Andrea Longobardo

SAMPLE RETURN MISSIONS

The Last Frontier of Solar System Exploration

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Edited by

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Elsevier
Radarweg 29, PO Box 211, 1000 AE Amsterdam, Netherlands
The Boulevard, Langford Lane, Kidlington, Oxford OX5 1GB, United Kingdom
50 Hampshire Street, 5th Floor, Cambridge, MA 02139, United States

British Library Cataloguing-in-Publication Data
A catalogue record for this book is available from the British Library

Library of Congress Cataloging-in-Publication Data
A catalog record for this book is available from the Library of Congress

ISBN: 978-0-12-818330-4

Publisher: Candice Janco
Acquisitions Editor: Peter Llewellyn
Editorial Project Manager: Allison Hill
Production Project Manager: Sruthi Satheesh
Cover Designer: Christian Bilbow

Typeset by Aptara, New Delhi, India

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CHAPTER 4

The Stardust sample return mission

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Chapter Outlines

4.1 Introduction

The *Stardust* spacecraft was the first mission to return solid samples from a body beyond the Moon. As the fourth NASA Discovery mission, it retrieved samples from the comet 81P/Wild 2, that is believed to have formed at the outer fringe of the solar nebula. The return of these samples provides unprecedented opportunities to compare astronomical (remote sensing) and sample analysis (ground truth) information for a known primitive solar system body. The samples make it possible to compare materials from the outer Solar System with sample-derived and astronomical data for asteroids, the parents of most meteorites, which formed much closer to the Sun. The samples returned by *Stardust* are the first primitive collected materials from a known body, and as such they provide contextual insight for all primitive meteoritic samples.

4.2 Mission overview

4.2.1 The target – comet 81P/Wild 2

Wild 2 is a Jupiter-family comet that has only been in its present orbit since 1974. Before 1974, it resided in an orbit with perihelion at 4.9 AU (near Jupiter's orbit)

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DOI: 10.1016/C2018-0-03374-5

and aphelion at 25 AU. This orbit had probably been stable for at least a few centuries (Sekanina 2003; Krolikowska and Suztowicz 2006). In 1974, a close encounter with Jupiter diverted Wild 2 into its current orbit, with perihelion at 1.58 AU and aphelion near 5.2 AU. Thus, 81P/Wild 2 is probably a "fresh" comet whose surface may have only recently been subjected to moderate solar heating. Wild 2 samples are expected to be remnants from the Kuiper Belt region of the solar nebula.

While 81P's history suggested it is an ideal object for the collection of primitive solar system materials, its selection as the target for *Stardust* depended largely on the comet's favorable orbit. A systematic search for comet flyby sample return opportunities showed that 81P/Wild 2 provided a trajectory to a "fresh" dusty comet with an encounter speed as low as 5.4 km/s (Tsou et al. 1994; Yen and Hirst 1997). Wild 2 met all four imperatives for the required *Stardust* trajectory: a dusty comet, a low comet encounter speed, reachable using a Delta II vehicle, and a small delta V requirement during flight.

4.2.2 Launch, orbital trajectory, and return

Stardust's trajectory allowed it to execute several scientific tasks during flight. *Stardust* was launched from Cape Canaveral on 7 February 1999. During its 7-year mission, *Stardust* made three heliocentric revolutions, all with perihelia at 1.0 AU. After the first revolution, the spacecraft performed an Earth Gravity Assist that raised its orbital aphelion to 2.7 AU and changed the orbital inclination to match Wild 2's orbit. The flyby encounter with 81P/Wild 2 and sample collection occurred at an encounter speed of 6.12 km/sec on 2 January 2004. The spacecraft returned to Earth on 15 January 2006. During the inbound portions of the orbits, the spacecraft's trajectory roughly paralleled that of contemporary interstellar dust particles entering the Solar System, resulting in a reduced relative speed with these particles. This allowed for 246 days of collection of contemporary interstellar dust on the back side of the sample tray assembly during two of the orbits. The trajectory also allowed for a flyby of the asteroid Annefrank on 2 November 2002 (Duxbury et al. 2004). After return of the sample capsule, the main spacecraft was diverted to a close encounter with comet 9P/Tempel 1 where it imaged the crater made by the Deep Impact mission (Veverka et al. 2013).

4.2.3 Spacecraft description

A description of the hardware components of *Stardust* can be found in Brownlee et al. (2003) (Fig. 4.1). The spacecraft carried several instruments including a camera (Newburn et al. 2003), a dust flux monitor (Tuzzolino et al. 2003), and a dust analyzer (Kissel et al. 2003). The primary 'instrument' was a deployable dust collector that used low density aerogel as a collecting medium (Tsou et al. 2003). The aerogel collection area is divided up into 130 2 × 4 cm rectangular and 2 trapezoidal cells (Fig. 4.2). The variable aerogel density for the Wild 2 collection side was 5 mg/ml to 50 mg/ml and was 2 mg/ml to 20 mg/ml for the interstellar capture cells on the collector's back side. In addition, pure,

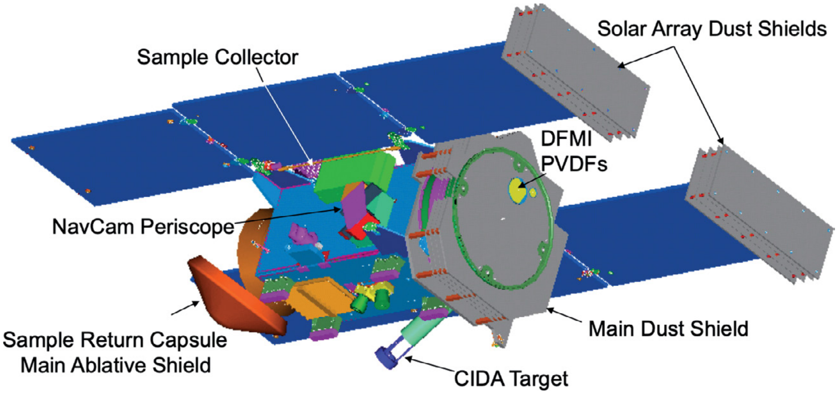

Fig. 4.1 ***Schematic of the Stardust spacecraft showing the positions of the onboard instruments.*** The gray portions of the diagram represent the leading edge Whipple shields that protected the spacecraft from cometary dust impacts.

100 micron thick aluminum foils wrapped the walls of the aerogel frames to facilitate cell removal, and the foils exposed portions were good targets for acquisition of dust impact craters. The Wild 2 and interstellar trays were mounted back to back and had a total exposed aerogel surface area of 1039 cm^2 and 1037 cm^2, respectively. The total exposed aluminum foil is about 15 percent of the exposed aerogel surface area.

