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Focused ion beam recovery of hypervelocity impact residue in experimental craters on metallic foils

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Abstract–The Stardust sample return capsule returned to Earth in January 2006 with primitive debris collected from comet 81P/Wild-2 during the flyby encounter in 2004. In addition to the cometary particles embedded in low-density silica aerogel, there are microcraters preserved in the aluminum foils (1100 series; 100 µm thick) that are wrapped around the sample tray assembly. Soda lime spheres (~49 µm in diameter) have been accelerated with a light gas gun into flight-grade aluminum foils at 6.35 km s^{-1} to simulate the capture of cometary debris. The experimental craters have been analyzed using scanning electron microscopy (SEM) and X-ray energy dispersive spectroscopy (EDX) to locate and characterize remants of the projectile material remaining within the craters. In addition, ion beam–induced secondary electron imaging has proven particularly useful in identifying areas within the craters that contain residue material. Finally, high-precision focused ion beam (FIB) milling has been used to isolate and then extract an individual melt residue droplet from the interior wall of an impact. This has enabled further detailed elemental characterization that is free from the background contamination of the aluminum foil substrate. The ability to recover "pure" melt residues using FIB will significantly extend the interpretations of the residue chemistry preserved in the aluminum foils returned by Stardust.

INTRODUCTION

The study of comets is fundamental to understanding early solar system processes (e.g., Brownlee 2003; Hanner 2003). To date, much of the knowledge of the composition of specific comets is from remote or in situ analysis (e.g., Kissel et al. 1986; Kissel et al. 2004). Yet the most definitive characterization can only really be achieved by using the diverse range of analytical instruments that are currently available in the laboratory (Zolensky et al. 2000). Some interplanetary dust particles (IDPs) have already been linked to cometary sources based on their mineralogical and optical spectroscopy properties (Bradley and Brownlee 1986; Bradley et al. 1999). However, it has not proven possible to define a specific parent body source.

In January 2004, the successful flyby of NASA's Stardust spacecraft with comet 81P/Wild-2 resulted in the capture of abundant cometary debris (Brownlee et al. 2004; Tuzzolino et al. 2004). In addition to the primary mission goal of the comet flyby, the reverse side of the sample tray assembly (STA) was exposed to an interstellar dust stream during parts of the outbound cruise phase (Brownlee et al. 2003). The cometary and interstellar dust particles were primarily captured in low-density, highly porous silica aerogel tiles (Tsou et al. 2003). A number of papers have dealt with the technique issues of material recovery from deep penetration tracks in aerogel generated by laboratory simulations or by low-Earth orbit (LEO) space exposure in order to prepare for Stardust's return (e.g., Graham et al. 2004; Westphal et al. 2004; Ishii et al. 2005a, 2005b).

The STA that holds the individual aerogel tiles is wrapped with aluminum foils 100 µm thick (1100 series). The space-exposed surfaces of these foils will also retain a record of the hypervelocity encounters with both interstellar and cometary particle populations. Previous studies of metallic surfaces exposed in space, e.g., those from the Long Duration Exposure Facility (LDEF), showed evidence of micrometer-size craters as a result of meteoroid or orbital debris collisions (e.g., Bernhard et al. 1993). Using analyses of impact residue chemistry preserved within the craters, it was possible to derive the original impactor composition (e.g., Bernhard et al. 1993; Brownlee et al. 1993). In addition to SEM/EDX studies, novel replication and residue recovery techniques enabled detailed TEM studies of the meteoroid debris (Teetsov and Bradley 1986; Bradley et al. 1986; Brownlee et al. 1993). These techniques will be employed on the Stardust foil samples. However, it is important to explore the new analytical capabilities that are now available for careful selection, preparation, and manipulation of specifically located micrometer-size material. Here we report on the use of focused ion beam microscopy to extract residue material from an impact preserved in aluminum foils to simulate potential Stardust recovery.

ANALYTICAL METHODS

Light Gas Gun Simulations

A number of metallic foils that have previously been exposed in low-Earth-orbit (LEO) as part of either dedicated experiments (such as those on LDEF) or as a target-of-opportunity (e.g., on the Solar Maximum satellite) could have been used to develop and test capabilities for recovery and analysis of impacted material (e.g., Bradley et al. 1986; Bernhard et al. 1993). As meteoroid impacts on space-exposed surfaces are likely to have occurred at velocities between 10–20 km s^{-1} (e.g., Brownlee et al. 1993), they are not a representative analogue for the Stardust encounter velocity of ~6 km s^{-1}. As a result, a comprehensive shot program was set up to provide analogous materials for laboratory investigation to support the interpretation of Stardust samples.

