

## Applied focused ion beam techniques for sample preparation of astromaterials for integrated nanoanalysis

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*(Submitted 12 February 2007; revision accepted 20 December 2007)*

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**Abstract**—Sample preparation is always a critical step in the study of micrometer-sized astromaterials available for study in the laboratory, whether their subsequent analysis is by electron microscopy or secondary ion mass spectrometry. A focused beam of gallium ions has been used to prepare electron transparent sections from an interplanetary dust particle (IDP), as part of an integrated analysis protocol to maximize the mineralogical, elemental, isotopic, and spectroscopic information extracted from one individual particle. In addition, focused ion beam (FIB) techniques have been employed to extract cometary residue preserved on the rims and walls of microcraters in 1100 series aluminum foils that were wrapped around the sample tray assembly on the Stardust cometary sample collector. Non-ideal surface geometries and inconveniently located regions of interest required creative solutions. These include support pillar construction and relocation of a significant portion of sample to access a region of interest. Serial sectioning, in a manner similar to ultramicrotomy, is a significant development and further demonstrates the unique capabilities of focused ion beam microscopy for sample preparation of astromaterials.

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### INTRODUCTION

Sample preparation of meteoritic material, particularly micrometer-sized interplanetary dust particles (IDPs) or the cometary dust particles recently returned by NASA's Stardust mission (Brownlee et al. 2006), for detailed microanalysis using electron microscopy, secondary ion mass spectrometry (SIMS), or synchrotron-based techniques (e.g., infrared spectroscopy) has long been a fundamental challenge. A variety of novel techniques have been developed and applied to this problem over the past three decades. For IDPs, a significant development was the ability to produce electron transparent (50–100 nm thick) serial sections of individual particles using ultramicrotomy techniques (e.g., Bradley and Brownlee 1986; Bradley 1988). Ultramicrotomy involves slicing an embedded sample with tens-of-nanometers precision using a diamond blade. Since its first utilization in astromaterials research, ultramicrotomy has been the method

of choice for IDP sample preparation whenever such samples were intended for transmission electron microscopy (TEM) studies, although the technique has been modified over the years to enable new analytical measurements. For example, use of sulfur as an embedding medium rather than traditional low-viscosity epoxy alleviated the carbon contamination problem in the preparation medium supporting IDP sections, thereby enabling measurements of the indigenous carbon content (e.g., Bradley et al. 1993). In recent years, other modifications of the embedding medium have included the use of acrylic polymers (Matrajt and Brownlee 2006), yet the principal mechanism for sectioning, i.e., ultramicrotomy, has remained the same (e.g., Joswiak and Brownlee 2006).

Since the late 1990s, focused ion beam (FIB) microscopy has been utilized in both sample preparation and analysis across a diverse range of scientific fields including meteoritics (e.g., Heaney et al. 2001). A detailed description of FIB instrumentation is given in Young and Moore (2005).

The technique uses a focused beam of gallium ions to ablate volumes of material at controlled rates and at precise locations within a sample. This FIB milling can be used to prepare electron transparent sections that can be removed from the bulk sample using either an external glass needle micromanipulator under an optical microscope (e.g., Lee et al. 2003) or a manipulator device within the instrument chamber (e.g., Graham et al. 2006; Zega and Stroud 2006). The sections are then subsequently mounted on TEM grids. The FIB technique makes it possible to prepare site-specific sections from a sample that has had the greatest impact on meteoritic sample preparation. Stroud et al. (2004) highlighted integrated studies of presolar grains from meteorites initially using SIMS for isotopic characterization, followed by use of FIB to extract a cross sectional slice containing the isotopically anomalous material. The section may subsequently be studied using TEM for structural and elemental observations. This integrated approach proved to be a significant advance, as previous studies required complex and time-consuming ultramicrotomy to produce planar TEM sections from an entire sample that had initially been characterized using SIMS (Keller et al. 2004). Herein we discuss the application of FIB to support: (i) integrated studies of an IDP using NanoSIMS, TEM, and synchrotron FTIR analytical techniques and (ii) extraction of cometary residue material deposited within microcraters by hypervelocity capture during the Stardust mission.

## METHODS AND MATERIALS

The IDP L2047,D23 was provided by the cosmic dust curatorial facility at the NASA Johnson Space Center; details of the stratospheric collection, curation, and initial characterization of IDPs are given by Warren and Zolensky (1994). Prior to isotopic imaging at Lawrence Livermore National Laboratory using a Cameca NanoSIMS 50 ion microprobe, L2047,D23 was washed in hexane to remove remaining silicone collector oil. Silicon microtweezers were used to remove the particle from the dimpled glass slide in which it was transported from the cosmic dust curatorial facility; it was then transferred to high-purity Au foil. The particle was pressed into the foil to produce a flat surface suitable for isotopic imaging. Details of the isotopic imaging protocol using NanoSIMS are given in Bradley et al. (2005) and Smith et al. (2005). The FIB extraction of material from L2047,D23 was carried out at FEI Company's Hillsboro demonstration facility using an FEI Strata DB-STEM 237 focused ion beam/field-emission scanning electron microscope fitted with an EDAX Genesis energy-dispersive spectrometer (EDS), an Omniprobe in-situ micromanipulator extraction system and a four-region (bright field, dark field, and two high-angle dark field) SEM-STEM detector. The FIB-extracted electron transparent section of L2047,D23 was analyzed using a Philips CM300 300 kV FEG-TEM fitted

with an Oxford Inca EDS. The Fourier-transform infrared (FTIR) spectroscopic measurements were collected using IR microspectroscopy beamline 1.4.3 at the Advanced Light Source, Lawrence Berkeley National Laboratory.