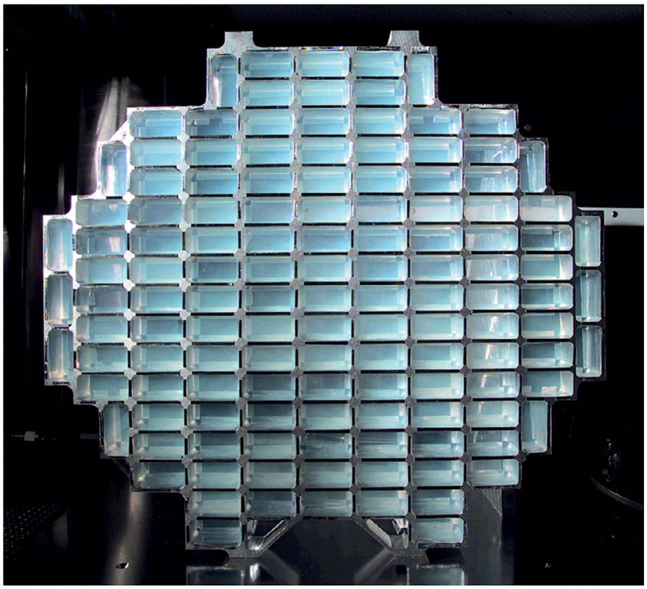

Fig. 4.2 ***The cometary collector tray contained multiple individual aerogel tiles.*** A second interstellar aerogel collector tray was placed back-to-back with the cometary tray.

4.3 Results

4.3.1 Flyby observations

An overview of the *Stardust* flyby of 81P/Wild 2 on 2 January 2004 can be found in Tsou et al. (2004). *Stardust* flew 236.4 ± 1 km from the comet's center when the comet was 1.86 AU from the Sun and the encounter occurred as planned. All the onboard instruments obtained data during the flyby and the deployed aerogel collector collected particles from the comet's coma.

4.3.1.1 Camera images

The *Stardust* camera obtained 72 images of the nucleus of 81P/Wild 2 during the flyby (Tsou et al. 2004). Close encounter imaging was done with a camera that covered the spectral range 380 nm – 1000 nm without filters using two exposure times – 10 ms for nucleus imaging alternated with 100 ms for nucleus tracking (Fig. 4.3).

Stereoscopic images of the nucleus show a diverse and complex variety of landforms not seen from earlier comet flybys of 1P/Halley and 19P/Borrelly. These include craters, excavation zones, flat-floored depressions, surface crusts, landslides, lineaments, terraces, spires/pinnacles (some 100 m in height), steep cliffs, overhangs, and small bright patches (potential vents or exposed ice). Wild 2 does not have smooth plains as seen on other comet surfaces. Most surface features are likely associated with ice sublimation processes. A triaxial ellipsoidal fit of the images yielded principal nucleus radii of 1.65 × 2.00 × 2.75 km (± 0.05 km). The longer exposures were used to identify the orientations and the approximate source locations of at least 20 collimated and partially overlapping jets of dust emitted from the nucleus.

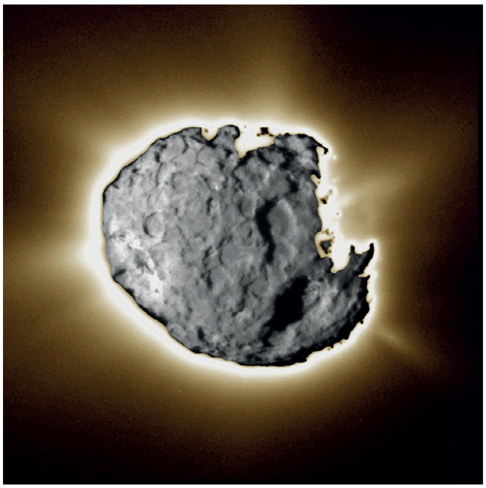

Fig. 4.3 A ***composite figure.*** made by superimposing long and short exposure images of the nucleus of 81P/Wild 2. The short exposure shows the surface features of the nucleus and the long exposure shows the gas/dust jets of gas emitted by the nucleus.

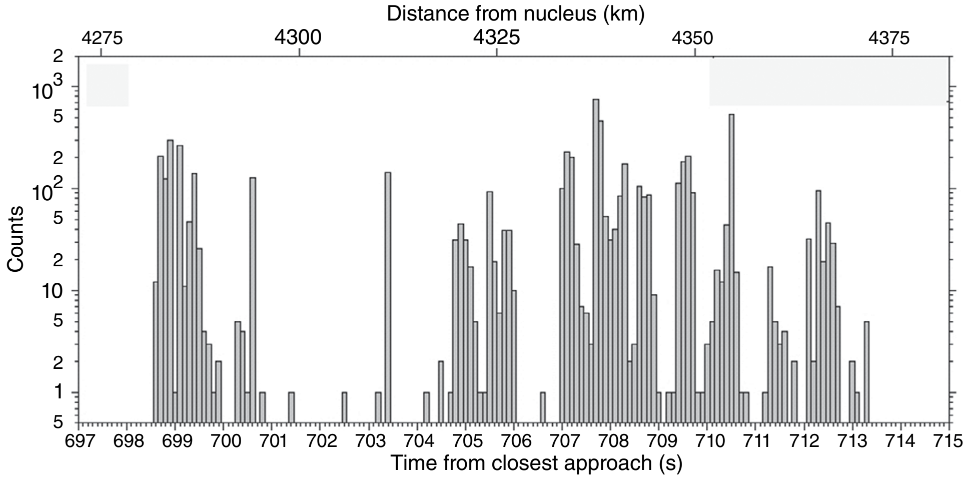

Fig. 4.4 Eighteen seconds of data from the Dust Flux Monitor taken when the spacecraft was ~4300 km from the nucleus of 81P/Wild 2, showing the variable impact rate seen as the spacecraft passed through the comet's coma *(adapted from Clark et al. 2004)*.

4.3.1.2 Dust flux monitor data

During the flyby the dust detectors recorded particle impacts of masses ranging from 10^{-11} to > 10^{-4} g. The impact distribution along *Stardust's* flight path was extremely non-uniform. Dust impacts occurred in short "bursts" that could contain nearly a thousand particles separated by intervals in which no dust arrived at all (Tuzzolino et al. 2004) (Fig. 4.4). The most likely explanation for this behavior is the ejection of larger particle aggregates from the nucleus that fragmented as they moved out into the coma (Clark et al. 2004). At least seven impacting particles were big enough (the largest ~4 mm in diameter) to penetrate the spacecraft's front bumper shield and be detected by the flux monitor's acoustic sensors (Green et al. 2004). These data indicated that the expected samples were successfully collected by the aerogel collector during the flyby.