The laboratory simulation experiments described in this paper were performed using the small caliber (5 mm bore) two-stage light gas gun (LGG) at the Johnson Space Center (JSC) in Houston, Texas. Glass spheres of known size range (Kearsley et al. 2006) and meteoritic materials (e.g., crushed Allende) were used as projectiles for calibration studies. Rather than accelerate individual particles, a "shotgun" approach is utilized by loading multiple projectiles into the small central cavity of a four-piece serrated sabot. The four sabot quadrants were designed to separate radially during free flight, yet allow a substantial fraction of the projectile ensemble to remain on straight trajectories and to ultimately reach the target site.

The LGG at JSC is fitted with a number of flapper valves, mechanical apertures, and a sabot catcher system that minimize the contamination so that only those projectiles that reside within <1 degree of the gun axis will make it on target. For these experiments, the target material used was Stardust flight-grade aluminum foil ~100 µm thick (1100 series) that was supplied to JSC by Peter Tsou (NASA/JPL). For each of the shots, the foils were wrapped around a 25 × 25 × 3.12 mm aluminum (6061, T6 series) plate, the latter simulating the Stardust collector frame.

The impact penetrations and residue material that are discussed in this paper are from JSC shot #2382, a shot that accelerated soda lime glass spheres (43–54 µm in diameter) into the aluminum foil target at 6.35 km s^{-1}. The velocity was measured using laser occultation methods and IR photo diodes for determination of the sabot pieces. Additionally, the velocity of the projectiles impacting the foil was measured using an impact flash detector. Typically sabot velocity and projectile velocity agree to better than 1%.

Imaging and Microanalysis

The foil target from JSC shot #2382 was initially imaged using a Leica MZ16 stereomicroscope fitted with a Leica DC500 12 mega-pixel CCD camera. The entire foil (25 × 25 mm) was attached to a large diameter pin-stub using conductive carbon paint. It was then imaged, analyzed and subjected to precision ion milling using an FEI Nova 600 dual beam microscope comprising of a Ga+ liquid metal source focused ion beam (FIB) and field emission gun scanning electron microscope (FESEM). The dual beam microscope was fitted with an EDAX Genesis energy dispersive X-ray (EDX) spectrometer and an Omniprobe tungsten needle nanomanipulator. The secondary electron imaging was performed at 5 kV with a beam current of 0.15 nA and the EDX single-point spot analysis and mapping were performed at 15–20 kV with a beam current of 0.26 nA. The FIB imaging and milling was carried out at 30 kV with a beam current ranging from 30–1000 pA. Imaging and elemental analysis of extracted residue were performed using 200 kV FEI Tecnai G2 F20 UT (scanning) transmission electron microscope (TEM) fitted with an EDAX EDX spectrometer and FEI TIA spectral processing software.

RESULTS

SEM/EDX Imaging and Analysis

From the secondary electron imaging of the foil target, we determined that the crater diameter ranged from 214 µm to 223 µm (Fig. 1a). The impact craters studied had completely penetrated the 100 µm 1100 series foil and terminated in the 6061 aluminum plate. As a result, the observed impacts have steep sidewalls and flat bottom morphologies (Figs. 1a and 2a). The reduction in the cratering efficiency as a result of the shock reverberation of the foil and plate leads to lower crater diameters than the predicted value (~236 µm crater diameter) from the calibration plot by Kearsley et al. (2006). The impact residue morphologies observed within the craters varied from thin films to vesicular glass. They are typical for those generated by Si-rich materials and are similar to those observed in LDEF craters generated by silicate-dominated meteoroids (e.g., Bernhard et al. 1993; Brownlee et al. 1993).

