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Fine-grained material of 81P/Wild 2 in interaction with the Stardust aerogel

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Abstract–The deceleration tracks in the Stardust aerogel display a wide range of morphologies, which reveal a large diversity of incoming particles from comet 81P/Wild 2. If the large and dense mineral grains survived the extreme conditions of hypervelocity capture, this was not the case for the fine-grained material that is found strongly damaged within the aerogel. Due to their low mechanical strength, these assemblages were disaggregated, dispersed, and flash melted in the aerogel in walls of bulbous deceleration tracks. Their petrologic and mineralogical properties are found significantly modified by the flash heating of the capture. Originating from a quenched melt mixture of comet material and aerogel, the representative microstructure consists of silica-rich glassy clumps containing Fe-Ni-S inclusions, vesicles and "dust-rich" patches, the latter being remnants of individual silicate components of the impacting aggregate. The average composition of these melted particle fragments is close to the chondritic CI composition. They might originate from ultrafine-grained primitive components comparable to those found in chondritic porous IDPs. Capture effects in aerogel and associated sample biases are discussed in terms of size, chemical and mineralogical properties of the grains. These properties are essential for the grain survival in the extremely hot environment of hypervelocity impact capture in aerogel, and thus for inferring the correct properties of Wild 2 material.

INTRODUCTION

In 2006, samples from comet 81P/Wild 2 were returned to Earth by the Stardust mission (Brownlee et al. 2006). With a relative velocity of 6 km s^{-1}, cometary grains have been captured in the coma of the comet through a dust collector made of low-density silica aerogel cells. The mission was a success because a large number of grains were collected, as illustrated by the numerous deceleration tracks in the aerogel. Optical examination of tracks has highlighted three types of morphologies, demonstrating a high diversity of impactor properties, from highly porous and friable aggregates to well consolidated monomineralic grains (Hörz et al. 2006; Burchell et al. 2008). Type A tracks are carrot-like with a terminal particle at the end. They are created by cohesive, dense mineral grains. Type B tracks have bulbous cavities from which one or several small secondary tracks emerge. They correspond to the impact of poorly cohesive polymineralic aggregates that disaggregated in the aerogel during the deceleration. The slender tracks emanating from the bulb correspond to dense minerals larger than a few micrometers that were present in the aggregates as nuggets. Type C tracks are only bulbous, suggesting that the impacting particles consisted only of a poorly cohesive fine-grained material. At the present time, the most studied grains are the terminal particles. They correspond most frequently to high-temperature minerals, including pieces of chondrule-like and CAI-like particles (Zolensky et al. 2006, 2008; Leroux et al. 2008a; Nakamura et al. 2008; Simon et al. 2008; Tomeoka et al. 2008; Chi et al. 2009; Jacob et al. 2009; Joswiak et al. 2009). Cometary material is also distributed along the track walls of the bulbs in the case of type B or C morphologies. This is well illustrated by synchrotron X-ray fluorescence and X-ray microtomography maps performed on entire tracks (Flynn et al. 2006; Ishii et al. 2008a; Lanzirotti et al. 2008; Ebel et al. 2009) or by transmission electron microscopy studies of particles extracted from aerogel (e.g., Leroux et al. 2008b; Rietmeijer 2009a; Stodolna et al. 2009; Velbel and Harvey 2009). The distribution of cometary material along the walls shows that many of cometary particles were fragmented during deceleration in aerogel, and thus lost their original architecture. In addition to this physical modification, heating effects have strongly modified the pristine microstructure of the grains.

Evidence for fine-grained aggregates from Wild 2 is also provided by studies of impact craters on aluminum foils of the Stardust dust collector (Hörz et al. 2006; Kearsley et al. 2008, 2009). These studies showed a wide diversity of crater sizes and shapes. As for the aerogel tracks, impact features on Al foils revealed Wild 2 dust particles dominated either by relatively large single mineral grains or by low-density aggregates with submicrometer-sized grains with an average composition close to that of CI chondrites (Kearsley et al. 2008).

