UNDERSTANDING THE COMET WILD 2 MINERALOGY IN SAMPLES FROM THE STARDUST MISSION

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ABSTRACT

The status of the solid materials and mineralogical and petrological results of the Stardust mission to comet 81P/Wild 2 are presented. This mission became the first successful sample-return mission since the Apollo project. This time the challenges were much less related to the availability of state-of-the-art analytical capabilities. Still, dedicated tools had to be developed to manipulate the samples that were all firmly embedded in the tracks they made when decelerating in the silica aerogel tiles of the collector. The comet particles were loosely bonded aggregates that shed their grains along the entire length of these tracks. It appears that most of the original comet minerals survived but interactions of debris with melted aerogel occurred and new minerals were made, adding to the incredibly, and unanticipated, diversity of the comet minerals, including some that so far were know only in meteorites. The latter alone showed that transport distances in the solar nebula extended all the way out to beyond Pluto into the Kuiper Belt of icy, comet-like, bodies. The full extent of the scientific yield of this mission is still unknown, promising but stressing current models of the formation of solar systems.

INTRODUCTION

It was during July 1969 that the very first samples that were collected in situ on another body other than planet Earth became available for laboratory studies. Scientists at that time were familiar with the interrelationships of certain rock types being associated with particular geological processes that operated as a function of location and geological time. Once used to the absolute “cleanliness” because of the absolute lack of weathering at the lunar rocks surface by processes attributable to a combination of water and biological activity, the lunar samples were entirely recognizable as basalt, anorthosite, or granite, for example. The unique lunar signature of these rocks was the highly reduced nature of mineral compositions. Without diminishing this research, it is fair, in hindsight at least, to say that the challenges in understanding the lunar rocks were more of a technical nature than intellectual. That is, the real intellectual challenge was, and to some extent still is today, a full understanding of the formation and evolution of the lunar soil for which there is no terrestrial analog. The Apollo program sparked a surge in new analytical techniques that still exist today albeit significantly improved in spatial and mass resolution. The ensuing drive towards in situ analyses at the nanometer-scale continued and has given us today’s fantastic array of techniques to tackle the most challenging samples in our laboratories. It is therefore that the laboratory analyses of samples from comet 81P/Wild 2 collected during the fly-by of the Stardust mission are not really a technical challenge except as I will show for mission-specific constraints. In this case, we are challenged to understand the data itself.
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The collector on-board the Stardust mission captured thousands of particles ranging from tens of nanometers to hundreds of microns. The collector was returned to Earth where it landed safely in a Utah desert on January 15, 2006. Thus ended this technically perfectly executed DISCOVERY-class NASA mission and an international team of scientists [1] stood ready armed with the most sophisticated, state-of-the-art techniques to extract all possible kinds of information from the comet samples down to the atomic level. Here, I refer the reader to the papers reporting the preliminary findings in a special issue of *Science* Magazine, Volume 314, December 15, 2006, on bulk particle and individual grain compositions and physical properties, organic chemistry, and isotope chemistry. Even casual perusal of these papers, and those presented at the 38th Lunar and Planetary Science Conference (http://www.lpi.usra.edu/meetings/lpsc2007/pdf/program.pdf), will show there were no technical limitations in studying these materials from the first sample-return mission since the Apollo program. 

I will limit myself to the minerals in the comet particles. The techniques that were used included a wide range of electron-beam based techniques, synchrotron XRD, XRF and topography, micro-Raman spectroscopy, and micro-FTIR spectroscopy [1,2]. Most sample preparation techniques for each of these analyses are well established in the laboratories. At this point it should be mentioned that the NASA-JSC curator, who is advised by a panel that reviews each sample request, allocates all comet Wild 2 samples that are distributed for these laboratory analyses. This procedure is flexible to accommodate special sample preparation requests. Initial inspection of the collector showed that all collector tiles (Figure 1) were intact and literally riddled with tracks caused by comet particles impacting at 6.1 km/s. It also became clear that for these particular samples it would be necessary to refine an iterative process of sample handling and the analysis techniques to be applied.