Aluminum foils containing the microcraters generated by cometary dust particle impacts at 6 km/s were recovered from the Stardust sample tray assembly (Hörz et al. 2006). The two foils (C2086W,1 and C2091N,1) were initially characterized at the Natural History Museum (UK) using a JEOL 5900LV scanning electron microscope (SEM) fitted with an Oxford Inca EDS. The typical analysis conditions were 20 kV and 2 nA. The Cameca NanoSIMS 50 ion microprobe at Washington University in St. Louis was used to determine the C, N, and O isotopic composition of the cometary material at high spatial resolution on the rim of an impact crater ~240  $\mu\text{m}$  in diameter on the surface of foil C2086W,1. Full details of the analytical protocols are given in McKeegan et al. (2006) and Stadermann et al. (2007). FIB extraction of cometary residue material from the two foils was performed at Lawrence Livermore National Laboratory using an FEI Nova 600 Nanolab dual-beam FIB/FESEM microscope with an EDAX Genesis EDS and both an Omniprobe in situ micromanipulator and an Ascend Instruments Extreme Access extraction system.

## INTEGRATED STUDIES OF AN IDP

IDPs represent some of the most primitive and small-scale extraterrestrial material available to study in the laboratory (Bradley 2005). To fully understand an individual IDP, it is necessary to study the different mineralogical, chemical, and isotopic properties that make up the overall composition. As has been shown previously, combined isotopic and mineralogical studies of meteorites and IDPs can yield new insights into the processes of their formation (Stroud et al. 2004; Keller et al. 2004; Bradley et al. 2005). The isotopic images acquired from the analysis of the surface of L2047,D23 using the NanoSIMS identified a small grain ~800 nm in diameter with a large  $^{15}\text{N}$  enrichment,  $^{14}\text{N}/^{15}\text{N}$  ratio of  $194 \pm 4$  compared to the (normal) ratio acquired for the whole particle of  $272 \pm 2$ . The combination of the NanoSIMS measurements and subsequent examination of the IDP using FESEM identified the location of the anomalous grain (Fig. 1a). To understand the anomalous grain in the context of its own composition and that of the surrounding material, a FIB-TEM section from the area was prepared.

In addition to the ability to mill or ablate material away, the interaction of ions (and indeed electrons) with introduced gases of specific compositions can result in deposition of material onto the surface of the sample (Stevie et al. 2005). In this study, this capability was used to deposit protective layers of Pt using initially the electron beam at 5 kV and then the FIB at 30 kV over the well-defined region of interest (ROI) containing the grain. These protective layers of so-called