4.3.2 Results obtained from returned samples

The *Stardust* Sample Return Capsule (SRC) returned to Earth at the Utah Test and Training Range on January 2, 2006 and was quickly recovered (Fig. 4.5). The SRC was transported to a temporary cleanroom where it was opened and the sample canister was removed and placed in a container purged by curatorial grade N_2. The canister was flown to NASA's Johnson Space Center and opened in a cleanroom made specifically to receive and curate the samples. Samples removed from the aerogel collector were subjected to a 6 month preliminary examination by prearranged teams that studied the chemical, physical, spectral, and isotopic nature of the samples before the samples were made available for general distribution to the science community.

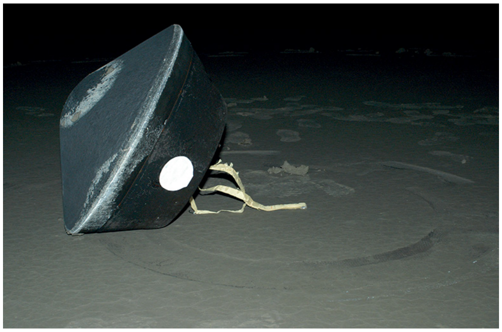

Fig. 4.5 The Stardust Sample Return Capsule as found during its recovery from the Utah Test and Training Range.

An extensive effort was made to assess contamination of the returned samples and concluded that contamination during the design, construction, and flight of the spacecraft, and during and after recovery of the SRC did not contribute significant material to the collectors (Sandford et al. 2010). The largest concern is associated with contaminant particles and structural carbon within the original aerogel, although these materials can generally be distinguished from the returned cometary samples.

4.3.2.1 Physical nature of the dust

One of the first science results from the returned samples was that Wild 2 contains a diverse range of particles. The aerogel capture track geometries clearly showed the presence of both strong solid materials that produced long thin tracks and friable particles that produced wide (bulbous) tracks (Brownlee et al. 2006; Burchell et al. 2008) (Fig. 4.6). Many of the grains are polymineralic. Except for surface abrasion, most grains >2 μm are well preserved, while many of the smaller ones were altered or destroyed during high speed capture into aerogel (Brownlee et al. 2006). Some submicron grains did survive capture, but it is clear that others melted and dissolved into melted aerogel lining track walls. The preferential destruction of the finest grained fraction affects the completeness of our full understanding of the comet's mineralogical composition.

4.3.2.2 Elemental composition

During the preliminary analyses of Wild 2 samples, results from the aerogel and foils were combined to seek a "comprehensive" elemental analysis of the Wild 2 particles (Flynn et al. 2006). Twenty-three tracks were analyzed by synchrotron X-ray Fluorescence (SXRF) to determine abundances for elements heavier than P. One track was also split lengthwise and analyzed by time-of-flight–secondary ion mass spectrometry (TOF-SIMS) analysis for some lighter elements, particularly Mg and Al (the silica aerogel prevented measurement of Si and O). Residues in 7 Al foil craters were also analyzed by scanning electron microscopy using energy-dispersive X-ray analyses (SEM-EDX)

Fig. 4.6 ***Cometary particles impacting Stardust aerogel collector tiles created several types of tracks.*** Single, strong particles created long, thin carrot tracks like the one on the left of the image. Weaker aggregate particles came apart during impact and produced more bulbous tracks like the two in the center of the image. Particles in this image entered from above and the surface of the aerogel is at the top of the image.

and TOF-SIMS. These 'bulk' compositions are compared to the elemental composition of CI chondrites, which were generally thought to represent the closest analogues to cometary material (Gounelle et al. 2006).

Since the mineralogy of the tracks varied so widely, it was difficult to arrive at a 'bulk' composition for the overall collected sample. Many terminal particles were dominated by a single mineral, generally olivine, pyroxene, or Fe-Ni-Zn sulfide (Zolensky et al. 2006) and the fraction of the total Fe detected in the studied terminal particles varied from 0 percent to almost 60 percent. The spatial distributions of other elements in each track were similarly varied. Thus, terminal particle analysis provides uncertain information on the bulk elemental composition of Wild 2.

The mean composition of the Wild 2 coma dust was calculated by Flynn et al. (2006) by summing the measured abundance of each element over all 23 analyzed tracks. It was found that approximately 90 percent of the material in an entering cometary grain ended up being distributed along the tracks, with only ~10 percent being present in the terminal grains. The Fe-normalized mean element abundances of Wild 2

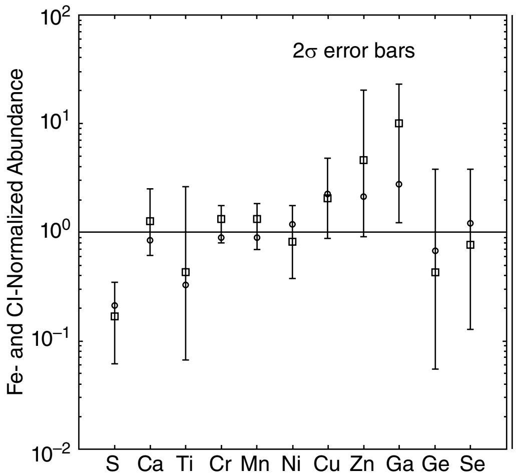

Fig. 4.7 ***CI- and Fe-normalized mean composition determined by summing the 23 whole-track analyses (squares) and by summing the same data set except for the particle having the highest Fe content (circles).*** The vertical bars show the degree of diversity of the mean composition (after Flynn et al. 2006). The horizontal line represents CI abundances.

tracks gathered in this fashion for Ca, Ti, Cr, Mn, Ni, Ge, and Se (Fig. 4.7) are consistent with CI values at the 2σ confidence level. Ge and Se were detected in only a few particles, so their values are very uncertain. Sulfur is depleted relative to CI values, and Cu, Zn, and Ga are enriched.

Westphal et al. (2009) reported additional compositional measurements of the Wild 2 tracks, based on SXRF measurements of the relative concentrations of the chemical state of iron. They reported significantly higher S/Fe atom ratios of > 0.31, which is higher than in most chondritic meteorites.

Analyses of impact residue in 7 Al foil craters provided additional element-to-Si ratios, although only Mg, Si, and Fe were detected in all analyzed craters. The Si-normalized mean composition detected in four craters has an abundance difference from CI of less than 50 percent for Mg, Ca, and Fe. An observed S depletion is consistent with track results. Residues in five craters were also analyzed by TOF-SIMS. The Si-normalized mean abundances are consistent with CI for Mg, Ca, and Ni, but small depletions were seen for Cr and Fe, consistent with the SEM-EDX results. Li, Na, and K appeared to be enriched.