In addition to acquiring crater diameters to assist in the confirmation of the original particle flux estimations of the

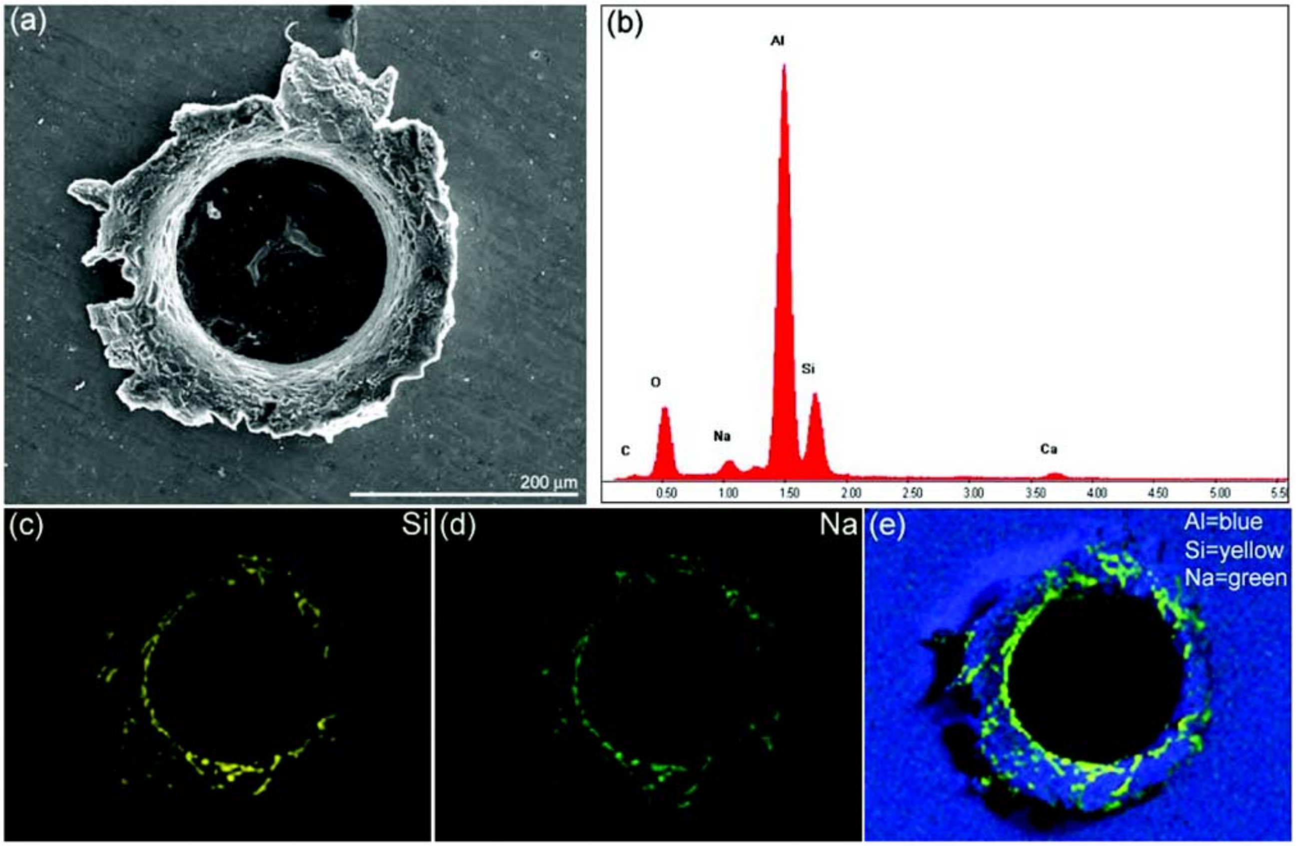

Fig. 1. a) A secondary electron image of an impact crater generated by a nominal 49 µm projectile at 6.35 km s^{-1} that has completely penetrated the 100-µm-thick foil. b) A typical X-ray energy-dispersive spectrum (EDX) acquired for the residue material preserved on the rim of the crater. There is significant contribution of aluminum foil substrate detected in the spectrum. c) An EDX map for Si locating the distribution of the projectile residue on the crater lip. d) An EDX map for Na corresponding with the Si map. e) An overlay composite map for aluminum (substrate) against Na and Si (soda lime glass residue).