The aim of this study was to describe the changes induced by collection in aerogel at the transmission electron microscopy scale. We first recall the essential parameters involved in a hypervelocity impact of a particle in aerogel. We then describe the consequences of the interaction of particles with aerogel. This includes thermal ablation, fragmentation, thermal effects on microstructure, and mixing with the aerogel at high temperature. We focus especially on the poorly cohesive aggregates of submicron grains of Wild 2, now present as fragments in the walls of bulbs. Then we discuss possible biases induced by the capture on the mineralogy, chemical composition, and size of the cometary grains. The study is based on analytical transmission electron microscopy (ATEM) investigations. Analytical methods are given in Leroux et al. (2008b).

IMPACT IN AEROGEL

Silica aerogel is made of long chains of nanometer-sized clusters of amorphous silica. Chains are interconnected, making a three-dimensional network with an open system of nano-pores (e.g., Fricke and Tillotson 1997). Ultra low-density silica aerogel has been successfully used as a dust collector in space (Tsou et al. 1988; see Burchell et al. [2006] for a recent review). Its role is to gradually slow down the dust to minimize its alteration during capture. Due to its low density, the dust projectiles penetrate deep inside the aerogel and dissipate the initial kinetic energy over a relatively long period of time (typically a few microseconds) compared with the impact on a dense target (typically a few tenths of nanoseconds). Aerogel is also chosen as a capture medium because of its relatively easy manufacture and its transparency, allowing easy location of the captured particles and further extraction for detailed characterization. For the Stardust mission, the relative velocity between the spacecraft and the coma was 6 km s^{-1}. With a density 0.005 g cm^{-3} at the front face and 0.05 g cm^{-3} at the rear face of each cell, the aerogel was designed to stop the particles in about one centimeter (Tsou et al. 2003).

Numerous efforts were made to estimate the conditions of impact in aerogel. They include modeling (e.g., Anderson and Ahrens 1994; Trucano and Grady 1995; Anderson 1998; Dominguez et al. 2004; Trigo-Rodríguez et al. 2008; Coulson 2009; Dominguez 2009) and experimental approaches (e.g., Barrett et al. 1992; Hörz et al. 1998; Burchell et al. 2001, 2008; Noguchi et al. 2007; Hörz et al. 2009; Stodolna et al. 2011). According to Anderson and Ahrens (1994), the impacting particles are much larger than the domain size of clusters and pores of the aerogel (both are nanometer scaled). The aerogel can thus be considered a continuous medium, like a fluid, in which the particles entered at high velocity (Coulson 2009). This situation is not fundamentally different from that of particles entering the Earth's atmosphere. The dust grains deposit their kinetic energy in a few microseconds on a distance of a few millimeters to one centimeter. The resulting macroscopic effect is the formation of clearly visible penetration tracks that are useful to locate the projectiles and fragments of projectiles. The exact conditions of pressure and temperature experienced by the aerogel and particles are not fully understood. At the impact point, a shock wave is generated in the aerogel leading to compression and heating. The shock wave propagates at the front of the path of the particle. The shock temperature of the aerogel is estimated within the range 10,000–20,000 K (Anderson 1998; Coulson 2009). However, the peak temperature in the impacting particle is one order of magnitude lower (Coulson 2009). The shock pressure is estimated between 0.1 and 1 GPa (Dominguez et al. 2004; Trigo-Rodríguez et al. 2008; Coulson 2009). The kinetic energy of the incident particle is then partly dissipated by compaction and heating of the aerogel. The incident particle also undergoes temperature and pressure increases. These capture conditions are sufficient to induce permanent local change in the structure and composition of the Wild 2 particles. The main effects are thermal ablation, physical disaggregation, and thermal modification such as melting. Both effects are discussed in the following paragraphs.

