklift. This aerogel tile C2078 was found to be especially hard, and generated quite an amount of aerogel debris during the operation. (c) A keystone fixed on a microforklift, including a cometary track 104 (1mm-long bulb+stylus). (d) The biggest keystone generated at the JSC Stardust curation facility, viewed from two different directions. This 8.5mm long keystone includes two spiral carrot cometary tracks #99 (7mm long, thinner track in this figure) and #100 (8mm long).

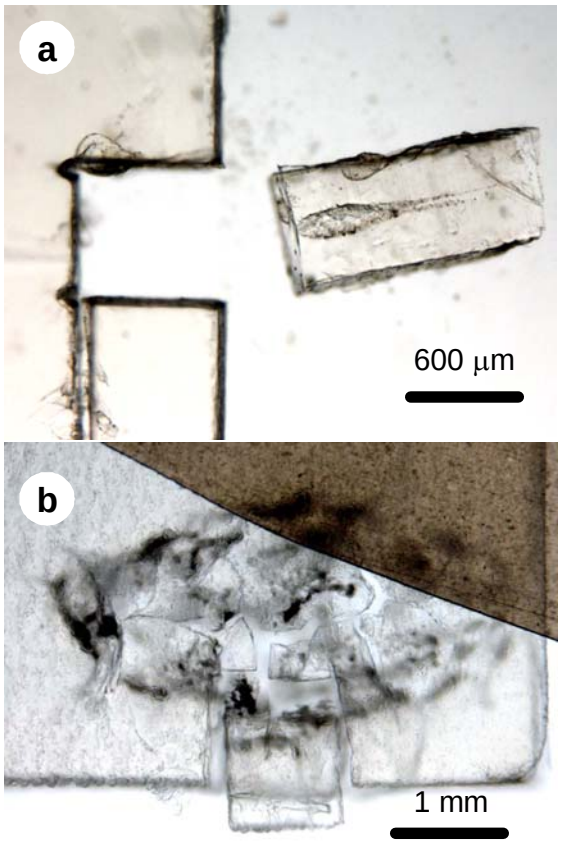


Figure 6. The Quikstone system. (a) A cometary track (track 5) extracted from an aerogel chip with the quikstone system. (b) A 1 mm^3 size quikstone including a part of 4.7mm-long bulb track (track 80) dissected using an ultrasonic diamond microblade attached to the quikstone system.

