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Chemical, mineralogical, and isotopic characterization of terminal particles from Stardust tracks

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Summary

We report on the past year of our consortium study of Stardust cometary tracks, with the goal of exploring the chemical, mineralogical and isotopic diversity of materials returned from comet 81P/Wild 2.

Track #	Length	Track Type
200	390 µm	A
201	1800 µm	A
202	5000 µm	B
203	5200 µm	B
204	800 µm	C
205	220 µm	C
206	200 µm	A

Table 1: Information on tracks extracted from Stardust Tile 31

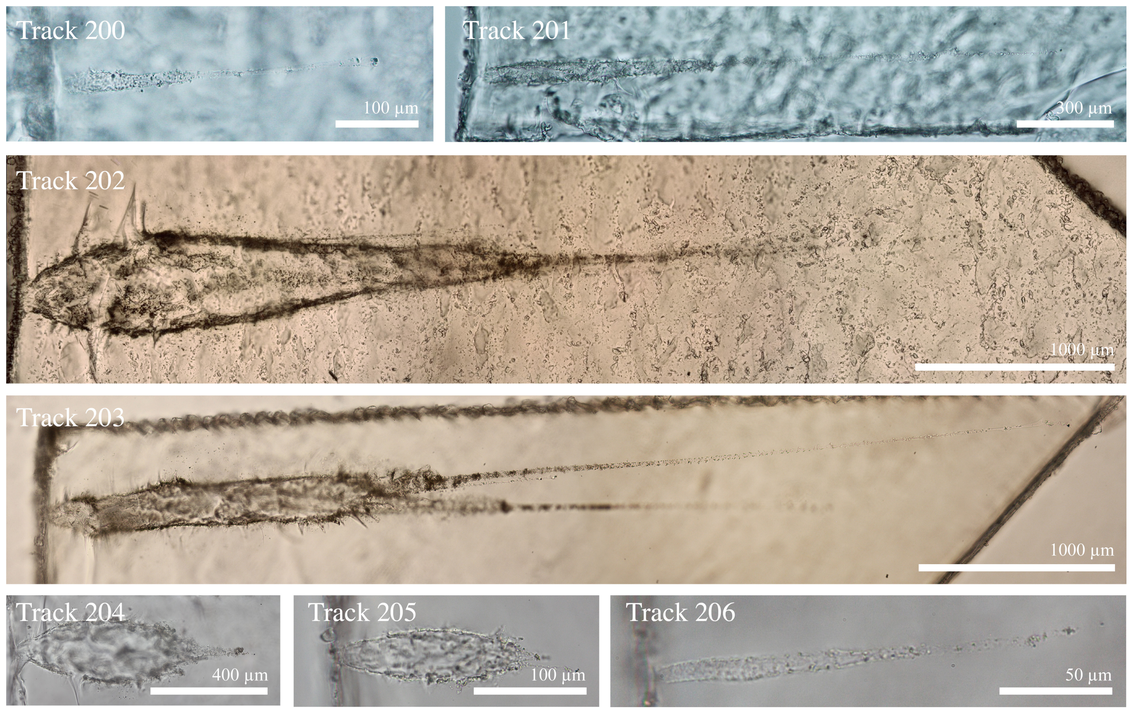

Figure 1: Optical photomicrographs of Stardust tracks extracted from Tile 31. Tracks 200, 201, 204, 205, and 206 are reflected light images, while track 202 and 203 images are transmitted light.

Methods

We studied seven tracks extracted from aerogel tile C2031 (Table 1; Figure 1), using the following work-flow and techniques:

Keystone Extraction
UCB Space Sciences Lab
Tracks were extracted from aerogel using the methods described in [1].

Synchrotron X-ray Microprobe
Advanced Light Source, LBNL
We measured the elemental composition of particles using fluorescence mapping at 10 keV and a 6 x 6 µm beamspot. We used spot X-ray Absorption Near-Edge Structure (XANES) analyses [4] and chemical mapping [5] at the Fe edge to identify silicates, sulfides, and metals.

Keystone Microsurgery
UCB Space Sciences Lab
We separated the terminal particles (TP) and fine-grained bulb material by laying the keystone on its side in a 4-µm-thick ultralene "surgical tent" with an access slit. We then sliced it into 100–300 µm thick wafers using the keystoning system.

Fine-grained Material

High-Pressure Sapphire Press
UCB Space Sciences Lab
In preparation for measurements of oxygen isotopes in fine-grained material captured in the track bulb [2], we used a newly developed high-pressure sapphire press. This press uses a C-coated sapphire window to press dissected bulb halves into indium in the center of a polished Al round. The round includes polished standards surrounding the indium, in a configuration identical to our standard "buckler" mount [2,3].