THERMAL REGIME DURING THE HYPERVELOCITY IMPACT IN AEROGEL

Collision of particles in the hypervelocity regime leads to extreme shock temperatures in aerogel (approximately 10,000 K at 6 km s^{-1}). Thus, it can be considered that particles entered into hot aerogel and were flash heated. Particles (that can be fragmented), in contact with the aerogel, may undergo thermal decomposition (melting and volatilization) and mixing with volatilized and melted aerogel. Due to the short duration of the shock process, the high-temperature transformations affect only the projectile surface. The interior of relatively large clasts can be well preserved from heating, but fine-grained aggregates dispersed into a fine fraction, up to individual components, can be totally consumed when exposed to the hot aerogel (Brownlee et al. 2006). If the interiors of the large grains (typically larger than 2 μm) are well preserved, surface heating leads to modification of particle size and shape. From the first use of aerogel to capture hypervelocity particles, it was noted that during the capture process, the captured grains had lost mass to the aerogel, and had gained a shell of densified aerogel (Bunch et al. 1991; Barrett et al. 1992; Hörz et al. 1998; Burchell et al. 1999, 2001). This mass loss is now interpreted as the consequence of thermal ablation due to friction with the aerogel during the implantation of the particle, which adds fragmentation for grains that are not mechanically resistant. Along the grains' trajectory, some matter has been left behind in the form of molecules or small condensed droplets of melt and vapor from the heated surfaces. The distribution of fragments along tracks is nicely demonstrated by Nakamura-Messenger et al. (2011) who developed a sample preparation method for entire aerogel tracks. Fine-grained debris is found even for robust particles that generate carrot-shaped tracks. This erosion leads to a particle size reduction, and also to a change of shape, with bumps that tend to be eroded by friction. Indeed, a number of Stardust grains have relatively rounded shapes (Fig. 1a). The removal of matter from the surfaces provides an almost immediate evacuation of the heat from grains (Anderson and Ahrens 1994). Therefore, the cores of the grains remain cold and well preserved from the capture effects. However, a melted rim is frequently observed around coarse grains (Fig. 1b), both for experimental impacts (Bunch et al. 1991; Barrett et al. 1992; Noguchi et al. 2007; Hörz et al. 2009; Stodolna et al. 2011) and Stardust samples (Tomeoka et al. 2008; Jacob et al. 2009; Joswiak et al. 2009). This rim is highly enriched in SiO_2, suggesting that the grain accreted melted aerogel. This accretion phenomenon reduces the ablation efficiency by protecting the particles for further friction during the travel of the projectile (Barrett et al. 1992). The relative importance of each effect (ablation and accretion) seems to be largely unknown. This applies, of course, for relatively large grains (several microns in diameter). For small or fragile particles, thermal effects can affect the whole grain, causing complete evaporation or melting.