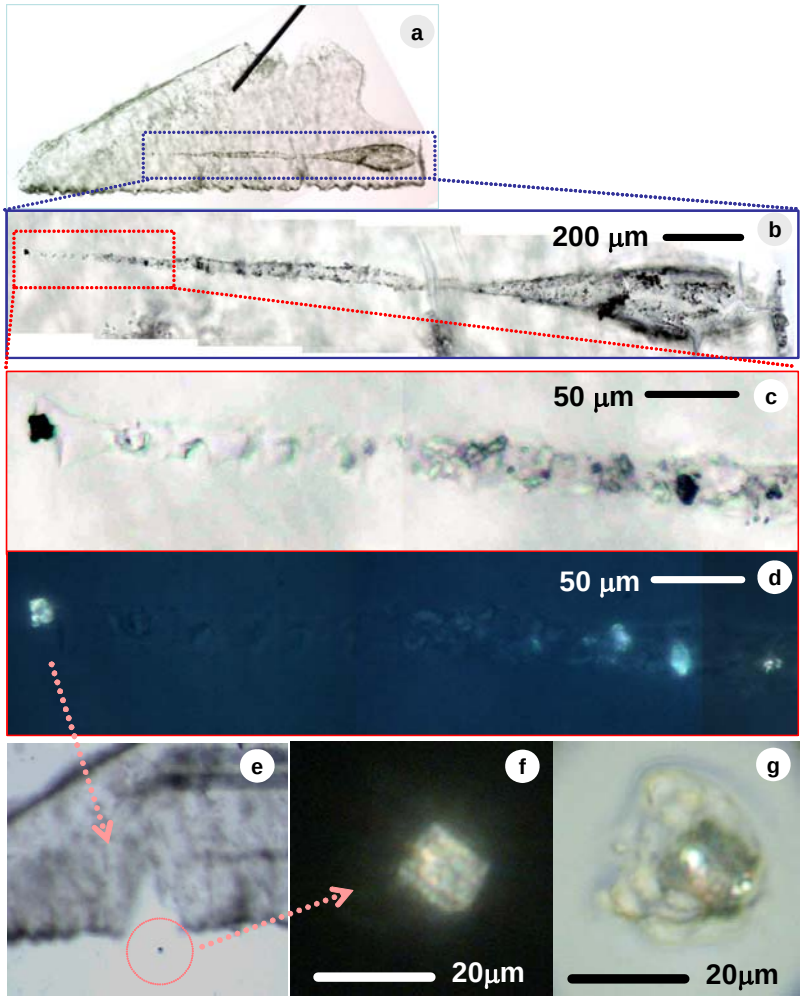


Figure 7. A series of images acquired following the extraction of a keystone including the cometary track 48 (from aerogel tile C2027, 2117 um-long track). (a) A photo mosaic of the entire keystone on a microforklift composed by three pictures taken under transmitted light by a Nikon compound microscope with a 10X objective lens. (b) A higher magnification mosaic image of the track 48 showing more details of the feature. 13 individual images were taken under both transmitted and reflected lights using a 20X objective lens with different focusing, and compressed as a 3D image using computer photo processing. (c) A bright field view of the boxed area of the track 48 in (b) including the terminal grain in with transmitted/reflected combined lights. (d) A dark filed view of the same area with (c) in a reflected cross-polarized light. (e) A captured image from a movie taken by a CCD camera attached to the stereomicroscope, right after the grain was removed from the keystone. (f) A dark field image of the extracted terminal grain of Track 48. (g) A bright field image of the extracted terminal grain.