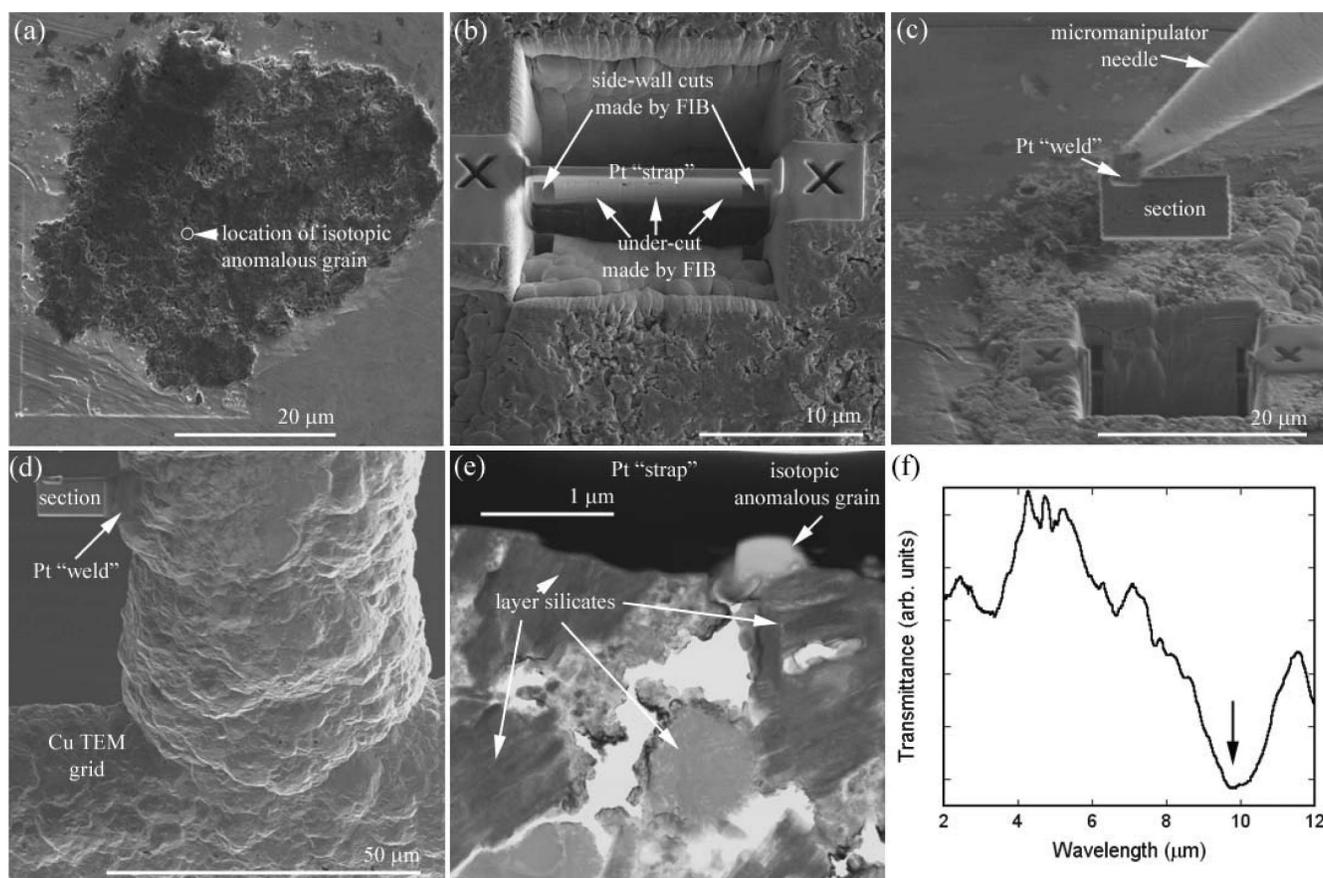


Fig. 1. a) 5 kV secondary electron image of IDP L2047,D23 pressed into a high-purity gold substrate. The white circle denotes the location of an isotopically anomalous grain that was identified during NanoSIMS isotopic imaging. b) 5 kV secondary electron image of the IDP after the FIB has been used to make trenches on either side of the region containing the anomalous grain. The Pt “strap” was deposited prior to the initial FIB trenching to reduce the effects of ion beam damage on the surface. The image also shows the side-wall and undercut FIB cuts made to enable the extraction of the approximately 1  $\mu\text{m}$  thick section. c) 30 kV ion-induced secondary electron image of the in-situ extraction of the section from the IDP. The needle-point is attached to the section using Pt to form a “weld.” d) 30 kV ion-induced secondary electron image of the section attached to a half-cut Cu TEM grid, again the section is attached to the grid using gas-deposited Pt to form a “weld.” e) 30 kV SEM-STEM bright field image of the section after it has been thinned to electron transparency (approximately 100 nm thick). f) The transmission mode infrared spectrum acquired from the section identifying a feature around 9  $\mu\text{m}$  that corresponds to layer silicates (marked by the black arrow).

“strap” assist in reducing ion beam damage to the ROI during FIB milling. In-depth discussion of the FIB milling process in preparation of electron transparent sections from bulk materials can be found in papers by Heaney et al. (2001); Lee et al. (2003), and Anderson and Klepeis (2004). For our work, after deposition of the protective Pt “strap”, a 30 kV focused Ga ion beam (approximately 5000 pA current) was used to mill trenches in the material on either side of the strap, leaving a section  $\sim 1 \mu\text{m}$  thick (Fig. 1b). The beam current was then reduced to 300 pA to make side-wall cuts and an undercut through the thinned section to release it and enable extraction with the in situ micromanipulator needle (Fig. 1c). Once extracted from the bulk sample, the section was moved to a specially constructed semicircular Cu TEM half-grid, also in the chamber. The section was attached to the TEM grid by depositing Pt to form a so-called “weld,” and the FIB was then used to cut the micromanipulator tip away from the

section, leaving the section attached to the grid. The section was then thinned using the 30 kV FIB at 300 pA beam current; once “electron transparency” was approached the current was reduced to 100 pA to minimize surface damage in the finished section. Thinning resulted in a section approximately 80–100 nm thick (Fig. 1d).