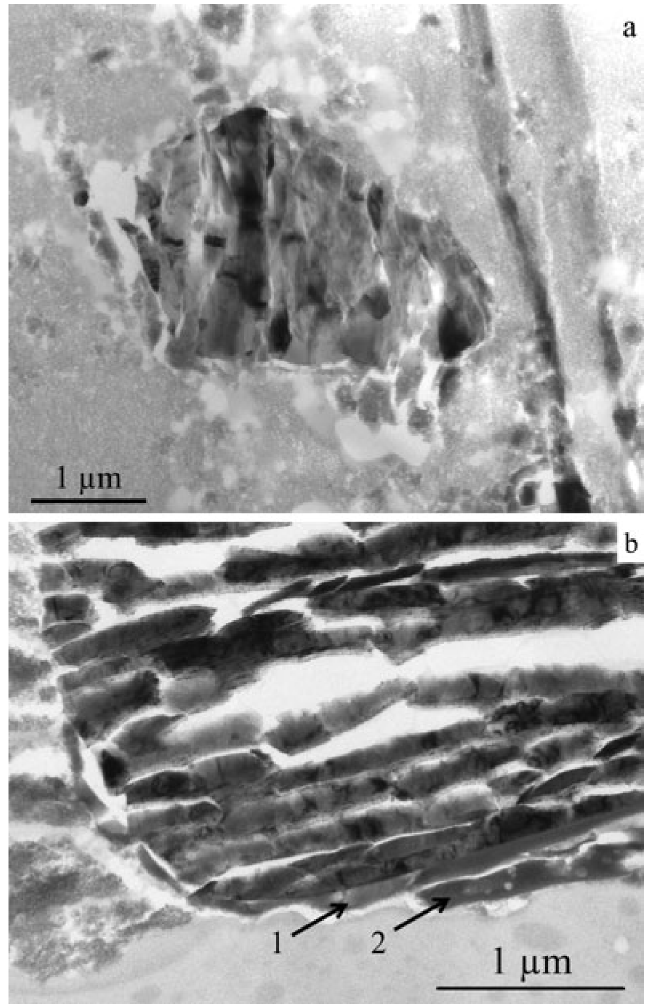

Fig. 1. Transmission electron microscopy bright-field images. a) Rounded forsterite (Fo_{100}) grain within the aerogel. Sample C2009,20,77,5,17. b) Interface between aerogel and a large single crystal Al-Ca-Mg-pyroxene terminal particle. The interface is composed of two layers. Layer 1 (arrowed) is a dense glass slightly enriched in SiO_2 compared with the pyroxene composition. Layer 2 (arrowed) is almost pure SiO_2 glass with only minor Al, Ca, and Mg elements. Sample C2017,2,99,1,12.

FRAGMENTATION OF AGGREGATE PARTICLES

In the wall of bulbs, the presence of abundant cometary material is clearly established, originating from ablation, and also from particles, which disaggregated into a large number of fragments during impact (Flynn et al. 2006; Hörz et al. 2006; Zolensky et al. 2006; Ishii et al. 2008a; Lanzirotti et al. 2008). Ablation products probably dominate in type A tracks, but type B and C tracks contain many discrete and small grains in the cavities, illustrating that the incident particles were severely disrupted. The fragments probably originated from very fine-grained materials weakly bonded to one

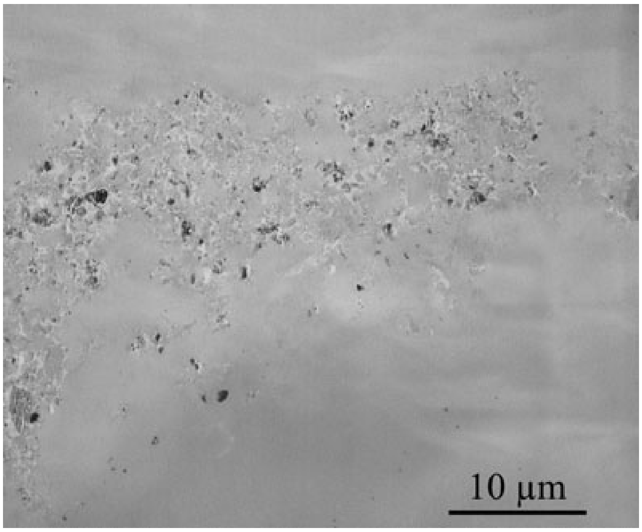

Fig. 2. Low-magnification bright field TEM image showing a large number of fragments dispersed into the aerogel (dark dots in the bright compressed aerogel). The sample has been prepared by the compression method (Matrajt and Brownlee 2006), which allows a good preservation of the fragments encased in the aerogel, whatever their size. Most of the fragments consist of silica-rich glassy objects, but some crystalline silicates are still present. Sample C2009,20,77,2,36.

another. Figure 2 is a low-magnification TEM image showing a large number of fragments within a piece of aerogel from track 77. The impacting particle was likely a loose aggregate, which disaggregated into individual components during the impact. Frequent styli emerging from the bulb show that a number of particles contained relatively large and dense crystalline nuggets stuck together with the fine-grained material (Hörz et al. 2006; Burchell et al. 2008). These coarse grains then can generate radial secondary small tracks containing discrete subgrains of the impactor.

The particle scattering in the bulbs shows that the dynamic pressure on particles during the deceleration in aerogel is large enough to induce disaggregation of fine-grained and poorly cohesive assemblages. This is not surprising because the tensile strength of cometary meteoroids has been estimated to be 0.1 MPa (Blum et al. 2006; Trigo-Rodríguez and Blum 2009). This is well below the dynamic pressure that affected the particle during impact at 6.1 km s^{-1} into the Stardust aerogel (range 0.1–1 GPa).