Terminal Particles

Embedding and Ultramicrotomy
Molecular Foundry, LBNL
Terminal particles were impregnated with epoxy on the end of epoxy bullets. After curing, we used the ultramicrotome to remove ~100-nm-thick slices, which were placed on TEM grids.

Transmission Electron Microscopy
Molecular Foundry, LBNL
TEMs used were the Philips CM200/FEG set to 200 keV with an Oxford EDS detector, as well as the FEI Titan TEM with beam voltages between 80-200 keV, and a 0.6 sr EDS detector. Quantitative EDS analyses were made for individual phases in track 202 TP5, TP6, and TP9, with a map time of ~11–30 min per phase.

Isotopic Analysis
HIGP, University of Hawai'i and Washington University
Fine-grained materials were measured in imaging mode using either the Cameca ims 1280 ion probe at UH or the Cameca NanoSIMS at Washington University. For terminal particles, the potted butts remaining after microtoming were mounted in our specially designed "buckler" mounts [2,3]. O isotopes were measured using the Cameca ims 1280 ion probe in multicollection mode. A ~2 µm, ~20 pA Cs^+ primary ion beam was used to measure individual phases within the TPs [e.g., 2].

Results

Track 200 (C2031,1,200)
Microscopy and synchrotron analyses indicated a ~2–3 µm TP of mixed silicate and sulfide. TEM confirmed that the TP consists of two silicate phases surrounded by a rim of Fe-sulfide. EDS analyses were not quantitative as they were enriched in SiO_2 from aerogel contamination, and small grain sizes led to mixed analyses.

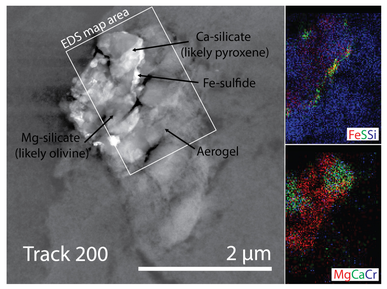

Figure 2: TEM and EDS analyses of the Track 200 TP.

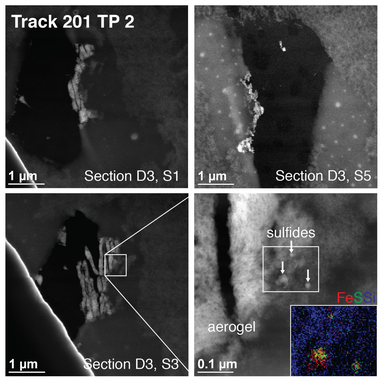

Figure 3: Microtomed sections of Track 201 TP2 showing aerogel and sulfide.

Track 201 (C2031,2,201)
The terminal particle at the end of the track was ~2 x 4 µm. Synchrotron analysis showed Fe-bearing material dominated by sulfide. The TEM sections were mostly plucked (Fig. 3), and residual material was SiO_2 with some embedded sulfides. Ion probe confirmed that the SiO_2 was melted and quenched aerogel. O-isotope analysis of the bulb's fine-grained material is pending.

Track 202 (C2031,3,202)
Track 202 has a 1500-µm diameter bulb and 14 terminal particles. Synchrotron analysis indicated that the terminal particles consist of 5 silicates, 3 sulfides, 2 metals, and 4 particles of mixed composition. TP5, TP6, and TP9 were identified by Fe XANES as silicates (Fig. 4).

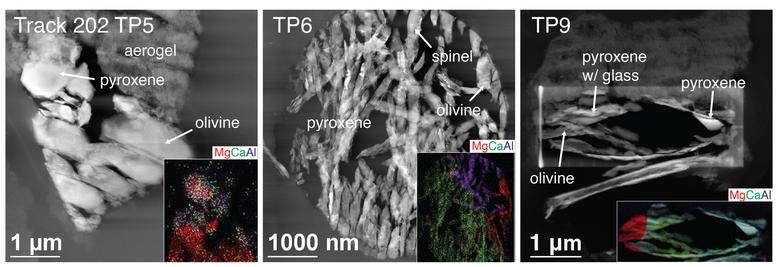

Figure 4: TEM and EDS maps of microtomed silicates in Track 202: TP5, TP6, and TP9.

EDS analyses for phases in Track 202 terminal particles are shown in Table 2.

- **TP5** consists of olivine and pyroxene; the pyroxene composition is slightly non-stoichiometric, possibly due to the presence of multiple-valence cations.
- **TP6** consists of olivine, pyroxene, and spinel. The spinel phase is primarily alumina-magnesia spinel, with ~5% fit to chromite, and an excess of SiO_2 from aerogel.
- **TP9** contains olivine, two pyroxenes, and glass. The pyroxenes primarily vary in Al and Ca. All phases show excess of SiO_2 from aerogel. The glass phase was small, yielding mixed analysis.