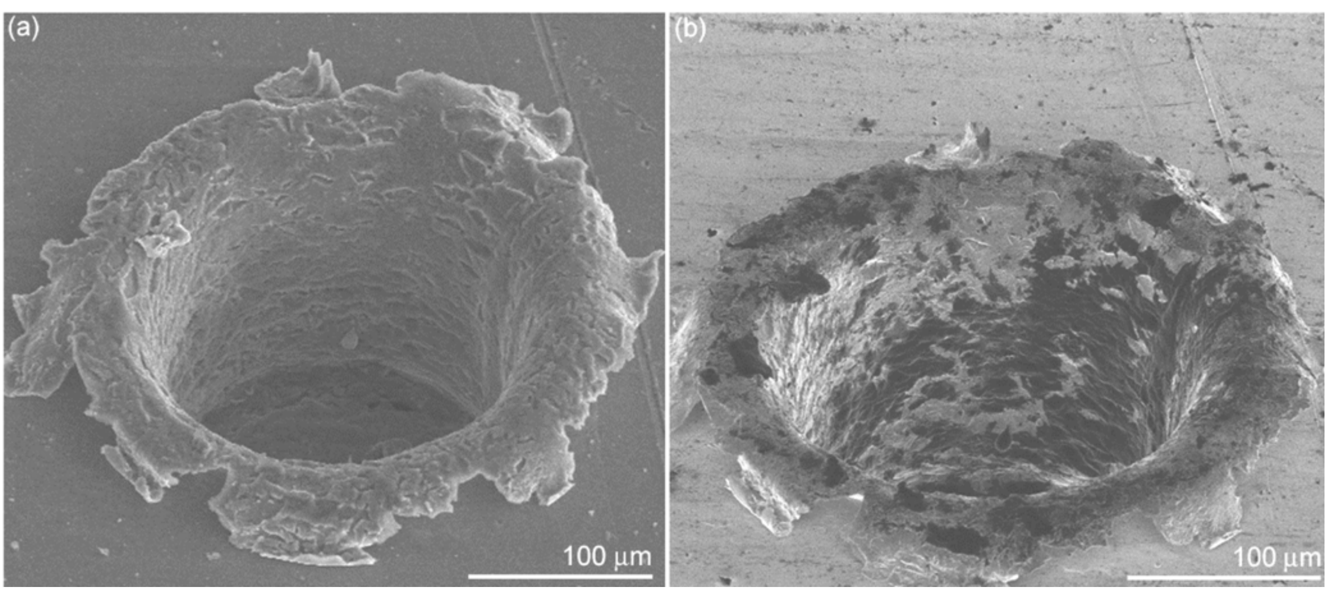

Fig. 2. a) A secondary electron image of a second experimental crater. b) An ion-induced secondary electron image of the same feature. Extraneous residue material is clearly identified on the walls and rim of the crater due to enhanced material contrast.

Stardust encounter (Tuzzolino et al. 2004), the craters will also contain remnants of the comet Wild-2 debris. A particularly useful technique for identifying residue material within craters is EDX analysis using either a single spot mode (e.g., Bernhard et al. 1993) or an elemental mapping mode (e.g., Graham et al. 2000). Both of these approaches were used to analyze the residue material generated by the soda-lime projectiles (Figs. 1b–e). As the impacts have penetrated into the 6061 aluminum plate, the melt residue composition may be a complex mixture of the foil and plate substrates as well as the remnants of the soda lime projectiles.

FIB Imaging

The traditional method for surveying and subsequent identification of impact craters on metallic surfaces is SEM imaging using secondary electron and backscattered electron image modes. Backscattered electron imaging (BEI) has proven particularly useful where there is substantial compositional contrast between a projectile residue and the impacted substrate, such as sulfide residues on borosilicate glass (e.g., Kearsley et al. 2005). It is, however, less effective when there is little inherent contrast, such as between silicate impactor residue and solar cell glass. In craters on aluminum foils, the compositional contrast in BEI might be expected to reveal residue easily. Unfortunately, the complex fine-scale crater topography masks much of the desired contrast.

Ion-induced secondary electron images can be acquired using the FIB (Phaneuf 2004). Potentially, there is an increase in the material contrast that can be observed in FIB-secondary electron images compared to conventional SEM secondary and backscattered electron images. Figure 2 shows a conventional secondary electron image and an FIB-secondary electron image with enhanced material contrast observed between the impact residue and the substrate in the FIB image. Unlike conventional secondary electron imaging, FIB imaging is a destructive technique as the interaction between the Ga+ ions and the substrate will result in the removal of material and the implantation of Ga. However at the low beam current (30 pA) used in this study, the loss of material from the FIB imaging was negligible. Ga implantation may interfere with EDX analysis of Na (there are major overlaps between the relatively broad peaks of Ga-L and Na-K X-ray lines), but is unlikely to compromise other methods of analysis.