The study of elemental abundances of whole tracks by synchrotron X-ray fluorescence revealed that the terminal grains have different relative elemental compositions compared with the material distributed along the tracks (Flynn et al. 2006; Ishii et al. 2008a; Lanzirotti et al. 2008). The same authors also observed that subareas within the track's walls are chemically heterogeneous, but the mean composition is consistent with that of CI chondrites. This fine-grained material is probably quite primitive, in contrast to terminal particles. Unfortunately, its detailed study is difficult as they have been thermally modified during impact.

HEATING EFFECTS ON THE FINE GRAINED MATERIAL

As seen above, thermal effects are clearly observed on coarse particle terminals, evidenced by a frequently melted rim around the grains. The situation is much more complex for the fine-grained material, which disaggregated in bulbs. Indeed, the grain size is much smaller, thus the grains are more exposed to the flash heating process. Second, the assemblages are likely very heterogeneous, containing a mixture of volatile and refractory material. Very little experimental work has been performed to study the consequence of the impact of poorly cohesive polymineralic fine-grained assemblages in aerogel. Indeed, this material cannot generally survive acceleration in light-gas guns. Most of the information comes directly from Stardust samples collected from the bulbs. Given the small grain size, analytical transmission electron microscopy provides observations, which are very valuable. Several studies have already described in detail the microstructure of the cometary material in the bulb (Keller and Messenger 2008; Leroux et al. 2008b, 2009; Rietmeijer et al. 2008; Roskosz et al. 2008; Tomeoka et al. 2008; Rietmeijer 2009a, 2009b; Stodolna et al. 2009; Velbel and Harvey 2009). This fine-grained material is found to be heavily thermally modified and transformed due to the interaction with the aerogel. Its typical microstructure consists of a dense silica-rich glassy matrix containing numerous Fe-Ni-S nanophases (Fig. 3a). The size of these opaque inclusions ranges from a few nanometers to one hundred nanometers in diameter. They have a frequent metallic core and sulfide shell (Zolensky et al. 2006; Ishii et al. 2008b; Leroux et al. 2008b). The high concentration of silica in the glassy matrix clearly shows that the components were melted and mixed with molten aerogel. The initial microstructure of the fine-grained material is thus destroyed by the impact. The widespread occurrence of Fe-Ni-S nanophases within the quenched glass has been interpreted by two processes, which probably occurred simultaneously. The first process corresponds to the thermal decomposition of iron-sulfide grains, followed by dispersal of droplets within the impact melt (Ishii et al. 2008b; Leroux et al. 2009). The second process implies reduction of ferromagnesian silicates while they are in a high-temperature molten state, due to the presence of a reducing agent in the particle assemblage or within the aerogel itself (Leroux et al. 2009).