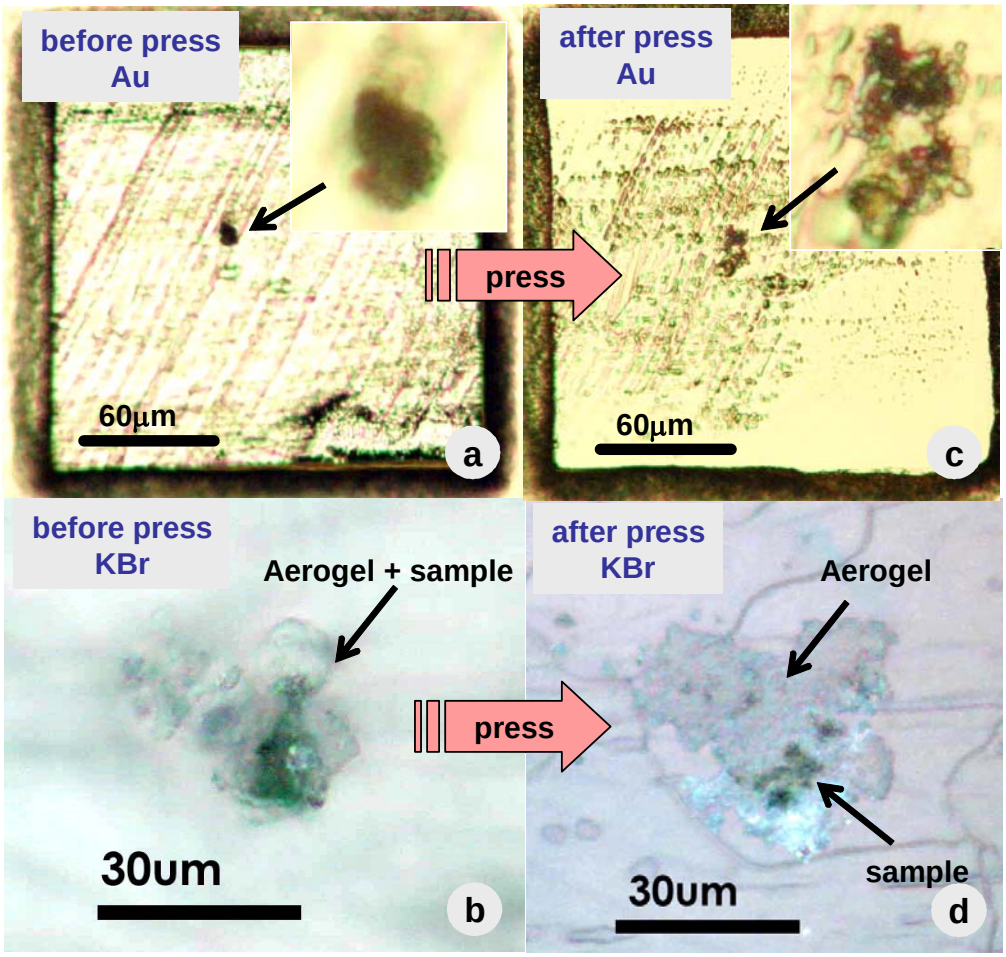


Figure 8. Preparation of a cometary grain for spectroscopy. (a) A cometary particle (C2054, 0,35,91,0) set on a Au mount. (b) A cometary particle (C2054,0,35,87,0) set on a KBr mount. (c) After pressing of the sample in (a). Note that the Au mount surface as well as the sample are flattened. (d) After pressing of the sample in (b).

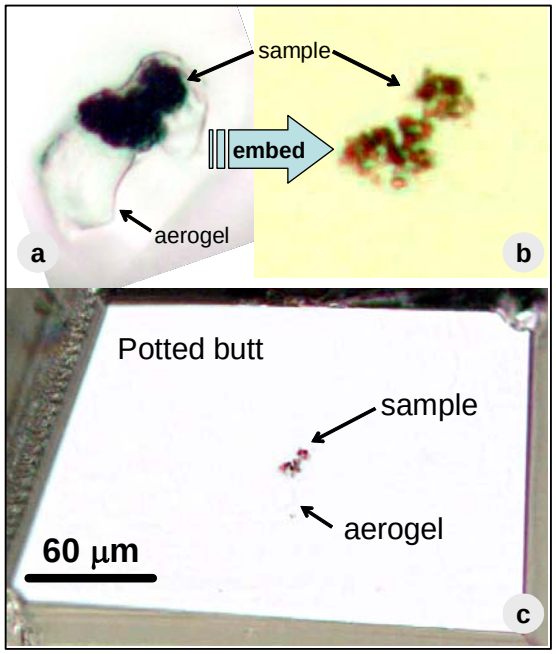


Figure 9. Embedding cometary grains. (a) A Wild 2 cometary grain #16 extracted from track 35 (from C2054 aerogel tile, C2054,0,35,16,0). This grain is surrounded by compressed aerogel observed in the lower and right side of the grain. (b): The same grain of (a) after embedded in low-viscosity Embed 812 epoxy and sliced using an ultramicrotome. The compressed aerogel is almost invisible in the epoxy. The shape of the grain is well preserved. (c) The potted butt of the grain. The ultramicrotomed thin sections from this potted butt are shown in Figure 11.

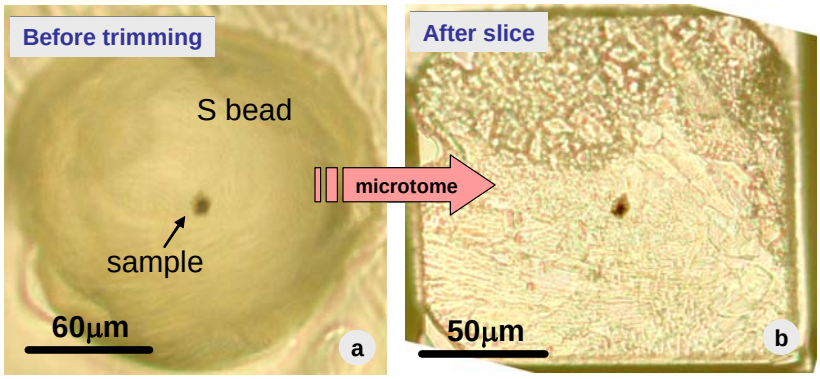


Figure 10. Embedding a cometary grain in beaded sulfur. (a) A pure sulfur potted butt of a Wild 2 cometary grain #1 (the terminal particle) of track 17. The pure sulfur embedding medium is crystalline, nearly transparent, dome shape attached to an epoxy base. (b) The same sample after slicing by ultramicrotome. Sulfur vaporizes immediately, making a patchy structure.

