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FIB/STEM Investigation of Four Impact Craters from the Stardust Comet Sample Return Mission Foils

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NASA's Stardust spacecraft flew through the coma of comet 81P/Wild 2 at 6.1 km/s and successfully returned to Earth with the first unambiguous cometary material. However, the collected cometary material experienced extensive alteration due to the high collection velocity [1]. Previous investigations of the Stardust collector foils have succeeded in returning surviving crystalline material [2,3], but further investigations are required to better characterize the comet's fine component.

We imaged two Stardust foils (C2113N-B and C2118N-B) at 1k to 2.5k magnification with a Mira Tescan Scanning Electron Microscope (SEM), locating two craters on each foil with diameters between 1.5 and 4.8 μm. The crater residues were elementally characterized with energy dispersive x-ray spectroscopy (EDS) using Cliff-Lorimer analysis. Cross sections of these craters were extracted and thinned to 100-150 nm with a FEI Quanta 3D Focused Ion Beam (FIB) operated at 30kV, maximizing the preservation of cometary material for potential future isotope analyses. A final polish was performed with a FEI Helios operated at 8 kV. We obtained high-resolution images of the crater cross sections using a Nion UltraSTEM 200 aberration-corrected scanning transmission electron microscope (STEM), and STEM-EDS maps with the JEOL 2200 FS STEM, equipped with an Oxford Aztec SDD-EDS, at the Naval Research Laboratory.

SEM images of the craters showed that three of the craters contained a single, rounded crater bottom suggestive of a compact impactor, whereas one crater contained a double indentation indicative of a complex aggregate grain of varying density. SEM-EDS analysis indicated that all four craters resulted from aggregate impactors that were dominated by Mg- and Si-rich materials coupled with iron sulfides. STEM imaging revealed narrow (10-100 nm) bands of residue blanketing the crater floors. Z-contrast STEM images, coupled with STEM-EDS mapping, showed heterogeneous melts of Si-, Mg-, and Fe-rich impactor material. EDS analysis of the residues showed Mg/Si ratios in the craters ranged from 1.42 $\pm$ 0.13 to 0.86 $\pm$ 0.07, and Fe/S ratios in the craters ranged from 1.75 $\pm$ 0.44 to 1.14 $\pm$ 0.15 (Fig. 1). Assuming a Fe/S ratio of 1 for iron sulfide, O/(Mg+Si+Fe) ratios in the residues ranged from 1.21 $\pm$ 0.25 to 2.13 $\pm$ 0.40, and (Mg + Fe)/Si ratios ranged from 1.01 $\pm$ 0.10 to 1.70 $\pm$ 0.18. Trace Ni was visible in three of the craters while trace Ca was only visible in a single crater.

Previous investigations of Stardust analog impactors have shown that S-loss is common in high-velocity impacts [4,5], suggesting that much of the observed Fe originates from iron sulfides rather than from silicate impactors. Additionally, previous analog studies have also shown post-impact abundances of Mg, Si, and Fe to be minimally altered, with only small losses of O relative to these elements [4,6]. The elemental compositions of the impactors are consistent with Mg-rich pyroxene and olivine combined with troilite or pyrrhotite. High O abundances relative to Fe, Si, and Mg are likely due to presence of aluminum oxides from the residue substrates. Similar residue compositions have been observed in previous Stardust crater analyses [2, 3], though our study differs from previous results in that no clear surviving crystalline grains were observed. While many of the crater residues appear partially melted, the complete lack of distinguishable grains may be due to the relatively small size of the grains (~10-30 nm) relative to the thickness of the FIB sections, preventing reliable identification with STEM-EDS mapping.

Our crater residue compositions are consistent with glass embedded with metal and sulfides (GEMS) typically found in interplanetary dust particles [7]. Determining whether or not the impactors were amorphous prior to collection is difficult given the violent nature of the collection process. Recent studies of Stardust aerogel samples have returned amorphous fine grained material consistent compositionally and morphologically with GEMS [8]. Our prior investigation of 11 craters from a single heavily cratered foil similarly returned a lack of crystalline material and compositions consistent with GEMS [9], suggesting that amorphous material may constitute a significant fraction of the fine component in comet Wild 2.

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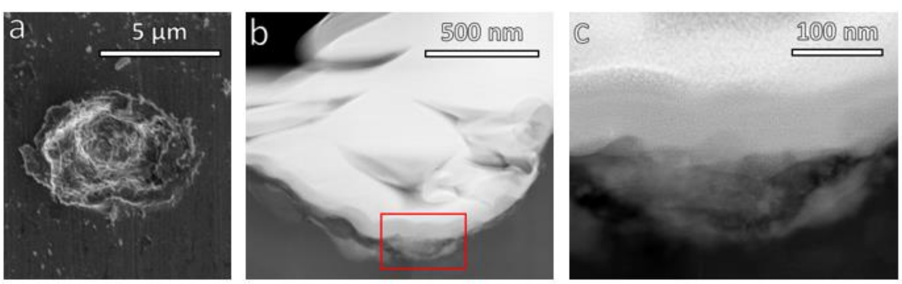

Crater	O at. %	Mg at. %	Si at. %	Fe at. %	S at. %
2113N-B #1	56.2	13.9	16.1	8.1	5.7
2113N-B #2	62.6	15.5	10.9	7.0	4.0
2118N-B #1	59.5	14.8	13.2	6.5	5.0
2118N-B #2	40.6	17.6	14.1	14.8	12.9

Figure 1. a) SEM image of crater 2 from foil C2118N-B. b) High-angle annular dark-field (HAADF) image of the same crater collected by the Nion UltraSTEM 200. The protective Pt cap deposited during FIB sample preparation appears brighter than the Al below. c) Magnified image (area of rectangle in image b) of the cometary residue. The contrast in the image is due to the combination of Mg-, Si-, and Fe-rich impacting material. The final table shows the O, Mg, Si, Fe and S atomic abundances in each crater. Crater 2118N-B #2 also contained 1.0 at. % Ca.

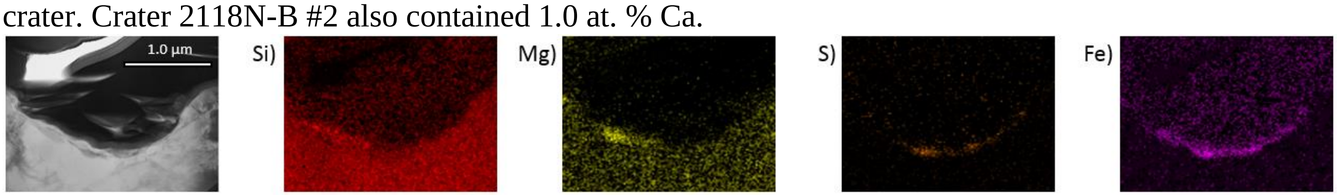

Figure 2. Bright field STEM image of crater 2 from foil C2118N-B, along with EDS maps of Si, Mg, S, and Fe from the same region.