As with ultramicrotomy, FIB preparation of an electron transparent section can result in a number of artifacts in the sample; these are discussed in both Lee et al. (2003) and Anderson and Klepeis (2004). For astromaterials, there are several significant issues related to the production of thin sections using FIB. (i)  $\text{Ga}^+$  ions are implanted into the sample during the milling process. This is problematic for detailed X-ray microanalysis by EDS, as the Ga L-lines lie very close to the mineralogically important Mg K-lines. However, modern spectral processing software is often capable of extracting or removing unwanted artifact peaks that are not

indigenous to the sample. (ii) Potential specimen damage during the FIB milling and polishing stages can form an amorphous layer a few nanometers (less than 10 nm) thick. This problem can be mitigated by the use of protective Pt and C “straps” deposited prior to milling, and by performing a low voltage (5 kV) polishing at the end of the sample preparation (Giannuzzi 2006). For the IDP sample preparation, the initial Pt “strap” was deposited using the electron beam rather than the ion beam to further reduce possible ion beam damage to the sample. (iii) There is the potential for loss or damage of volatile material such as organic molecules. However, a number of recent papers studying biological material have included structural studies on samples prepared using FIB and have found little evidence of significant FIB-induced damage of organics (Heymann et al. 2006). (iv) FIB requires specialized skills and expensive instrumentation compared to sample preparation techniques such as ultramicrotomy. (v) Depending on the composition of the sample and the nature of the lift-out, the FIB extraction process can be more time-consuming than other techniques, although it is unlikely that these could offer comparable accuracy in site-specific extraction of samples. (vi) Material surrounding the ROI is consumed during the trenching stages of the FIB milling, making it more difficult to extract serial sections. Solutions to this latter problem are discussed below.

An electron transparent section can be characterized using a number of analytical techniques including TEM, FESEM, and NanoSIMS. In the example discussed here, the section was initially imaged using the 30 kV SEM-STEM detector permanently mounted in the dual-beam microscope chamber. The use of SEM-STEM in astromaterials research is still in its infancy, although recent studies have shown the potential of the technique (Lee and Smith 2006). While the SEM-STEM detector does not have the resolution of a conventional TEM, the images acquired in real time allow a monitoring of specimen thickness during the final stages of thinning, i.e., if the material within the section can be imaged at 30 kV, then the section is sufficiently thin to be imaged in the TEM at 200–300 kV. For example, in the SEM-STEM bright-field image of the IDP section, it was possible to i) see a small grain at the top of the section that could be the isotopically anomalous feature, and ii) texturally infer that the section is dominated by a layer silicate phase (Fig. 1e). The subsequent detailed TEM analysis of the section confirmed the layer silicate phase to be serpentine, based on the  $\sim 0.7$  nm basal lattice fringe spacing acquired using HRTEM. The small grain in the top surface of the section was identified as amorphous carbon. In addition to the detailed TEM study and reexamination using the NanoSIMS, the FIB section can also be used for a number of other analytical techniques, such as synchrotron X-ray fluorescence and IR spectroscopy. In this example, prior to destructive re-examination using NanoSIMS, the section was subjected to infrared spectroscopy to further characterize the amorphous carbon grain (Bradley et al. 2005), and which also identified

the serpentine phase (Fig. 1e). Further analysis of the section in the NanoSIMS confirmed that the amorphous carbon grain did contain the  $^{15}\text{N}$  enrichment (Fig. 2). The implications of the  $^{15}\text{N}$  enrichment are discussed in detail in Smith et al. (2005) and the overall significance and context of this integrated study are discussed in Bradley et al. (2005). This example shows it is possible to derive mineralogical, isotopic, and optical spectroscopic information, the latter enabling direct comparisons with astronomical observations, all from a single FIB-TEM section. The ability to perform integrated studies on the nano-scale phases within individual particles will significantly impact future astromaterials research, for example, with sample return missions such as Stardust where there are only nanograms of material in each sample available to study.

### EXTRACTING COMETARY RESIDUE FROM MICROCRATERS

The hypervelocity capture of Wild-2 cometary dust (at  $\sim 6.1$  km/s) in the low-density silica aerogel and on the additional surface provided by the aluminum 1100 series foil wrapped around the sample tray assembly presented new challenges for sample preparation to enable detailed analytical measurements of these precious particles. From the aerogel, individual impact tracks were extracted from the bulk tiles using microneedles (Westphal et al. 2004) and ultrasonic blades (Ishii et al. 2005; Ishii and Bradley 2006). Once extracted, ultramicrotomy can be used to prepare thin sections suitable for analysis by multiple techniques, e.g., TEM, FTIR, and NanoSIMS (Matrajt and Brownlee 2006). For craters preserved on aluminum, techniques for extraction of residue material remaining from the original impactor were developed during LDEF (Long Duration Exposure Facility) studies by Teetsov and Bradley (1986), who used a combination of microreplication and ultramicrotomy. More recent efforts by Leroux et al. (2006) and Graham et al. (2006) have shown that FIB techniques can be used to extract complete cross sectional slices of craters (less than  $\sim 10$   $\mu\text{m}$  in diameter) or recover individual residue fragments and melt droplets. In this paper we describe the expansion of and modification to the recovery technique discussed in Graham et al. (2006), as applied to real Stardust craters, not laboratory analogues.

The optical and electron microscopy studies of the aluminum foil during the preliminary examination period (January to August 2006) identified a diverse range of craters from several hundreds of micrometers to sub-micrometer in diameter (Hörz et al. 2006). During the Stardust preliminary examination, only a limited number of craters greater than 50  $\mu\text{m}$  in diameter were released for study (Hörz et al. 2006). To maximize the available compositional information from the preserved cometary residues, these craters were analyzed as a part of a multi-technique consortium that included isotopic measurements.