Residue Extraction Using FIB

FIB microscopy has now become a well-established technique in materials science, especially for preparing site-specific electron transparent sections from bulk materials (e.g., Phaneuf 2004). For detailed elemental and isotopic studies of the cometary impact residue deposited in aluminum foil craters, it is important that the material can be recovered. Depending on the size of the craters and the distribution of the residue within the craters, there are two approaches that can be used with FIB. For small craters, typically 10–15 μm in diameter, it is possible to prepare complete TEM cross-sections of the entire crater that contain both the residue and the substrate (see Leroux et al. [2006] for an in-depth discussion of this methodology). Complete cross-sections work extremely well when the residue is deposited as a film over most of the interior surface of the crater. However, as was shown in LDEF studies, the deposition of residue material within craters was highly varied. The residue ranged from thin-films, to more massive melt-liners and isolated melt beads/droplets, and may even include unmelted fragments of projectile material (Brownlee et al. 1993). Therefore, the second application of FIB is to recover isolated residue material from within a crater. Figures 3a and 3b show an impact that contains a micrometer-size droplet (approximately 7 μm × 11.5 μm) in addition to the typical thin film of melt residue. Normally, a protective layer of Pt 2–3 μm thick is deposited on the top surface of the material that is going to be subjected to ion milling, as the initial process can result in ion beam damage up to a depth of 10 nm within the surface of interest. As the melt droplet in Fig. 2 was the product of extreme alteration to the original projectile material (during the hypervelocity capture), it was considered that protection by deposition of Pt was unnecessary. The FIB was initially used to remove material from the interface between the droplet and the wall of the foil (Fig. 3c) with a beam current of 1000 pA and 30 kV accelerating voltage. To ensure that the droplet did not fall into the crater pit, a small Pt “strap” was deposited onto the droplet and continued to the crater wall before the droplet was released from the crater. The tip of the Omniprobe tungsten needle was attached to the outer surface of the droplet using Pt, after which the FIB was used to remove the remaining interface material and the Pt “strap,” at a reduced 300 pA beam current at 30 kV. This enabled the bulk of the droplet to be extracted from the wall of the crater (Fig. 3d). Within the chamber of the dual beam microscope, the Omniprobe tungsten needle was moved over to the TEM grid holder and the droplet was attached to the arm of one of the copper grids using Pt deposition. The needle-droplet interface was removed using the FIB, leaving the droplet attached to the TEM grid (Fig. 3e). The droplet was then thinned to electron transparency (~100 nm thick) using the 30 kV FIB at 300 and then 100pA beam current (Fig. 3f).

DISCUSSION

The hypervelocity capture of cosmic dust particles results in varying degrees of alteration. Meteoritic silicate melt glasses were frequently observed lining the walls of the LDEF craters (Bernhard et al. 1993; Brownlee et al. 1993). Therefore, it might be argued that alteration of the original crystallographic structure during hypervelocity capture severely limits the use of cometary impact residues in