Figure 1. The dust collector shown on the left is a gold foil-covered aluminum frame about 50 cm in diameter. Each rectangular space was fitted with a tile of under-dense silica aerogel. The returned tiles were all intact and contained hundreds of deceleration track including the huge track at top-center of the tile shown on the right. The tracks were made by impacting comet particles that were decelerated over a distance of generally less than 1 cm. Image credits: NASA/JSC (left) and Don Brownlee (Washington University, Seattle, Washington) (right). The image on the right graced the cover of *Science* Magazine, Volume 314, December 15, 2006.

The especially developed, under-dense silica aerogel tiles that would allow the intact capture of individually impacted comet particles dominated the design of the entire exposed collector surface as it is shown in Figure 1. But of course the Al-frame itself inadvertently became a dust collector. Instead of producing a declaration track, each particle that impacted these surfaces...
produced a unique crater (Figure 2) but only a very small fraction of Wild 2 grains survived in the craters that contain mostly quenched residues of melted particles.

Figure 2. Scanning electron microscope image of a crater in the aluminum frame of the Stardust collector caused by the impact of a comet Wild 2 particle. The crater diameter is 140 µm. By using hypervelocity impact calibration experiments it is possible to reconstruct particle size/mass from crater dimensions and morphology ([see reference [3]]. Residual debris from the comet particle is visible at the bottom and on the crater rim. Image courtesy: Stadermann at Washington University, Saint Louis, Missouri.

During the fly-by of comet Wild 2, the onboard Dust Flux Monitor Experiment found that the particles ranged from $10^{-14}$ to $10^{-7}$ kg, and that they arrived in “intense swarms of particles” that are best explained as evidence for fragmentation of initially larger particles that were ejected from the nucleus [4] (Figure 3). From this experiment it was estimated that the aerogel tiles should contain $2800\pm500$ particles of 15 µm or larger [4]. Analyses of deceleration track dimensions and shapes and of crater morphologies and dimensions showed that individual comet particles ranged from dense mineral grains to loosely bound polymineralic aggregates ranging from tens of nanometers to hundreds of microns in size, and most of the collected dust mass was contained in just a few particles >100 µm [3]. Yet, there are thousands of individual deceleration tracks and craters. From detailed analyses of a small number of available tracks it was concluded that “The bulk of the comet 81P/Wild 2 (hereafter Wild 2) samples returned to Earth by the Stardust spacecraft appear to be weakly constructed mixtures of nanometer-scale grains, with occasional much larger (over 1 micrometer) ferromagnesian silicates, Fe-Ni sulfides, Fe-Ni metal, and accessory phases” [5].

Figure 3. Composite image of the nucleus of comet 81P/Wild 2 (B&W), which is $5.5 \times 4.0 \times 3.3$ km. Its bulk density is 0.3 to 0.4 g/cc. Particles were ejected in individual dust jets shown as the light streaks projecting outwards from the coma. The Stardust mission collected some of the ejected particles at a distance of $235\pm1$ km. Image courtesy: NASA JSC/JPL.

SILICA AEROGEL AND DECELERATION TRACKS

The under-dense, micro-porous (10 nm) aerogel was a rigid three-dimensional network of nanometer-sized SiO$_2$ clusters linked together forming chains. It had a high surface area (1000 m$^2$/g) and very low thermal conductivity (~0.02 W/mK) making it a perfect thermal insulator. It was designed for intact capture of particles impacting at 6.1 km/s. Initial inspection of the tracks
caused by impacting comet particles showed that intact capture had not occurred. Based on a small number of hypervelocity impact experiments using mineral or composite-mineral projectiles that were shot into aerogel [3], complete intact capture was not expected. What happened was that (1) original particle aggregate structures were destroyed, (2) many mineral grains were fractured severely and in some cases with aerogel penetrating along fractures, (3) grains were encapsulated in aerogel, (4) grains were melted, and (5) numerous nanometer grains were dispersed in silica glass that represents melted aerogel [1,5–7].