### Extraction of Residue Material from Crater Rims

The preliminary SEM-EDS analysis of the crater from foil C2091N<sub>1</sub> identified abundant residue material (Fig. 3a). However, residue material was deposited on the base, sidewalls, and rim/lip of the crater, across very complex topography that did not allow the use of the classical lift-out methodology discussed in the previous section. Also, it was highly desirable not to consume substantial amounts of the precious Stardust residue during FIB milling to generate a section, or to deposit reworked material elsewhere in the crater. A region of interest (ROI) containing Mg-rich residue was found on the lip of the crater (Fig. 3a). Prior to the FIB milling, both C and Pt “straps” were deposited over the ROI. A C “strap” was deposited first, to physically isolate the residue from overlying Pt, and thereby reduce possible interfering EDS peak overlap between Pt M lines and the K lines from any sulfur that might be in the cometary dust residue. The selection of a ROI located on the crater lip (rather than on the crater wall or base) took advantage of the raised position that yielded a relatively simple geometry for section retrieval, and a free rear surface. In the classical FIB-TEM preparation method, trenches are made on either side of the section to enable side-wall cuts and, more importantly, to permit an oblique angle undercut that releases the section from the bulk material. Although highly variable and sample/material dependant, for a lift out section that is 20  $\mu\text{m}$  in length, the trenched boxes on either side of the section are typically 20  $\mu\text{m}$  in length by 10  $\mu\text{m}$  in width, and are milled to a depth of approximately 7  $\mu\text{m}$ . The FIB trenching step in the preparation sequence typically results in the destruction of a substantial amount of the sample, particularly if the ROI is only a few micrometers in diameter. However, in this Stardust foil example, the position of the raised and overturned crater lip, above an undisturbed foil surface, meant that the section required no undercut to break the section free from the substrate. As a result, it was only necessary to mill initial narrow vertical 30 kV FIB cuts at 1000 pA beam to cut through the entire depth of the overturned crater lip on either side of the ROI (Fig. 3b). This slicing method of sectioning provides an important advantage because it dramatically reduces the area of damage and volume of material lost during the initial preparation stage. However, with no underlying substrate supporting the section, there was a high risk of section collapse and ultimately loss of the ROI during the lift-out stage. This problem was overcome by a novel use of the gas deposition capabilities of the FIB. Carbon was deposited to create a supporting pillar structure between the underlying foil surface and the edge of the section. The micromanipulator needle was then Pt-“welded” to the section, and then the FIB was used to remove the material holding the section to the crater wall (Fig. 3c). Finally, the point of contact between the section and the support pillar was ablated away using the FIB. The section was then successfully moved over to a Cu TEM half-grid within the chamber. Once attached to the grid, the section was further FIB-thinned using a 300 pA beam

current, reduced to 100 pA for the final thinning to an electron transparent thickness of  $\sim 80$  nm (Fig. 3d).

### Serial Sectioning of Residue Material

The initial SEM-EDS study of the crater preserved on foil C2086W<sub>1</sub> identified abundant residue material, including a fragment approximately 20  $\mu\text{m}$  across (Fig. 4a). Isotopic imaging of the fragment using the Washington University NanoSIMS identified a presolar grain (McKeegan et al. 2006) approximately 250 nm in diameter (Fig. 4a). The location of the residue fragment on the interior side-wall of the crater lip meant that it was not possible to prepare a FIB section using the methodology described above for through-cut of an overturned crater lip. For example, the combination of complex sample topography and instrument chamber geometry prohibited deposition of a protective carbon layer over the ROI prior to use of the ion beam. Therefore the initial imaging of the sample was carried out using only SEM secondary electron imaging. Furthermore, great caution had to be taken when using the FIB to image and mill the material surrounding the residue fragment. The sample topography effectively prevented extraction of a limited ROI containing the presolar grain. Instead, the entire fragment was removed from the interior wall of the crater. The FIB was initially used to make a series of trench cuts around the residue fragment at beam currents between 1000 and 7000 pA (Fig. 4b). A Pt “weld” was deposited between the fragment and crater wall to reduce the risk of loss during attachment of the micromanipulator needlepoint. Once the micromanipulator was attached to the fragment, the Pt “weld” was ablated away using the FIB and the fragment was lifted away (Fig. 4c). The fragment was moved over to an aluminum grid mounted onto a standard pin-mount, where it was attached using Pt “welds” (Fig. 4d). This new location allowed access to the ROI. Protective C and Pt layers were then deposited over the ROI containing the presolar grain using FIB. As with the crater lip liftout, the orientation of the fragment now removed the need for broad trenching to accommodate undercuts using the FIB. Therefore, there was no substantial loss of the surrounding material since it was possible to simply make side-wall FIB cuts and then extract the section with the micromanipulator. Rather than extract only the section containing the presolar grain, multiple sections (typically 1.5 to 2  $\mu\text{m}$  thick) in series from the fragment were recovered using this side-wall or so-called “bread slicing” technique (Fig. 4e). The sections were then attached to a Cu TEM half-grid and thinned to electron transparency using the FIB conditions described above (Fig. 4f). Using this novel slicing technique, it was possible to extract seven sections from the region of the fragment containing the presolar grain and its surrounding material.