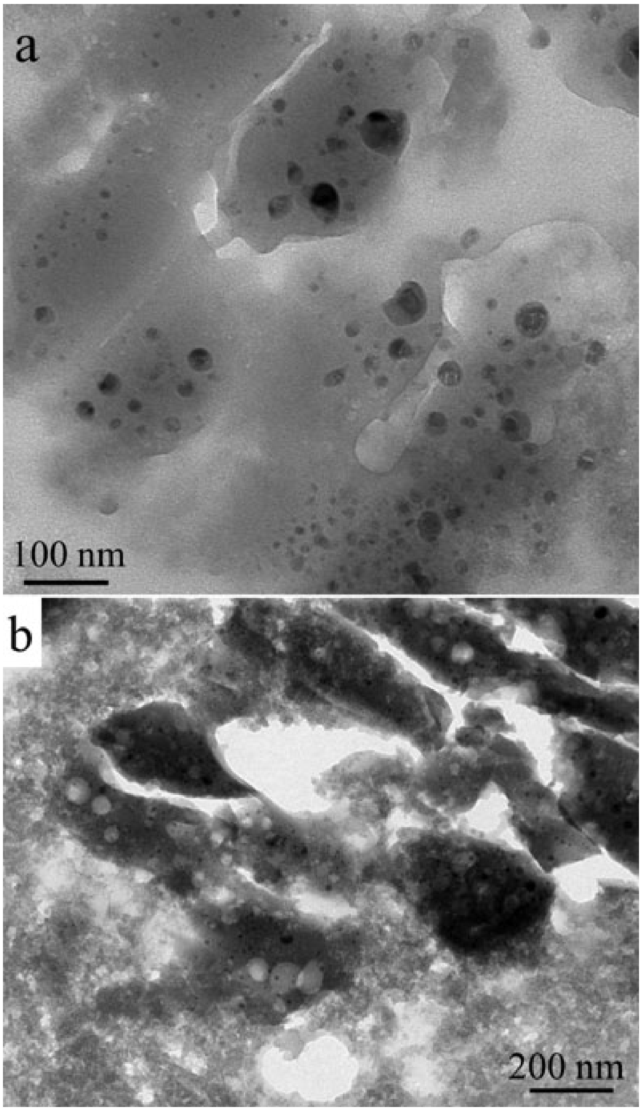

Fig. 3. Bright-field TEM images. a) Dense silica-rich glassy matrix containing Fe-Ni-S nanophases (dark dots). Sample C2054,0,35,51,3. b) Silica-rich glass with vesicles (rounded bright areas within the dense glass). Sample C2054,0,35,52,3.

Another frequent feature in the silica-rich glassy matrix is the presence of vesicles (Fig. 3b). The number and size of vesicles vary widely across studied glassy regions, including when they are very close to one other. We mentioned earlier that the shock temperature in aerogel was very high upon particle impact, enough to melt or to vaporize the aerogel locally. The vesicular microstructure could have originated from boiling of melted aerogel and trapped during the subsequent rapid quenching. These vesicles could also originate from degassing of volatiles or from thermal decomposition of minerals (such as small iron-sulfide, for instance) present in components of Wild 2 itself, which did not survive the flash heating of the collection. This could be also the case of phyllosilicates and carbonates, both being major phases originating from aqueous alteration. Phyllosilicates have not been clearly detected to date in the Stardust samples, while there are rare occurrences of carbonates (Mikouchi et al. 2007; Wirick et al. 2007). For both phases, the thermal decomposition occurs below 800 °C. If the grain sizes are small, these minerals should not generally survive the high temperatures. However, Barrett et al. (1992) showed that fine-grained serpentine does survive capture in aerogel if it is present within more refractory grains. Laboratory simulations of hypervelocity capture have been conducted with powder of phyllosilicates (Noguchi et al. 2007). TEM investigation of these grains showed that melting and vesiculation occurred at the surface of the grains along a zone ranging from 0.5 to 1 μm thick. If the grain size of hydrated silicates in Wild 2 (if present) was lower, they were likely fully consumed in the hot aerogel, the vesicles being the consequence of the thermal decomposition. High vesiculation within an amorphous residue was also observed in experimentally shocked phyllosilicates onto Al-foils (Wozniakiewicz et al. 2010). The presence of minerals commonly associated with aqueous alteration in meteorites has been recently confirmed by the discovery of low-temperature Ni-Zn-Cu-Fe-sulfides in the Stardust collection (Berger et al. 2011).

Within the glassy matrix, the cometary material is not distributed homogenously. Elemental distribution maps performed by TEM nano-analysis (Fig. 4) showed the presence of nonvesicular "dust-rich" submicron patches dispersed in molten aerogel (Leroux et al. 2008b). These are frequently separated from each other by up to several micrometers. This configuration is consistent with loosely aggregated particles that were disaggregated and for which the individual silicate fragments were scattered into molten aerogel. The presence of these patches shows that mixing of melted cometary silicate components and molted aerogel has been incomplete. This is due in part to liquid immiscibility in the liquid state, as shown on most of the binary phase diagrams with SiO_2 (e.g., Mysen and Richet [2005] and references thererin). This miscibility gap is a strong thermodynamic barrier, which precludes full mixing between silicates and SiO_2 melts (see Leroux et al. [2008b] and Roskosz et al. [2008] for details). As a consequence, if the original microstructure of the components has been lost, chemical compositions have been preserved despite a significant impregnation of SiO_2 from the aerogel. The absence of vesicles for most of "dust-rich" patches shows that they probably originate from anhydrous silicates.