Particles impacting the under dense aerogel each carved its own individual deceleration track. Track morphology is a first-order indicator of physical characteristics of the comet particle [1,3]. That is, a thin, slender track indicates a particle that was a compact mineral grain with perhaps a few attached smaller particles or ultrafine-grained aggregate material. In general, extensive particle fragmentation led to substantial lateral tracks off of the main track. Most tracks have a bulbous cavity with, or without (rare), a single stylus or two bifurcating stylii (Figure 4). In a typical track most grains of the original particle were deposited near the entry hole, but with hundreds of grains distributed along the entire track including the stylus (Figure 4). Track lengths ranged from 50 \( \mu \text{m} \) to about 1 cm measured from the penetration hole to the end of the stylus; most track lengths are in the millimeter range.

Most track features suggested that the comet particles were weakly constructed mixtures of nanometer-size grains with occasional grains, \( \sim 1 \) to \( \sim 10 \ \mu \text{m} \), and terminal grains up to 20 \( \mu \text{m} \) at the end of a stylus. In such cases the original comet particle could be envisioned as one or a few large grains (\( > 10 \ \mu \text{m} \)) covered and held together by ultrafine aggregate material that might be a mixture that included micron-sized mineral grains. Of course this hypothetic original texture of the initial particle was completely lost when during deceleration the micrometer and nanometer
grains were gradually but continuously stripped off leaving a “naked” terminal particle. This type of aggregate particle is found among interplanetary dust particles (IDPs) that were collected in the Earth’s lower stratosphere (Figure 5). This typical aggregate IDP contains numerous nanometer scale Mg,Fe-rich silica grains and a few 1 to 2 \( \mu \text{m} \) grains that can be silicate minerals, Fe-sulfides, or amorphous aluminosilica grains. The “tail” of the sulfide IDP consists of fine-grained material similar to the matrix of aggregate IDPs. These aggregate IDPs and the larger micrometer cluster-IDPs that are random mixtures of such aggregate IDPs, sulfide IDPs (as shown), and similar-sized silicate IDPs (not shown) [8,9] are presently the best available natural analogs representing comet Wild 2 particles. The sulfide IDP experienced flash heating when decelerating in the Earth’s atmosphere followed by ultra-fast cooling that weakened its crystalline structure (Figure 5). It caused the shattering during ultramicrotome sectioning. Many of the collected comet grains show a similar structure in the transmission electron microscope.

Figure 5. Two interplanetary dust particle (IDPs): on the left a scanning electron microscope image of an aggregate IDP ~12 \( \mu \text{m} \) in diameter (W7029B13; NASA number S-82-27575; shown courtesy of NASA/JSC) on a nucleopore-filter (background), and on the right a transmission electron microscope image of an ultramicrotome thin section from a sulfide IDP ~10 \( \mu \text{m} \) in diameter (black) that was embedded in an epoxy (gray background) and placed on a holey carbon thin film substrate. White areas show where sample material was lost (unpublished data by F. J. M. Rietmeijer).

The origin of the cavity near at the entry hole is not entirely clear. It was suggested that it was caused by the sudden release of copious volatiles from a volatile-rich particle. This interpretation is consistent with the observations on aggregate IDPs that are rich in carbon materials that include poorly graphitized carbons, pure amorphous carbons, organic C-O carbon, and volatile hydrocarbons [8,10]. It remains yet to be calibrated experimentally what was the extent of thermal interactions between a comet Wild 2 particle and the under-dense silica aerogel during hypervelocity impact. At this time we have to rely on modeling studies that considered a much higher aerogel density than that of the under-dense aerogel that was actually used. These studies predicted temperatures up to 10,000 K [3], which are almost certainly too high. The actual temperatures reached were probably half this value (personal communication with Bill Anderson, Los Alamos National Laboratory), but still sufficiently high to cause melting and evaporation of silica aerogel. Some of the silica vapor generated at the earliest stage of particle deceleration may have escaped into space through the penetration hole but most of it probably pushed a thin layer of silica melt into the aerogel thereby causing (localized?) the compaction of un-melted aerogel. That is, the bulbous part of the tracks could be an “explosion cavity” caused by silica vapor and an as yet unknown amount of indigenous volatile comet species.
It appears the aerogel performed admirably in absorbing and dissipating most of the kinetic energy of the comet particles. The majority of the generally big terminal grains (Figure 6), and smaller (1 to 5 µm) grains scattered along the entire track walls, survived with little evidence for melting or interactions among these grains with melted aerogel. Most of these grains were heavily shattered, and there is evidence that silica aerogel had penetrated these cracks in some of the shattered mineral grains. The olivine grain (Figure 6) shows a distinctive narrow rim that was identified as silica-rich quenched melt with multiple embedded electron-opaque inclusions. The boundary between such rims and the mineral grain are typically knife-sharp. There is no evidence for mixing of these compositionally different materials. In fact, one of the most astonishing, at least to me, and predominant feature of this particular Stardust capture technique is the apparent lack of chemical exchange between comet Wild 2 particles and the silica aerogel capture cells down to the nanometer level [1,5–7].