One of the significant disadvantages of applying ultramicrotomy to astromaterials sample preparation is that the diverse range, size, and properties of the mineralogical

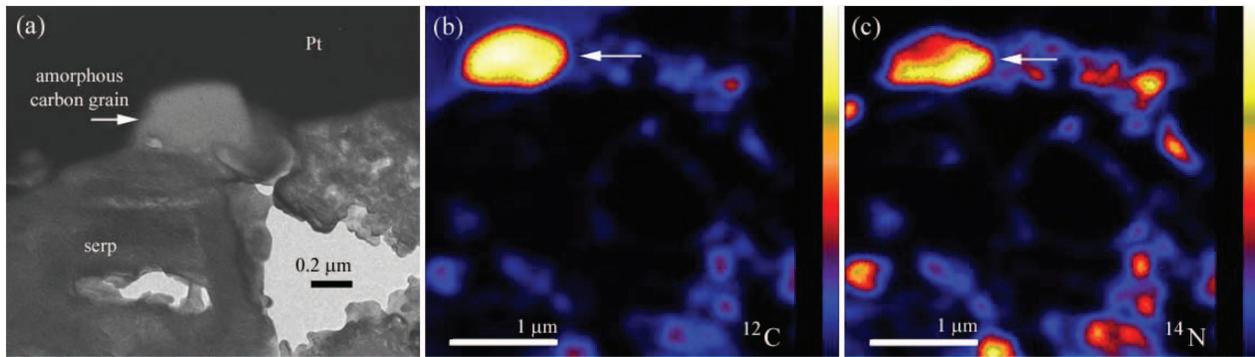


Fig. 2. a) A 300 kV TEM bright field image of the amorphous carbon grain (white arrow) within the FIB section. The C-rich grain is surrounded by serpentine (serp) and was tentatively identified as the location containing the  $^{15}\text{N}$  isotopic enrichment. The grain was confirmed as the carrier of the N isotope anomaly by overlaying the false color NanoSIMS isotope ratio images for  $^{12}\text{C}$  (b) and  $^{14}\text{N}$  (c) with the TEM image (a). The NanoSIMS images have a point to point resolution of  $\sim 50$  nm and a pixel dimension of  $100\text{ nm}^2$ ; the images use an 16-bit color scheme with white-to-yellow representing high intensity and blue-to-black representing low intensity. Additional information concerning the NanoSIMS data is contained in Bradley et al. (2005).