The fine-grained material is thermally damaged, but the chemical signature is still well preserved at the

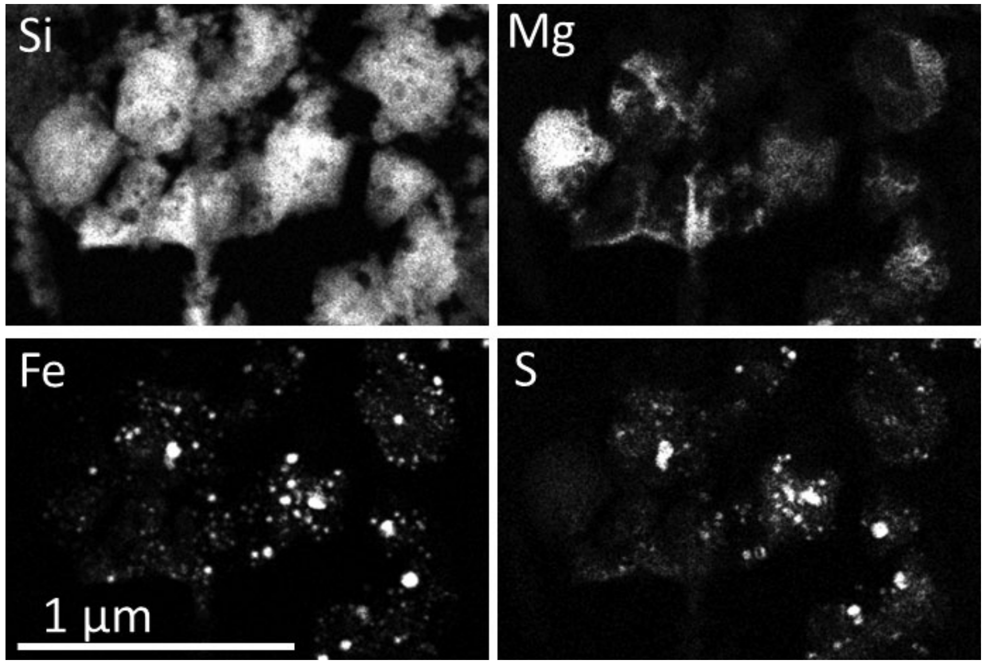

Fig. 4. X-ray intensity maps for Si, Mg, Fe and S for a silica-rich clump in sample C2054,0,35,52,3. The distribution of Mg is strongly heterogeneous within the glass and outline "ghost silicates." Iron is found mainly in the form of Fe-Ni-S inclusions.

submicron scale. The concentration of Wild 2 material within the silica-rich glass is highly variable, frequently within the range 5–10% (Leroux et al. 2008b). The bulk compositions are very close to a CI composition (including sulfur), except the Si and O elements derived from the aerogel (Leroux et al. 2008b, 2009; Stodolna et al. 2009; Rietmeijer 2009a, 2009b). The fine-grained material of Wild 2 is thus reasonably primitive in terms of chemical composition, even at the submicrometer scale. Similar conclusions were deduced from samples extracted from the walls of bulbous tracks (Stephan et al. 2008a, 2008b). The destruction of these fine-grained aggregates in aerogel prevents thorough study to decipher their origin. They may be crystalline mineral assemblages (mixture of silicates and sulfides), glass with embedded metal and sulfides (GEMS—see Bradley 1994) such as that found in chondritic porous interplanetary dust particles (CP-IDPs), or other unprocessed interstellar material.