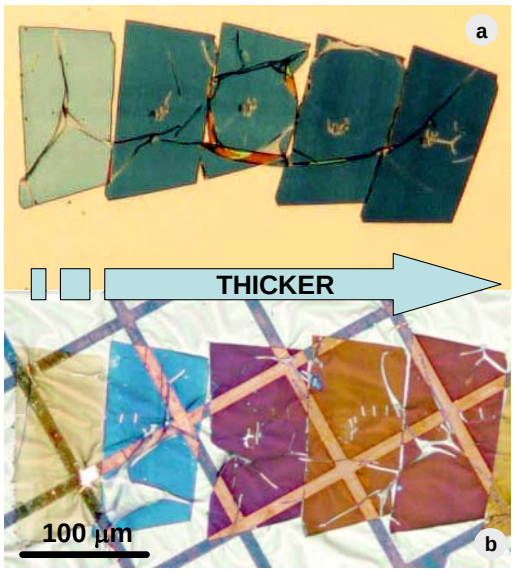


Figure 11. Ultramicrotomed sections. (a) Ultramicrotomed thin sections mounted on a silicon wafer substrate. (b) Ultramicrotomed thin sections mounted on an amorphous carbon film supported Cu TEM grid. These slices are from the same potted butt grain sample shown in Figure 9. The sections show color differences due to varying thicknesses. In (a) the thin section at the far left is 50 nm thick and 100 nm thick at the far right. In (b) the far left thin section is 40 nm in thickness, and the reddish thin section (far right) is 150 nm thick.

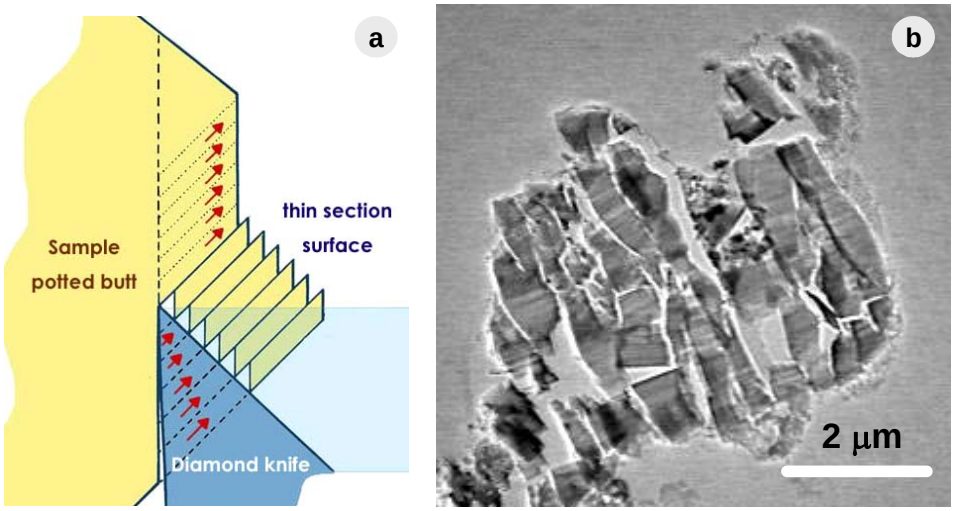


Figure12. Ultramicrotomy-induced damage (chattering). (a) Schematic image of structural damage caused by the diamond knife. Adopted and modified after Reid (1975). (b) A bright field TEM image of an ultramicrotomed thin section of a Wild 2 cometary grain (C2027, 3,32,3,2), one of the terminal grain from so-called Twin tracks 32&69.