Flynn et al. (2006) analyzed ~300 ng of Wild 2 dust and the material appears depleted in S and Fe relative to Si and enriched in the moderately volatile minor elements Cu, Zn, and Ga relative to CI. These trends were previously reported in the fine-grained, anhydrous chondritic IDPs (Schramm et al. 1989; Flynn et al. 1996). However, the abundances of Cu, Zn, and Ga are not well determined in the latter, suggesting that Wild 2 particles and anhydrous IDPs may better reflect the composition of the solar nebula.

4.3.2.3 Mineralogy

Before the return of *Stardust* samples there were a number of different opinions concerning what Wild 2 coma dust would be like. Possibilities included (a) materials very similar or identical to anhydrous chondritic interplanetary dust particles (IDPs) (Bradley 2014), (b) amorphous nebular condensate silicates (Rietmeijer et al. 2009), (c) products of annealing of nebular condensates (Kimura et al. 2011), or (d) materials largely made up of presolar grains with direct interstellar heritage (Engelhardt et al. 2017). Wild 2 could have also been an interstellar visitor like 1I/ʻOumuamua (Jewitt et al. 2017). The actual Wild 2 samples did not match any of these possibilities.

The mineral chemistry of the collected samples is a remarkably complex mix of unequilibrated phases. The most abundant phases are the ferromagnesian silicates olivine and pyroxene (Fig. 4.8), and are similar to materials found in most anhydrous chondritic

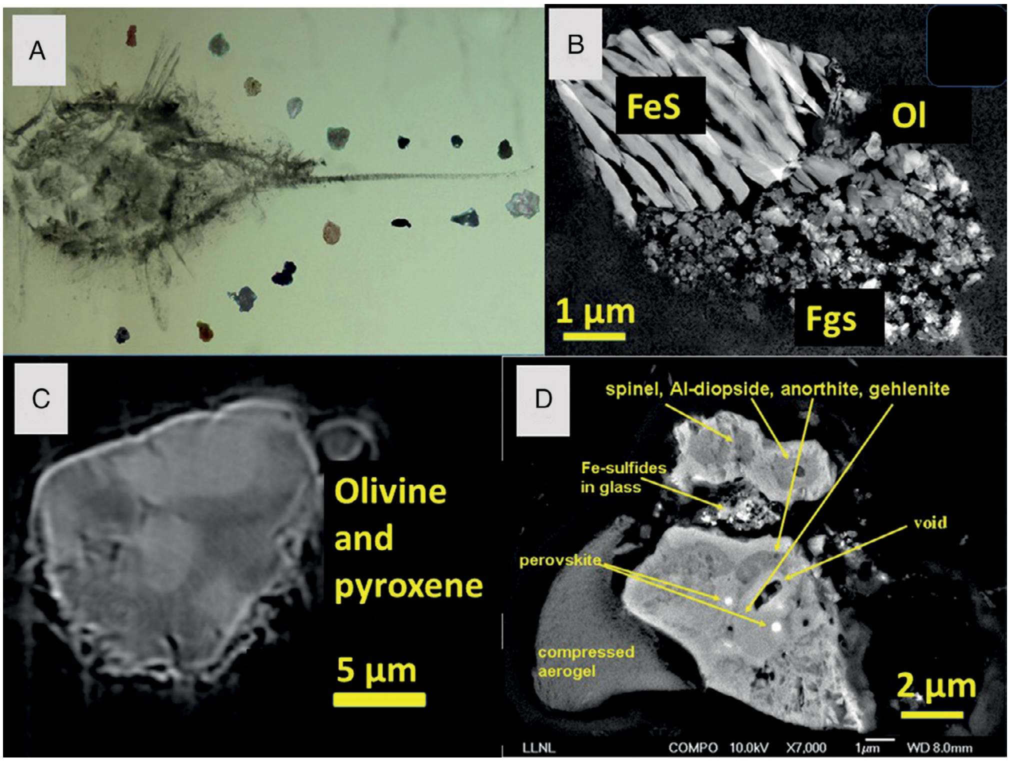

Fig. 4.8 ***Wild 2 coma grains.*** (A) Transmitted light view of track 35 (1.5 mm long) with images of extracted grains shown alongside; grains vary from 8–23 μm in diameter and are not shown to scale. (B) Back-scattered electron image of a terminal grain from track 57. The troilite (FeS) and olivine (ol) crystals apparently shielded the fine-grained material (Fgm) from destruction during capture (after Brownlee et al. 2006). (C) X-ray computed tomographic image of a terminal grain from track 35 identified by Nakamura et al. (2008) as a chondrule, containing olivine and pyroxene. (D) Back-scattered electron image of a CAI terminal grain from track 25 (Inti) (after Zolensky et al. 2006).

IDPs and unequilibrated or unaltered chondrites. The samples also contained chondrule fragments (Nakamura et al. 2008). The expectation that amorphous silicates and fine-grained annealed products of these minerals would predominate in Wild 2 grains was clearly incorrect, at least for micron and larger grains.

The major element compositional range of olivine, a reflection of the formation conditions and thermal history of astromaterials, is the largest of any known astromaterial, and the distribution is very flat, with no expected peak for forsterite (Frank et al. 2014). Comparisons of Wild 2 samples with other available astromaterials initially proved to be difficult since Wild 2 samples require study, by necessity, at the micron size scale – a scale for which there was a lack of comparable information for most other astromaterials. Subsequent detailed measurement of olivine compositions in chondrite matrix revealed how unique the range is for Wild 2 samples (Frank et al. 2014; Joswiak et al. 2014a; Brownlee and Joswiak 2017; Defouilloy et al. 2017). The flat olivine compositional distribution for Wild 2 samples indicates no thorough heating of the samples occurred after they were accreted into the comet. It also suggests that the formation regions of the olivine in Wild 2 samples differed from that of any of the carbonaceous chondrites. In addition, Wild 2 grains include a population of Ca-enriched, Mn-depleted olivine crystals not found in any other known astromaterial (Frank et al. 2014). Wild 2 also contains low-iron, manganese-enriched forsterites (called LIME olivines) that are commonly found in IDPs and carbonaceous chondrites and proposed to be early nebular condensates (Klock et al. 1989; Ebel et al. 2012).

In contrast to the major elements, Frank et al. (2014) reported *depletion* of Cr from the FeO-rich olivines in Wild 2, comparable to that attributed to mild thermal metamorphism petrologic grade (3.05–3.2) chondrites. Since Cr is highly mobile under thermal metamorphism as low as 200 °C, it is a sensitive indicator of heating events and it shows greater depletion in smaller grains (Grossman and Brearley 2005). Thus, olivine minor element compositions suggest that some, but not all, Wild 2 materials experienced thermal metamorphism prior to incorporation into their ice-rich parent body. Unfortunately, it is unclear whether the mineralogical criteria for thermal metamorphism derived from coarser chondrule silicates in ordinary and carbonaceous chondrites can be applied to the fine-grained Wild 2 samples. It is also not known what the initial Cr contents of olivine were across various early solar system environments.