Element (Wt%)	O	Mg	Al	Si	Ca	Ti	Cr	Mn	Fe
TP5 olivine	42.2	26.2	bdl	18.6	397 ppm	bdl	bdl	400 ppm	13.0
TP5 pyroxene	46.0	14.3	2.8	27.1	1.7	0.23	0.71	bdl	7.2
TP6 olivine	42.4	25.6	bdl	19.2	bdl	bdl	bdl	bdl	12.8
TP6 pyroxene	44.7	9.5	4.2	25	12.6	866 ppm	bdl	bdl	3.9
TP6 spinel	42.8	11.9	34.8	0.88	bdl	bdl	1.1	bdl	8.5
TP9 olivine	43.9	22.3	0.31	22.7	0.30	bdl	bdl	913 ppm	10.4
TP9 pyroxene 1	44.2	10.0	bdl	26.6	14.6	0.33	0.15	0.14	4.0
TP9 pyroxene 2	44.8	9.4	5.4	24.3	11.7	0.33	0.16	930 ppm	3.9

Table 2: Quantitative EDS analyses for phases in Track 202 TP5, TP6, and TP9.

TP5 and TP6 were measured for O-isotopes. Their O-isotopic compositions (open symbols, Fig. 5) are consistent with other Wild 2 silicates. Ion probe analysis of TP9 is pending.

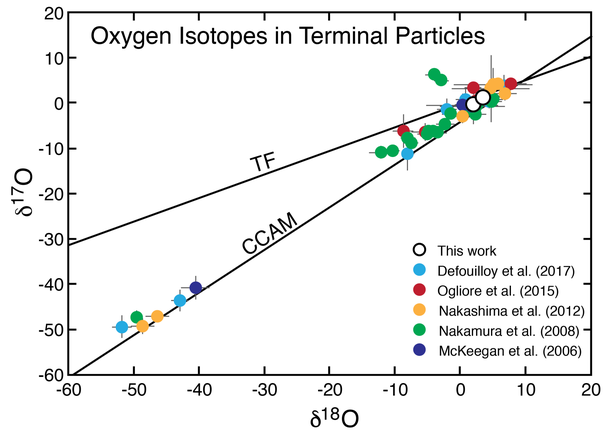

Figure 5: O-isotopic compositions of Track 202 TP5 and TP 6, compared to previously measured Stardust particles.

In Fig. 6, we show the scatter-plot of Fe chemical-map data indicating the presence of discrete metal particles in the bulb of track 202. These particles are not identifiable visually, so could not be targeted for extraction using the wafering technique. These might be isolated in the future using new fluorination techniques [6-8].

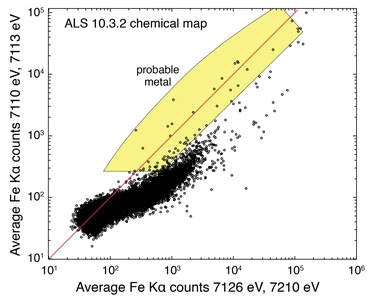

Figure 6: Fe spectral features for particles in Track 202 bulb material.

Track 203 (C2031,4,203)
Synchrotron analysis of the two large TPs indicates they are sulfides. A chemical map of the bulb, similar to Fig. 5, indicates presence of small metal particles. Sulfur-isotope measurements of the TPs are pending.

Track 204 (C2031,5,204)
The very small TP is identified as a sulfide by synchrotron X-ray analysis. We dissected the bulb material and pressed both sides into In in preparation for O-isotopic analysis. Analyses are pending.

Track 205 (C2031,6,205)
Track 205 has no identifiable TP. Chemical mapping at Fe showed that the bulb is dominated by Fe^{2+}-bearing material.

Track 206 (C2031,7,206)
A very small TP was identified as a sulfide from its faint signal during synchrotron X-ray analysis.

References: [1]Westphal A. J. et al. (2004) Meteorit. Planet. Sci. 39, 1375-1386. [2] Ogliore R. C. et al. (2015) Geochim. Cosmochim. Acta 166, 74-91. [3] Westphal A. J. et al. (2011) Meteorit. Planet. Sci. A 74, 5274. [4] Westphal A. J. et al. (2009) Ap. J. 694, 18-28. [5] Ogliore R. C. et al. (2008) Proc. 39th LPSC, 2363 [6] Westphal A. J. et al. (2004) Proc. 35 LPSC, 1860. [7] Martin A. A. et al. (2016) Meteorit. Planet. Sci. 51, 1223. [8] Haas, B. et al. (2018) these proceedings. [9] Defouilloy C. E. et al. (2017) Earth Planet. Sci. Lett. 465, 145-154. [10] Nakashima D. et al. (2012) Earth Planet. Sci. Lett. 357-358, 355-365. [11] Nakamura T. et al. (2008) Science 321, 1664-1667. [12] McKeegan K. D. et al. (2006) Science 314, 1724-1728.

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