![Figure 6. High-resolution TEM images of terminal grains extracted from the stylus of two different tracks in aerogel. On the left is a pyrrhotite grain with the typical shattered appearance of many collected grains (photo credit: LLNL/JPL/NASA JSC). Many terminal grains have single-crystal electron diffraction patterns indicating that the original crystalline properties survived, although the thermal pulse and rapid cooling associated with the capture process had structurally weakened the grains. On the right is a Mg-rich olivine crystal with the same shattered artifact. Its narrow rim is Si-rich glass (light gray) with many nanometer Fe-Ni-S inclusions. Such rims occur on many grains (photo credit: NASA JSC).](image)

**TERMINAL GRAINS**

The terminal grains at the very end of the deceleration track carved by an impacted Wild 2 particle range from ~5 to 20 µm. They included [1,5,11]

1. Mg-rich, single mineral grains of (Mg, Fe)-olivine [(Mg,Fe)2SiO4] (Figure 6), often containing MnO and Cr2O3 each up to 1 wt%, and low-Ca (Mg, Fe)-pyroxene [(Mg,Fe)SiO3],
2. Composite grains of about equal amounts of these two minerals,
3. Mainly Fe,Ni-sulfide grains (nominally FeS) (Figure 6), including an 8 µm composite grain that was a large FeS crystal, a small low-Ca pyroxene, and a patch of fine-grained chondritic aggregate material,
4. Fe,Ni metal grains,
5. One unique polymineralic refractory grain that is an ensemble of several minerals, viz. Ti-rich clinopyroxene [Ca(Mg,Ti,Al)(Si,Al)2O6], anorthite [CaAlSi2O8], gehlenite [Ca2Al2SiO7], corundum [Al2O3], V-bearing osbornite [(Ti,V)N] as sub-100-nm-sized grains within spinel [MgAl2O4], perovskite [CaTiO3], and FeS, and
6. An alkali-rich composite grain of Na,K-bearing silicates that was a sub- to micron grain composite of roedderite \([(Na,K)_{2}(Mg,Fe)_{5}Si_{12}O_{30}]\), richterite (an amphibole mineral) but no OH reported yet in this Wild 2 mineral, Na- and K-bearing silica glass, Mg-pyroxene, and FeS \([1,12]\).