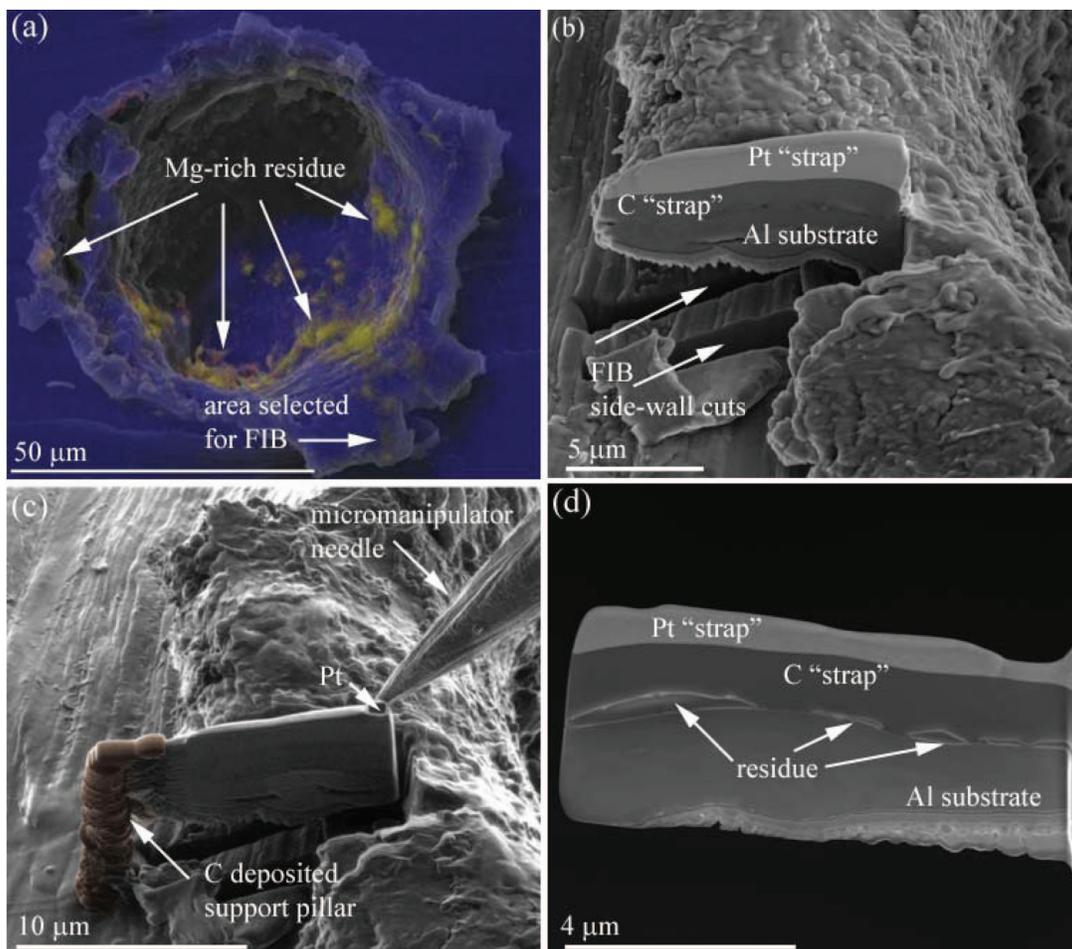


Fig. 3. a) Composite X-ray elemental maps for Al (blue), Mg (red), and Si (green) overlaid on a 20 kV backscattered electron image of an impact crater preserved on Stardust Al foil C2091N,1. The cometary residue is denoted by the yellow patches. The lower right edge of the crater lip in the image was selected as a site for FIB extraction of Mg-rich residue. b) 5 kV secondary electron image of the crater lip containing the region of interest (ROI) after the protective C and Pt "straps" have been deposited. The arrows point to the bottom of the two side-wall cuts where they milled into the underlying foil. The resulting ROI section is approximately  $1\text{ }\mu\text{m}$  thick. c) 5 kV secondary electron image of the section supported by the gas-deposited C support pillar (false-colored brown) prior to extraction by the in situ micromanipulator. d) 5 kV secondary electron image of the section after it has been mounted on the Cu grid and thinned to electron transparency ( $\sim 80$  nm).