The measured Mn content in >200 of Wild 2 olivine grains having a broad range of Fe content show distinctly different trends than seen in olivines from specific chondrite groups (Frank et al. 2014; Brownlee and Joswiak 2017), suggesting that comet olivine formed in a broader range of environments than these specific chondrite groups.

Comprehensive results for Wild 2 pyroxenes have not yet been published, but a number of Wild 2 particles, named "Kool" grains (Kosmochloric high-Ca pyroxene and FeO-rich olivine), contain assemblages of FeO-rich olivines, Na- and Cr-rich clinopyroxenes (usually augites), poorly-crystallized albite or albitic glass, and spinel (Joswiak et al. 2009; 2012). Kool grains have been reported in some chondritic IDPs. The textures, grain sizes, and mineral assemblages of these grains are consistent with high temperature formation processes, rather than direct condensation or thermal annealing of amorphous condensates. The O isotopic composition of one Wild 2 Kool grain has been reported, and is comparable to some type II (FeO-rich) chondrule olivines from OC, R, and CR chondrites (Krot et al. 2006; Connolly and Huss 2010; Kita et al. 2010; Isa et al. 2011). However, actual Kool grains have not yet been observed in chondrites.

The discovery of high temperature materials like chondrule and CAI (Ca-Al-rich inclusions) fragments (Fig. 4.8C,D) among Wild 2 grains was unexpected and contrary to the idea that comets formed in isolation from the inner Solar System (Zolensky et al. 2006; McKeegan et al. 2006; Joswiak et al. 2012, 2014a,b). CAIs containing olivines, pyroxenes, sulfides, and refractory oxides have been reported from at least 5 different particle tracks, suggesting that these high-temperature components constitute ~2 percent of the collected sample (Joswiak et al. 2017). Mineral assemblages, chemistries, and bulk particle compositions indicate these grains are most similar to fine grained CAIs in carbonaceous chondrites.

Some Wild 2 grains have igneous mineralogies, textures, and bulk oxygen isotope compositions consistent with an origin as fragments of chondrules like those found in carbonaceous chondrites (Nakamura et al. 2008; Matzel et al. 2010; Joswiak et al. 2012; Ogliore et al. 2012; Gainsforth et al. 2015). The abundance of chondrule fragments in Wild 2 is > 5–10 percent and could be much higher. The exact relationships of Wild 2 chondrules to those in chondrites is not known, although the similarities are striking. It remains to be determined if Wild 2 chondrules and CAIs sample the same populations of components found in chondrites (Westphal et al. 2017). Regardless, they must have formed via high temperature processes (Gainsforth et al. 2015). These igneous materials probably require large scale mixing in the early Solar System, although there are proposals for high-temperature processes in the outer Solar System (Sanborn et al. 2017; Kruijer et al. 2017). The common presence of these materials in interplanetary dust particles of likely cometary origin suggests that they are common in comets.

Wild 2 samples include abundant sulfides. These are predominantly troilite (FeS), pyrrhotite $Fe_{(1-x)}S$, with lesser pentlandite (ideally $(FeNi)_9S_8$), but the occurrence of unusual sulfides (including ZnS) implies complex sulfide formation processes (Zolensky et al. 2006; Westphal et al. 2009; Schrader et al. 2016). The rare presence of cubanite ($CuFe_2S_3$) has been interpreted as evidence for possible rare aqueous processing (Berger et al. 2011), although a primary origin for this phase is also possible. Wild 2 Fe-Ni sulfides plot within the Fe-Ni-S ternary plot as two modes: either pyrrhotite/troilite, or pentlandite, with few compositions between (Zolensky et al. 2006). This limits the extent of possible aqueous processing since the aqueous alteration seen in hydrous IDPs produces assemblages bridging the gap between the pure end member phases. Understanding the sulfide mineralogy in the returned samples is complicated by the presence of FeS formed by melting of pre-existing grains during capture in the aerogel.

Chondritic meteorites often contain materials resulting from the activity of aqueous fluids (Zolensky and McSween 1988; Brearley 2006). Searches in Wild 2 samples for minerals unambiguously requiring formation via aqueous fluids have largely been unsuccessful. A possible exception is the cubanite grain mentioned above. Another is a magnesium carbonate reported by Mikouchi et al. (2007). Several Ca carbonates have also been reported, but these were ascribed to contamination from aerogel impurities. Magnetite and chromite have been reported from Wild 2 grains (Changela et al. 2012; Bridges et al. 2015). In chondrites and IDPs such phases have been proposed to result from aqueous alteration and oxidation of metal and Fe-Ni sulfides (Kerridge et al. 1979; Zolensky and McSween 1988), but they can also be produced in the absence of aqueous fluids (Lauretta and Schmidt 2009). The lack of phyllosilicates in analyzed Wild 2 materials could be ascribed to post-alteration thermal neomorphism impact shock or to capture heating, but heated phyllosilicates have characteristic textures (Nakamura 2005; Tonui et al. 2014) not observed in the Wild 2 materials. Thus, there is currently no unambiguous evidence for liquid water having been present on the comet.

GEMS (Glass with Embedded Metal and Sulfides) are common sub-micron sized assemblages in anhydrous chondritic IDPs, but found in only one meteorite (Ningqiang) (Rietmeijer 1994; Bradley 1994; Zolensky et al. 2003). They have been variously proposed to be radiation-damaged early nebular solids or preserved presolar materials, and they have been vigorously searched for in Wild 2 materials. A few Wild 2 components have been proposed to be GEMS (e.g., Gainsforth et al. 2016), but since very similar silica-sulfide rich aggregates are a major byproduct of the capture process of chondritic materials in silica aerogel (Barrett et al. 1992), an unambiguous identification of a true GEMS assemblage has proven to be elusive (Ishii 2019). GEMS are easily degraded by modest heating and it is possible that Wild 2 contained abundant submicron GEMS that were melted to form the silica-rich melt on track walls. It is also possible that Wild 2 does not contain GEMS.

4.3.2.4 Organics

Comets may have had a significant role in delivering volatiles and organics to the early Earth and these materials may have played a role in the origin of life (Oró 1961; Chyba and Sagan 1992). Considerable emphasis was placed on searching for organics in the returned samples (Sandford 2008, 2009). This task was made difficult by the small sizes of the samples, the complexity of the organic materials present, the fact that organics fared relatively poorly during hypervelocity collection, and the presence of structural carbon in the aerogel collection material. Nonetheless, it was possible to identify cometary organics in the samples by the presence of non-terrestrial D/H and $^{15}N/^{14}N$ isotope ratios or by clear associations with surrounding mineral grains.