The comet Wild 2 olivine, pyroxene, and sulfide compositions are similar to those common to aggregate and cluster IDPs. The range of molar ratios, Mg/(Mg+Fe), for olivine and (low-Ca to Ca-free) pyroxenes from 1 to 0.52 in the comet is identical to the compositional ranges for these minerals in IDPs. The comet olivine composition of Mg/(Mg+Fe) = 0.04, which is almost pure fayalite \([Fe_{2}SiO_{4}]\), has no counterpart among the IDPs. The comet particle with a FeS crystal and fine-grained chondritic aggregate material has its analog among these IDPs (Figure 5) \([13]\), and suggests that the nanometer grains in the comet \([5]\) might resemble the materials in the matrix of aggregate IDPs (see Figure 5). The FeNi grains in the comet are also represented among the IDPs \([8,13,14]\). So far, refractory and alkali-rich terminal particles are rare and much less abundant than the common olivine, pyroxenes and Fe,Ni-sulfides. This also compares to the database for aggregate and cluster IDPs \([8,9]\) with only two highly refractory IDPs \([15]\), but alkali-rich IDPs have yet to be found among these particles (see reference \([8]\)). The alkali-rich silicates in a comet are reminiscent of rare hyper-alkaline chondrules that are common quenched-liquid droplets, but of uncertain origin found in all ordinary chondrite meteorites (see reference \([16]\)), that make up about 85% of all meteorites. Chondrule formation almost certainly involved thermal processing of solar nebula dust and modification in asteroids.

We do not know the origins of aggregate and cluster IDPs, but they are completely different from any meteorites in our collections, and their aggregate textures suggest that they are as close as we can get to the dust particles that accreted in the solar nebula 4.56 Gyrs ago to form comet nuclei, asteroids, and planets. It is reassuring that the comet Wild 2 particles do not contain new minerals. However, the association of these minerals in the terminal grains, and probably also the smaller grains scattered along the tracks, in this small mass of Wild 2 debris, raises some interesting challenges. Submicron up to few micrometer amorphous aluminosilica grains with Al/(Al+Si) (element) ranging form ~0.5 to ~0.95 with minor amounts of Fe, Mg, Ca, and Al in variable relative proportions are present in aggregate IDPs \([8]\). These amorphous grains are considered to be indigenous phases that represent the least-processed solar nebula dust \([9]\). Chemically similar amorphous grains in comet Wild 2 particles could be the confirmation that this interpretation of the IDP phases was correct. Despite the fact that most of the micron-size minerals in the comet survived capture without melting, it remains to be shown that all initially crystalline aluminosilica-rich minerals survived intact. That is, grains with this particular composition are not the result of comet grains interacting with a silica-melt.

Iron silicide spheres <100 nm near the entry hole of deceleration tracks showed that submicron FeS grains from the comet had reacted with the silica melt during hypervelocity impact capture. The compositions of these quenched-melt silicide spheres ranged from hapkeite (Fe$_2$Si) to suessite (Fe$_3$Si) and to Fe$_7$Si$_2$ \([7]\). So although most of the micrometer grains in the weakly constructed mixtures that were the Wild 2 particles \([5]\) can be retrieved from the aerogel capture cells, the same does not seem to be true for the most abundant of nanometer-scale grains in these particles.
NANOMETER COMET DUST IN AEROGEL

During hypervelocity impact a sheet of silica melt was pushed into the ever-widening bulbous part of a deceleration track. This melt can be extracted as rather non-descript irregular porous glass beads typically larger than \(~5\ \mu m\) but not much bigger than \(~20\ \mu m\) (Figure 7). Seen in microtome sections of \(~70\) to \(80\) nm thick, the glass matrix can be massive or have vesicles of widely ranging size and highly variable spatial distributions along with numerous, typically spherical, electron opaque inclusions ranging from a few nanometers up to about \(150\) nm (Figure 7). The larger inclusions tend to have sub- to euhedral shapes.

![Image](C2004_1_44.1)

**Figure 7.** On the left is the scanning electron microscope image of an irregular clump of quenched silica-rich melt extracted from the wall of aerogel track #41 (modified after reference [7]). The transmission electron microscope image on the right (scale bar is 500 nm) is an ultramicrotomed section through a Si-rich glass clump. It shows the shard-like fracture behavior and the random distributions of vesicles of variable dimensions and randomly scattered electron opaque, spherical Fe-Ni-S inclusions (reproduced from reference [6]). This Si-rich glass is also present as partial rims on mineral grains (see Figure 6).