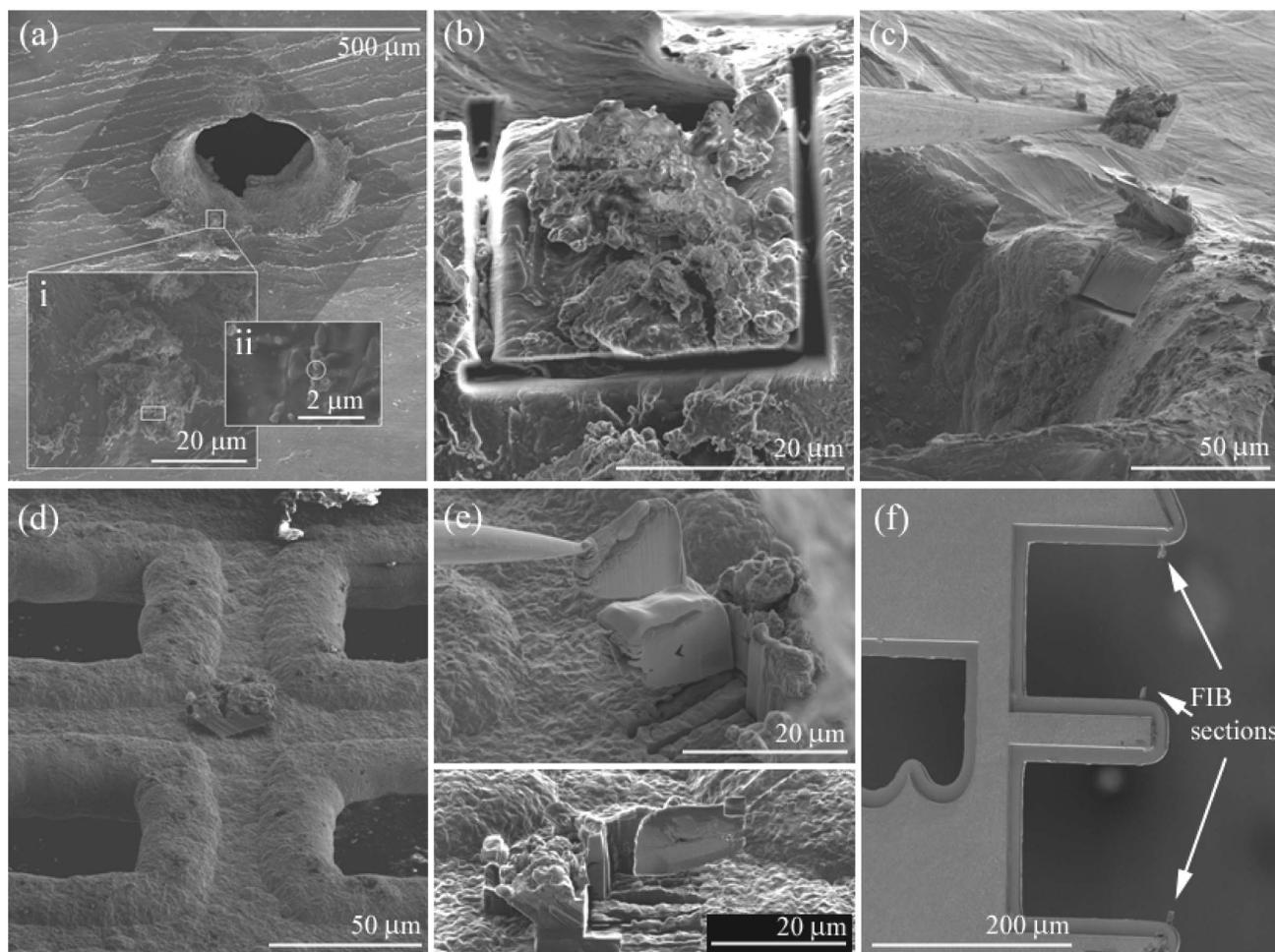


Fig. 4. a) 5 kV secondary electron image of a large micrometer-sized impact penetration preserved on the surface of Stardust Al foil C2086W,1. Insert (i) shows a 5 kV high-magnification secondary electron image of the cometary residue fragment that was analyzed using the NanoSIMS at Washington University, St. Louis (McKeegan et al. 2006; Stadermann et al. 2007). Insert (ii) shows the 5 kV secondary electron image of the location within the fragment that contains an isotopically anomalous grain (denoted by the white circle). b) 5 kV secondary electron image of the residue fragment after the FIB had made the side-walls and undercut to enable the extraction. c) 5 kV secondary electron image of the fragment as it was extracted from the interior wall of the crater using the in-situ micromanipulator. d) 5 kV secondary electron image of the fragment after it was re-located onto an aluminum substrate. e) 5 kV secondary electron image (upper micrograph) and 30 kV ion-induced secondary electron image (lower micrograph) of the extractions of sections 5 and 7 from the fragment using the “bread slicing” or serial sectioning technique. f) 5 kV secondary electron image of the Cu grid with three sections extracted from the bulk residue fragments, mounted and further FIB thinned to electron transparency.

phases within a sample can often result in “plucking” or dropout of material during the serial slicing, as well as structural deformation within the section. This loss of material can negatively impact the overall mineralogical interpretation of the sample under the TEM. With the FIB serial section technique, the problems are greatly reduced as the sections have a greater structural integrity. However, unlike ultramicrotomy, material between each FIB slice is ablated away, resulting in a potential loss of mineralogical continuity when analyzing each section. Despite this disadvantage, serial sectioning using the FIB is a particularly important evolution of the technique that combines site-specific recovery with the benefits (traditionally provided by ultramicrotomy) of serial sections from a single small sample.

## SUMMARY

FIB microscopy provides a means to perform integrated studies on individual IDPs and Stardust cometary debris, helping us to explore the relationships between the isotopic, mineralogical and elemental properties at the nanoscale. Such studies are providing new insights into early solar system processes and the interstellar environment (e.g., Floss et al. 2004; Stroud et al. 2004; Bradley et al. 2005; Floss et al. 2006; Stroud and Zega 2006).

As a routine sample preparation technique for meteoritic materials, the classic FIB-TEM liftout has only had limited use outside of these few integrated studies (e.g., Heaney et al. 2001; Lee et al. 2003). This is, in part, due to the reputation of

FIB preparation as destructive because the trenching process requires substantial loss of material from the sample. Furthermore, the number of sections that have been harvested from small sample volumes has typically been limited to one or two, comparing most unfavorably with the multiple sections that can be produced by ultramicrotomy. However, the novel “bread-slicing” FIB technique described in this paper for generating serial slices highlights a new capability for extracting multiple sections from a residue fragment 20  $\mu\text{m}$  in diameter. This technique is now being applied to IDPs as a method of serial sectioning like ultramicrotomy but with the advantage of less loss of petrographic information due to grain pluck-out and shattering. FIB microscopy is a mature, yet still evolving, sample preparation technique that is meeting the needs of the next generation of analytical instruments (e.g., the NanoSIMS and SuperSTEM) and samples (e.g., Stardust).

*Acknowledgments*—This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory in part under contract no. W-7405-Eng-48 and in part under contract no. DE-AC52-07NA27344. The Advanced Light Source is supported by the Director, Office of Science, Office of Basic Energy Sciences, Materials Sciences Division, of the U.S. Department of Energy under contract no. DE-AC03-76F00098 at Lawrence Berkeley National Laboratory. This work was supported by NASA’s cosmochemistry program (grant nos. NAG5-10696 and NNH04AB471), SRLIDAP (grant no. NNH04AB491), and Stardust Participating Guest program (grant no. NNH06AD671). Sheri Kurland and Trisha Rice are thanked for extensive access to FEI Company’s FIB laboratory at the Hillsboro demonstration facility for the sample preparation work on the IDP sample. We would also like to thank the associate editor Don Brownlee and the reviewer Caroline Smith for their useful comments, which have enhanced this manuscript.

*Editorial Handling*—Dr. Donald Brownlee

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