Organics found in Wild 2 samples show a heterogeneous and unequilibrated distribution in both abundance and composition. Some of the organics are similar, but not identical, to those in IDPs and carbonaceous meteorites, but there is evidence for additional organic materials not found in meteorites (Sandford et al. 2006). These additional organics are more labile, richer in oxygen and nitrogen, and aromatic-poor compared with meteoritic organics.

Comparisons with IDPs and meteorite organics are problematic since the hypervelocity impacts associated with aerogel collection resulted in the destruction and alteration of some of the collected organics. IR mapping of tracks shows that the aerogel surrounding some (but not all) tracks contains excess absorption by aliphatic $-CH_3$ and $-CH_2-$ groups, suggesting that some of the organic material in the arriving particles was vaporized during impact and redistributed into the surrounding aerogel (Sandford et al. 2006; Bajt et al. 2009). It is therefore difficult to determine the actual abundance ratio of organics to mineral phases in the original particles.

Infrared spectra of individual particles and organic regions within them show absorption bands of $-CH_3$, $-CH_2-$, $C{=}O$, and CC groups (Keller et al. 2006; Sandford et al. 2006; Rotundi et al. 2008; Bajt et al. 2009). The observed aliphatic CH stretching features of *Stardust* particles resemble those seen in IDPs in terms of peak shapes, positions, and the $-CH_2-/-CH_3$ band depth ratio, but differ somewhat from those seen in primitive carbonaceous chondrite meteorites like Orgueil and Murchison.

The presence of aromatic organics is seen in both IR and Raman spectra of collected particles (Keller et al. 2006; Sandford et al. 2006; Rotundi et al. 2008). Raman spectra of Wild 2 samples are dominated by the aromatic D and G bands near 1360 and 1590 Δcm^{-1} and are superimposed on a fluorescence background of variable intensity. The D and G band parameters of the samples indicate the presence of amorphous carbonaceous materials that scatter across the entire meteoritic field, but are best matched to the range seen in IDPs.

The technique of X-ray Absorption Near Edge Spectroscopy (XANES) has been very useful for the analysis of *Stardust* samples (Sandford et al. 2006; Cody et al. 2008; Matrajt et al. 2008; Wirick et al. 2009). C-XANES spectra of *Stardust* samples look similar to those of meteoritic organics and most closely resemble those of IDPs. C-, N-, and O-XANES spectra reveal considerable chemical complexity across the range of organic samples analyzed. The cometary organics contain low concentrations of aromatic and/or olefinic carbon relative to aliphatic and heteroatom-containing functional groups, e.g., amide, carboxyl, and alcohol/ethers. The atomic ratios for N/C and O/C derived from XANES data reveal a wide range in heteroatom content and these ratios are higher than those seen in primitive meteoritic organic matter (Fig. 4.9). The wide range in chemistry, both in elemental abundances and specific organic functional groups, suggests that the comet Wild 2 organics likely have multiple origins.

Organic species have also been detected using two-step laser desorption / laser ionization mass spectrometry (L^2MS) (Sandford et al. 2006; Clemett et al. 2010). L^2MS

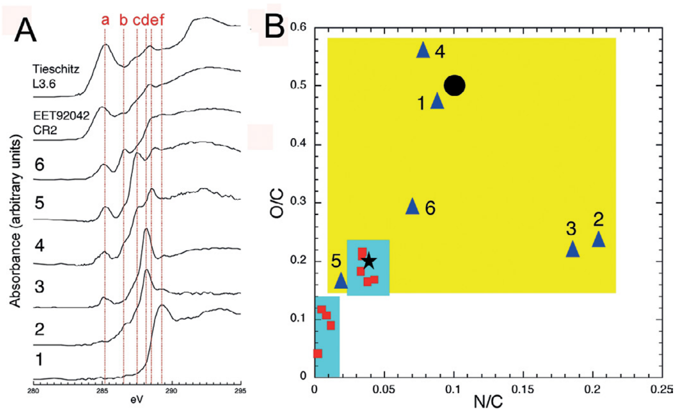

Fig. 4.9 (A) XANES data from different Wild 2 grains show a range of spectra. Specific organic functional groups are highlighted in the figure. on the left (dashed lines a to f): (a) $C = C$ at ~ 285.2 eV; (b) $C = C$–O at ~ 286.5 eV; (c) $C = O$ at ~ 287.5 eV; (d) N–$C = O$ at 288.2 eV; (e) O–$C = O$ at 288.6 eV; and (f) C–O at 289.5 eV (B) Wild 2 particles (numbered triangles) show unusually high N/C and O/C ratios relative to chondritic organic matter (squares). Average values for comet Halley particles and stratospheric IPDs are marked by the black star and the solid circle, respectively *(figure adapted from Sandford et al. 2006)*.

mass spectra obtained from individual particles and aerogel along impact tracks show the presence of multiple polycyclic aromatic hydrocarbons (PAHs) and their alkylated derivatives. Two distinct populations of PAHs can be distinguished. In the first population, benzene and naphthalene (1- to 2-ring PAHs) and their alkylated variants are seen in the absence of moderate mass (3- to 6-ring) PAHs. These distributions are uncharacteristic of meteorites and IDPs, but closely resemble the pyrolysis products of meteoritic macromolecular organics and have been observed in high-power laser L^2MS measurements of aerogel tiles (Spencer et al. 2009). This suggests that many lower mass PAHs may not be cometary but instead originate from impact processing of C original to the aerogel. The second population of PAHs has a more complex compositional distribution in which the dominant observed species are naphthalene ($C_{10}H_8$; 2 rings), phenanthrene ($C_{14}H_{10}$; 3 rings), and pyrene ($C_{16}H_{10}$; 4 rings), along with their alkylated homologs extending up to at least C4-alkyl. This second distribution resembles that found in matrix material in the Murchison carbonaceous chondrite and some IDPs (Clemett et al. 1993, 2010).