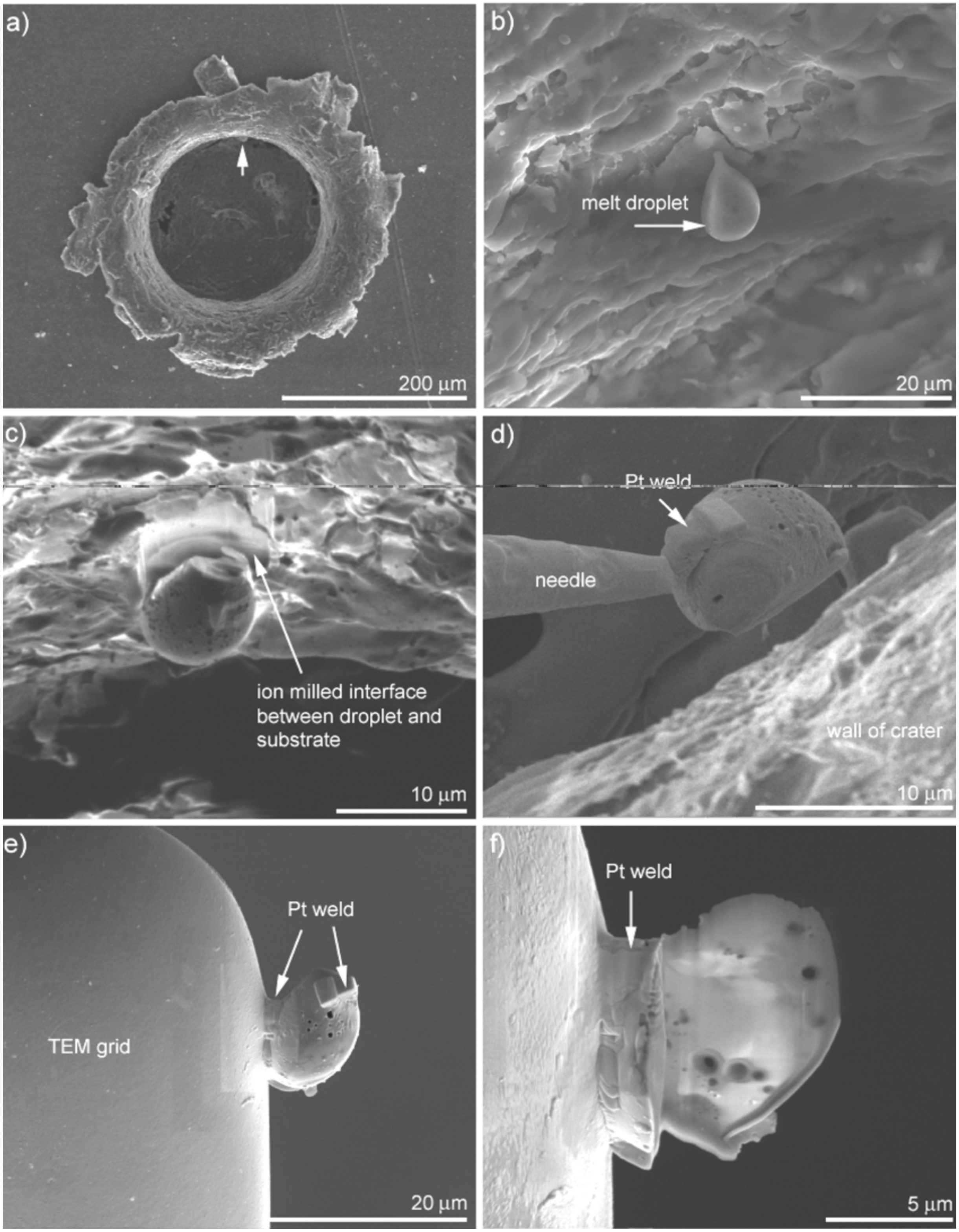

Fig. 3. Secondary electron images showing the extraction and subsequent thinning of a residue droplet from the interior wall of an impact. a) The impact containing the melt droplet (see the white arrow marker). b) The melt droplet prior to ion milling. c) High-precision FIB milling was then used to remove the bulk of the material attaching the droplet to the interior wall. d) The in situ extraction of the droplet from the crater wall using the Omniprobe tungsten needle nanomanipulator. e) The droplet welded to the copper TEM grid prior to ion thinning, using the FIB, to electron transparency thickness. f) The droplet after final thinning.