The inclusions are either a Fe-Ni-S phase or Fe-Ni metal. The metal compositions range from \(\text{Ni}/(\text{Fe+Ni})\) (atomic) zero to \(~0.55\) [6], and energy filtered TEM maps show that some fraction of these metal grains has an Fe-S rim often just a few nanometers wide (Figure 8)—they are mostly kamacite. The Fe-Ni-S grain compositions are more complex. In general they are either Ni-free or contain a few atomic percent Ni. The sulfur contents seem to be a complete range from pyrrhotite \((\text{Fe}_7\text{S}_8)\) and troilite \((\text{FeS})\) (or, nominally FeS, [5]), with \(S = 50\) at% to almost pure low-Ni or pure Fe-metal. The diffraction data show they are a mixture of kamacite and Fe-sulfide, probably a pyrrhotite phase [6]. There is some evidence that in reality the range may not be continuous but favors very low \(S\) FeS \(~2\) at% \(S\) and FeS with \(~15\) at% \(S\) and close to stoichiometric FeS [7]. With exceptions among the smallest Fe-Ni-S inclusions, they all have a core-mantle structure whereby the core has a lower S-content than the mantle. Furthermore, the mantle is either a well-defined rim but with highly variable thickness relative to the core diameter or a highly irregular structure such as a tail-like feature [6]. The irregular rim features suggest that the original Fe-Ni-S comet grain was freely moving through the silica melt. If so, it further suggests that “cold” original grains penetrated into an already hot aerogel wherein they were heated (perhaps even melted) and subsequently had enough time to develop a core-mantle structure. Their uniformly nanometer sizes could be a clue that such a scenario was possible as a function of the time-temperature path in hot aerogel that is in fact an almost perfect thermal insulator. It will require much more theoretical modeling and laboratory experiments to constrain this scenario, if it is the correct one, quantitatively.
Also included in the silica glass matrix are scattered patches of amorphous Mg-Ca-Al-rich materials patches and single-crystal olivine and pyroxene grains that are typically ~500 nm or smaller [6]. The mineral grains are often surrounded by a partial rim of silica or a silica-rich glass [6]. The database is still insufficiently rigorous but partial rims of Si-rich glass with opaque inclusions appear also to be favored on micrometer grains such as terminal grains (see Figure 6).

CHALLENGES AHEAD

The currently available laboratory instruments are more than adequate to extract every tiny bit of information present in the particles from comet Wild 2 that were delivered to Earth by the Stardust mission. Currently most analyses are preformed on “keystones,” which are slices of aerogel containing an entire track. The technique to extract a keystone from an aerogel tile was especially developed to handle Stardust samples [17]. In addition to ultramicrotome sections, analyses are also preformed on grains still embedded in aerogel. Many grains for mineralogical studies are embedded in an epoxy at the NASA/JSC Stardust Curatorial Facility that can be “polished” or sequentially cut in ultra-thin sections. The exact location of each sample, or allocation, is documented and available for future retrieval, which is a monumental task.

So far most of the Wild 2 grains found have their analogs among the aggregate and cluster IDPs that were considered our best analogs for cometary dust. Even the refractory terminal grain was among the IDPs. There are of course preconceived notions about the properties of comet and asteroid dust. For example, a cluster IDP with large anorthite grains was interpreted as asteroidal debris [18] but comet Wild 2 shows that this mineral also occurs in comets. A major intellectual challenge will involve a new assessment of what is comet dust and what is asteroidal dust, or perhaps there are no firm distinctions. If so, we have to conclude that transport distances in the solar nebula were much larger than was heretofore predicted by the models, which is indeed a favored notion. In this X-wind model, dust is ejected from the solar nebula mid-plane through its bi-polar outflows and then thrown back into the nebula at great heliocentric distance in the region of the Kuiper belt [1] (Comet Wild 2 is from this belt). Before, we can be entirely sure, many more grains need to be extracted from the aerogel tiles and studied in detail. It will be necessary to determine the extent of comet dust modification, which could well be a function of the original grain size and composition. Future analyses will have to determine how bizarre—at
least for a comet—some of the terminal grain compositions, e.g. the alkali-rich grain, really are
or did we just happen to extract the only one. The status currently is that the analyses have barely
scratched the tip of the iceberg that is a comet nucleus.

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