Amines and amino acids have also been detected. These were not found in individual cometary grains, but were instead detected within the general volume of aerogel tiles using liquid chromatography with UV fluorescence detection and time of flight mass spectrometry (LC-FD/ToF-MS). Glavin et al. (2008) detected a suite of amines and amino acids, including glycine, in acid-hydrolyzed, hot-water extracts of *Stardust* aerogels and Al foils above background levels. Most of the primary amines detected were also present in the flight aerogel witness tile that was not exposed to the comet, indicating that they were terrestrial in origin. However, excesses of methylamine (MA) and ε-amino-n-caproic acid (EACA) in comet-exposed aerogel suggested that these volatile amines were captured from comet 81P/Wild 2 and present in an acid-hydrolyzable bound form in the aerogel (Glavin et al. 2008). Subsequently, Elsila et al. (2009) showed that the EACA had a $\delta^{13}C$ value of −25 ± 2‰, indicating a terrestrial origin (EACA is likely due to contamination from the Nylon-6 used to bag samples during curation). In contrast, glycine was observed to have a $\delta^{13}C$ value of +29 ± 6‰, which strongly suggests an extraterrestrial origin. This represents the first detection of a cometary amino acid.

4.3.2.5 Isotopes

Isotopically anomalous grains are found in Wild 2 grains at approximately the same level as in chondritic IDPs and the most pre-solar-rich chondrites (Stadermann et al. 2008). Thus, the composition of Wild 2 is *not* dominated by isotopically anomalous presolar grains.

The isotopically anomalous presolar grain abundance in Wild 2 samples has been best measured by detailed SIMS analysis of craters in Al foil that surrounded each aerogel cell. These indicate pre-capture abundances of 600–830 ppm for O-rich presolar grains and at least 45 ppm for SiC grains larger than 300 nm (Floss et al. 2013). This abundance is at the upper level for that reported for chondritic IDPs and higher than found in most chondrites. If the comet contains isotopically normal interstellar grains there is no existing method to determine their abundance.

High precision oxygen isotope analyses reveal the range of compositions of the returned silicates. Many silicates fit a pattern of oxygen composition vs. Fe content that is similar to CR chondrite olivine (Defouilloy et al. 2017). The oxygen isotope compositions reveal components with affinities to carbonaceous and ordinary chondrites, the presence of relict grains in chondrules, and ^{16}O-rich materials that include CAIs and Mg-rich condensates.

Organic materials in meteorites and IDPs often show non-terrestrial values for D/H and $^{15}N/^{14}N$ (Messenger 2000; Keller et al. 2004; Busemann et al. 2006) and the same is true for the organic materials returned from Wild 2, showing a large excesses in D and ^{15}N (McKeegan et al. 2006; Matrajt et al. 2008). These isotopic anomalies demonstrate conclusively that the associated organics are not terrestrial contaminants and provide insights into the types of environments and processes involved in their formation.

The distribution of anomalous enrichments of D and ^{15}N in *Stardust* samples are highly heterogeneous and the range of excesses span a similar range as that seen in IDPs. The two anomalies do not directly correlate in either location or magnitude; materials are seen that contain none, one, or both of the D and ^{15}N excesses. The decoupling of D and ^{15}N anomalies and the variable magnitudes of the effects suggest that they were formed by multiple interstellar/protostellar processes and environments that predate the formation 81P/Wild 2 (Sandford et al. 2001; McKeegan et al. 2006). Their presence also

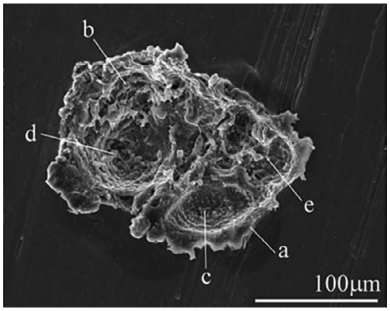

Fig. 4.10 ***Wild 2 particle impact crater in Al foil.*** The impactor was a loose aggregate composed of Mg silicate, Ca bearing silicate, chondritic, and sulfide components that produced the complex multi-pit crater lined with comet residue *(Kearsley et al. 2008)*.

indicates that these organics have experienced little alteration since their incorporation into the cometary nucleus.

4.3.2.6 Craters

While the aerogel collectors were *Stardust*'s primary means of capturing cometary samples, the forward facing aluminum foils that held aerogel capture cells in place were also exposed to the incoming flux of cometary particles. Particles impacting the foil created hypervelocity impact craters that could be individually studied (Hörz et al. 2006). The morphologies of these craters indicated that they were made by particles varying from individual dense mineral grains to loosely bound, polymineralic aggregates.

Residual impactor material was found in some craters and was studied by energy dispersive X-ray microanalysis (Kearsley et al. 2008). These showed that some particles included coarse (>10 µm) mafic silicate grains dominated by a single mineral species of density around 3–4 g cm^{-3} (such as olivine). Other grains were porous, low-density aggregates from a few nanometers to 100 µm, with an overall density that may be lower than 1 g cm^{-3}, containing mixtures of silicates, sulfides, and possibly glass. In one large aggregate crater (Fig. 4.10), the combined diverse residue composition is similar to CI chondrites. On the whole, the inferred mineral assemblages are very similar to the most common species reported from aerogel tracks.

4.3.2.7 Interstellar particles

Contemporary interstellar dust grains passing through our Solar System were first observed by detectors aboard the Ulysses spacecraft (Grün et al. 1994) and subsequently verified by data from detectors on board the Galileo, Cassini, and Helios spacecraft (Krüger et al. 2019). The original goal of the *Stardust* mission was to collect some of this "fresh" interstellar dust, although this dropped to a secondary goal as the mission developed. The Ulysses and the other spacecraft data indicated that the maximum size of the interstellar grains would be ~ 1 µm and that they would be relatively rare. The effort to collect interstellar dust suffered from the fact that *Stardust's* dust collection periods occurred close to solar maximum, when the flux of interstellar grains to the inner Solar System is lowest. As a result, only a dozen grains spread across the entire Stardust Interstellar Particle Experiment (ISPE) aerogel collector were expected to be collected.

It was not clear when the spacecraft was launched how such minuscule and dispersed grains would be recognized and analyzed. Fortunately, Prof. Andrew Westphal (UC Berkeley), developed a plan for the public to locate the grains through one of the largest distributed planetary science efforts in history. The returned ISPE aerogel cells were scanned using an automated system, which recorded millions of microscope focusing 'movies' across each aerogel cell. These movies were placed online and more than 20,000 volunteers (self-named "dusters") searched them for traces of captured ISPE grains. Over half of the ISPE aerogel tray has been scanned in this effort (Westphal et al. 2014a,b).

Using this procedure, features of special interest were identified and a subset considered to have the greatest possibility of being interstellar grains were removed from the aerogel cells. These were mounted between 70 nm thick sheets of Si_3N_4 for protection. The resulting mounts were transparent to synchrotron X-rays, permitting analyses to be performed on the grains while still encased in aerogel. Analytical techniques used included Scanning Transmission X-ray Microscopy (STXM), Fourier Transform Infrared Spectroscopy (FTIR), X-Ray Fluorescence spectroscopy (XRF), and X-Ray Diffraction (XRD) (Westphal et al. 2014a). These analyses were carefully chosen to be non-destructive to the interstellar candidate grains. The chemistry of the majority of these samples were found to be consistent with secondary impact ejecta from *Stardust's* solar panels or sample return capsule, injected into the aerogel when interplanetary dust particles or comet coma grains impacted on these parts of the spacecraft.