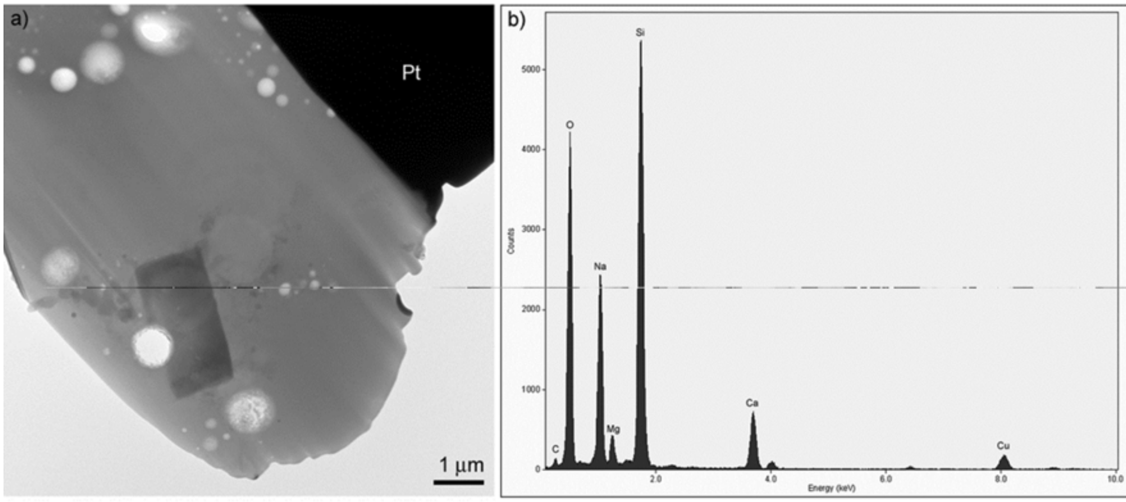

Fig. 4. a) A bright-field TEM image of amorphous glass melt droplet. b) The EDX spectrum acquired from the core of the droplet. Note that, after removing the droplet from the crater wall, the significant Al peak observed in Fig. 1b is nearly absent. There is also no evidence of Ga peaks in the spectrum that might have been implanted during the FIB milling. The only extraneous elemental peak observed in the spectrum is Cu from the TEM grid.

understanding the mineralogical composition of the comet. However, it is noteworthy that, in addition to the melt glasses, some LDEF craters contained well-preserved mineral grains, and some even contained solar flare tracks (Brownlee et al. 1993). We conclude that it is important to demonstrate a capability to recover cometary material from the craters. For LDEF craters and previous LEO retrieved materials (e.g., the thermal blanket from the Solar Maximum satellite), the impact residues were recovered from the substrates using micro-replication and ultramicrotomy techniques (Teetsov and Bradley 1986; Bradley et al. 1986). Although these techniques were successful in the recovery of meteoroid material (e.g., Brownlee et al. 1993), their methodology requires high skill levels, is time-consuming, and can result in the loss of material. FIB methodology requires equal skill and is also time-consuming, depending on the size of the structure to be ion milled. The significant advantage of the FIB methodology is the ability for controlled site-specific recovery of residue material from a crater. Furthermore, the microtomed sections prepared from LDEF craters contain both residue and the substrate material. The presence of the substrate constitutes background elemental contamination and it is highly desirable to limit or remove it from any subsequent elemental analyses. The TEM/EDX analysis of the FIB-prepared section showed that the droplet was essentially “substrate-free” with Cu from the TEM grid as the only extraneous peak observed in the spectrum (Fig. 4). In addition, the Na peak observed in the EDX spectrum would suggest that there was limited loss of volatiles during hypervelocity capture.

Previous studies of residue chemistry preserved in craters have involved mapping techniques such as EDX or SIMS (e.g., Bunch et al. 1991; Bernhard et al. 1993; Graham et al. 2000). However, unless the impact features are particularly shallow in depth, there will be a significant issue with regards to the exposure of the interior surface of the crater to the instrument detector due to geometry. The effect of this is an incomplete line-of-sight of emitted X-rays or ions to the detector, typically resulting in only the rims of the crater showing the location of residue material (e.g., Fig. 1 and Stephan et al. 2005). In addition, instruments such as the NanoSIMS have very specific geometric requirements for sample preparation with specimen height and topography being critical factors. The ability to prepare electron transparent sections of either an individual melt residue as discussed herein or entire cross-sections of microcraters (Leroux et al. 2005) maximizes the potential of coordinated studies. It has previously been shown from recent integrated studies of IDPs that a single FIB section can be investigated using multiple techniques to gain mineralogical, chemical and isotopic information, and the same approach will be applied to Stardust samples (Floss et al. 2004; Bradley et al. 2005).

CONCLUSION

Cometary material from a known source is a significant addition to the current repository of extraterrestrial materials available for laboratory studies. However, the ability to interpret the nature of the materials will depend on the level of micro-analytical characterization that can be performed. Whether it is particles embedded in aerogel or residue fused to the walls of microcraters, the captured cometary debris must be liberated from the collection substrate. While FIB microscopy is not the only method available to recover material, it is the only one that can be demonstrated to work at the spatial resolution suitable for material generated by hypervelocity particle collisions in nonporous targets.

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