BIAS DUE TO THE CAPTURE EFFECTS

The capture in aerogel induces several biases that are important to take into account to correctly establish the original nature of the material from Wild 2. Elemental distribution maps performed on entire tracks clearly show that the terminal particles are different from material trapped in bulbs (Flynn et al. 2006; Ishii et al. 2008a; Lanzirotti et al. 2008), showing that aerogel acted as a filter during the collection. The coarse-grained crystalline components are found at the end of tracks and are relatively well preserved. In contrast, the fine-grained material originated from friable aggregates is scattered along walls of tracks and is found severely damaged. The survival of Wild 2 material thus depends on the mineralogy and structure of the incoming particles. As seen above, the extreme conditions of the capture affect the grain size by fusion of the surface and removal of the molten layer by ablation. When grains are large, they could survive, despite a reduction in size that it is difficult to quantify. When the grains are small (typically less than 1 µm in diameter), they were often completely consumed in the molten aerogel.

Due to the capture thermal effects, the mineralogical signature of Wild 2 can be strongly biased. For example, the melting temperature of forsterite is approximately 300 °C higher than that of enstatite, leading to a preferential preservation of forsterite compared with enstatite. This selective effect has been experimentally demonstrated by impact experiments into aerogel of olivine and pyroxene powder using a light-gas gun at a velocity similar to the relative velocity between particles from Wild 2 and the Stardust spacecraft (Stodolna et al. 2011). Olivine grains were found to be well preserved, while most of the enstatite grains were severely damaged by significant melting in the shocked aerogel. This can lead to an erroneous olivine/pyroxene ratio in the Stardust collection. In general, minerals survive better when their melting temperature is high. This selective effect could explain the apparent absence in the Stardust aerogel of fragile minerals such as phyllosilicate (if present on Wild 2) as they have a decomposition temperature below 1000 °C.

For a given phase, the composition may also have marked influence on phase survival. This is the case for minerals that have a large range of solid solution. The melting temperature is therefore not unique, but depends on the composition. For example, olivine is a solid solution between two endmembers Mg_2SiO_4 (forsterite) and Fe_2SiO_4 (fayalite). Forsterite has high melting temperature, close to 1900 °C, but the melting temperature of fayalite is a much lower 1200 °C. The melting temperature varies somewhat linearly between the two endmembers. In the Stardust samples, Zolensky et al. (2006, 2008) showed that olivine has an extremely wide compositional range, but a pronounced peak at forsterite. This peak is certainly significant, but iron-rich olivine grains were probably consumed preferentially in the hot aerogel, in particular for the small grains, causing a bias in the Mg-Fe composition diagram of olivine. Similar effects are expected for other solid-solutions in minerals of Wild 2.

CONCLUSION

Wild 2 particles suffered extreme conditions during their hypervelocity impact capture. The dominant effect corresponds to an extremely high shock temperature in the aerogel. It can be considered that particles entered into hot aerogel and were flash heated. The large and dense minerals survived well, mainly because they dissipated their kinetic energy by ablation of the melted surface. In contrast, the fine-grained aggregates were strongly damaged and are not representative of the unheated Wild 2 material. Such aggregates were disaggregated, and the fragments have been melted and mixed together with melted aerogel. However, some individual silicate remnants, as islands of "ghost minerals," are still detectable by chemical mapping, although the original petrologic properties have been destroyed. It is likely that bulk chemistry is preserved in the silica-rich glassy clumps. The melted fragments have previously been shown to be composed of a limited number of subgrains with an average composition around the chondritic CI composition. The capture generates some biases that are important to take into account to best determine the mineralogy of the Wild 2 grains from the recovered Stardust samples. As these silica-rich glassy matrices in the Stardust samples are becoming better studied, it may be possible to compare them with other primitive objects such as matrix of chondrites or aggregate IDPs. Such an approach has been developed recently by Rietmeijer (2009c). Discovery of well-preserved fine-grained material is still possible because thermal effects are highly heterogeneous. For instance, Brownlee et al. (2006) showed in their fig. 2 an apparently well-preserved fine-grained matrix with an approximate chondritic composition. The aggregate was found in contact with large enstatite and FeS grains that might have protected the fine-grained material from heating and interaction with the aerogel.

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