Westphal et al. (2014a) reported on the mineralogy and bulk composition of the first three recognized *probable* interstellar grains (Fig. 4.11). Sample *I1043,1,30,0,0* (named "Orion") is a mixture of shocked forsteritic olivine (Fo >90), spinel, iron metal nanoparticles and one additional unidentified iron-bearing phase. Elemental abundances, normalized to magnesium and the composition of CI meteorites, show ten-fold enrichments in Al and Cu, depletions for Si, Ca and near normal Fe, Cr, Mn, and Ni. Sample *I1047,1,34,0,0* ("Hylabrook") contains shocked, partially amorphized, olivine (Fo >80) with a rim of poorly crystalline Mg-silicate, amorphous alumina, amorphous metal oxides (Cr and Mn), and an Fe-bearing oxide phase which may include reduced iron nanoparticles. The major elements Mg, Si and Fe are present in CI-like relative proportions; Mg-normalized minor heavy elemental abundances show depletions in Ca and Ni, and enrichments in Cr, Mn, and Cu, relative to CI. The nature of the third candidate, Sample *I1003,1,40,0,0* ("Sorok"), is less certain. The capture track morphology was consistent with an interstellar grain, but no Fe, Mg, or Al were detected by SXRF. Either the grain was relatively Fe-poor compared to Orion and Hylabrook, or relatively little of the original projectile survived capture.

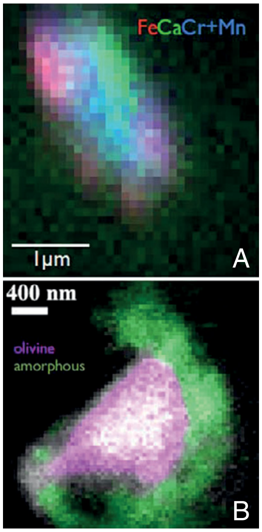

Fig. 4.11 ***Element maps of two of the first recognized probable ISPE grains derived from SXRF measurements.*** (A) Map of Sample I1043,1,30,0,0 (Orion). Blue is olivine, purple is spinel, and green is an unidentified Ca-bearing phase. (B) Map of I1047,1,34,0,0 (Hylabrook). Pink is olivine, and green is an amorphous phase. *(figure is after Westphal et al. 2014a)*.

A search for interstellar particle impact craters was also made on the Al metal foils surrounding the aerogel cells (Westphal et al. 2014a,b). Twenty-five crater-like features were identified during an automated scanning electron microscope-based search. Elemental analysis by Auger electron spectroscopy and/or energy dispersive X-ray spectroscopy (EDS) indicated that 21 of these features were secondaries from impacts on the spacecraft solar panels or defects in the foil. The remaining four impact craters (*I1044N,3*; *I1061N,3*; *I1061N,5*; and *I1061N,4*) have residues consistent with an extraterrestrial origin, consisting of Fe-, Mg-silicates, and/or Fe with associated S. Oxygen isotopic measurements of two of the crater residues were found to be consistent with solar system values, the remaining two craters could not be analyzed for O.

None of the compositional, isotopic, or mineralogic information from these samples (aerogel tracks or craters in Al foil) *requires* an interstellar origin. The strongest evidence for an interstellar origin comes from the directionality of the features. None of tracks for the interstellar candidates in the aerogel were in the angular range where IDPs should have their maximum flux. The ISPE track directions are slightly different from those expected from Ulysses and Galileo dust data, but a slightly shifting interstellar dust radiant hypothesized by Westphal et al. (2014a) would permit an interstellar dust origin for these tracks. Westphal et al. (2014a) used a statistical argument based on the expected flux of interplanetary dust vs. interstellar dust grains impacting the Al foils (from Landgraf et al. 1999) to similarly argue that all four of the potential Al craters mentioned above were most likely from interstellar impacts.

To summarize, several grains captured in ISPE aerogel and several residues found in craters in adjacent Al have been proposed to be of interstellar origin. However, no definitive evidence has yet been collected from any *Stardust* samples of an interstellar origin.

4.4 Conclusions

The *Stardust* mission was the first mission to bring back to Earth samples from outside the Earth-Moon system. The samples collected from the coma of Comet 81P/Wild 2 contained an enormous diversity of solar system materials in terms of elemental composition, mineralogy, organics, and isotopic structures. This diversity revolutionized our understanding of the processes and environments operant in the early protosolar nebula. Key points resulting from the study of the returned materials include:

- Comets clearly do not consist solely of presolar materials. Indeed, isotopically anomalous presolar grains are rare in Wild 2 samples for >μm solid grains. Many of the materials in the returned samples show evidence for high temperature formation in the protosolar nebula and are similar to the materials found in primitive meteorites.
- The returned comet dust is primitive. The returned materials do not appear to have been significantly altered after their incorporation into the comet. This has preserved a heterogeneity that demonstrates that these materials come from a wide variety of formation environments and have different detailed histories. The comet silicates seem to represent a more diverse sampling of nebular environments than seen in specific meteorite groups, unlike chondrite groups whose defining properties partially relate to regional differences in their source regions. Comets are likely to contain a broader mix of materials from nebular environments dispersed in both time and space.
- Large scale mixing must have occurred in the early protosolar disk. While comets may live predominantly in the outer Solar System, the composition of Wild 2 samples suggests they contain significant amounts of material that formed or was altered much closer to the Sun. Indeed, nearly all of the collected particles >2 μm in size are high temperature materials that include CAIs, chondrule fragments, and condensates.
- The elemental composition, mineralogy, isotopic patterns, etc. of Wild 2 particles are similar, but not identical to, primitive meteorites and anhydrous IDPs. They cannot be related to any specific meteorite group, but contain components found in various groups.
- Organics are present but severely under-represented in the returned samples due to their collection at hypervelocity. Material like meteoritic IOM is present, but there is evidence that comets may contain an additional, less aromatic organic component.

It seems unlikely that identification of chondrule and CAI fragments, measurement of the abundance of isotopically anomalous pre-solar grains, and a quantitative and detailed understanding of the complex mix materials formed in numerous nebular materials could ever have been known without the laboratory study of returned comet samples

Acknowledgements

This work was supported by the National Aeronautics and Space Administration through the NASA Emerging Worlds Program under Cooperative Agreement Notice NNH17ZDA001N issued through the Science Mission Directorate.

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