Micro-craters in aluminum foils: Implications for dust particles from comet Wild 2 on NASA’s Stardust spacecraft


1. Introduction

NASA’s Stardust spacecraft flew through the coma of comet 81P/Wild 2 in January 2004 to capture cometary particles for return to Earth, to be followed by detailed mineralogical, compositional and isotopic analysis in the laboratory [1]. The principal particle capture medium was low-density silica aerogel [2] residing in a modular collector frame that was wrapped with aluminum foil (Al1100 alloy of temper 0; ca. 100 μm thickness), to hold the aerogel in place and facilitate careful post-flight removal of some 132 individual aerogel blocks, each some 2 × 4 cm across and 3 cm deep (Fig. 1). Consistent with expectations, the preliminary examination (PE) of the collectors revealed numerous penetration tracks in aerogel and hypervelocity craters on the metal surfaces, with both types of impact features displaying surprising morphologic diversity, as shown by Hörz et al. [3]. Analysis of preserved cometary residues in the foil craters showed a wide range of elemental compositions, indicating both monomineralic and polymineralic impactors throughout the crater size range [4]; similar observations apply to the impactor residues in aerogel tracks [5,6]. Cometary particles collected by Stardust show mineralogical and compositional heterogeneity down to the nanometer scale, which must also be reflected in their physical properties. In contrast to previous micrometeoroid collections from low Earth orbit (LEO) which have all had poorly constrained speeds and undetermined impact angles, trajectories of Wild 2 particles relative to the Stardust collectors were highly constrained [7] with encounter velocity a constant 6.1 km s⁻¹ and impact angle normal to the exposed surface. In this paper we discuss the application and limitations of light gas gun (LGG) laboratory impact experiments in the interpretation of the particles responsible for Stardust aluminum foil craters.

2. Preliminary examination (PE) of Stardust foils

Following return of Stardust to Earth in January 2006, the Al-foil surfaces were examined by an international team, using a wide

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range of techniques. An important goal of the time-limited PE was to document the number and size distribution of dust particles encountered during flight through the Wild 2 coma [3]. An initial optical scan, limited to craters > 20 μm in diameter, was made of all the foils in situ on the collector. Selected foils were then removed for more detailed study at multiple institutions, using Scanning Electron Microscope (SEM) methods to yield sufficient spatial resolution for precise measurement of craters less than 5 μm in diameter. Based on pre-mission tests, all laboratories adopted a uniform protocol for crater measurements to ensure internal consistency of all Stardust PE measurements. By common agreement, it was decided that the reported crater dimension would be the average of three top lip diameters, as illustrated in Fig. 2. It is usually easy to make top lip diameter measurements by SEM on craters of >20 μm diameter, although rim-width may vary around a crater due to detachment, leaving only a thin lip. Stardust craters had a wide range of size from approximately 480 μm down to 100 nm diameter, and a diverse range of shapes including simple “bowls” (comparable to Fig. 2) and complex features with multiple overlapped depressions (Fig. 3) that suggest heterogeneous internal mass distribution, and aggregate structure for some particles [3,4].

3. Laboratory experiments

Interpretation of craters on Al surfaces exposed in low Earth orbit has been aided by previous experimental studies [8,9]. For Stardust, a unique case where impact speed and angle were very well constrained, we undertook an extensive suite of laboratory simulations before and after sample return, using LGG at the University of Kent in Canterbury and NASA’s Johnson Space Center in Houston. Our initial work [10] concentrated on establishing the relationship between projectile diameter and impact crater diameter at 6.1 km s\(^{-1}\) for a range of small monodispersive spherical silicate projectiles (Fig. 4), down to a minimum size of ca. 9.5 μm, which yielded craters of ca. 40 μm top lip diameter. Also, the effects of projectile density were explored for four different materials (0.4 g cm\(^{-3}\) to 7.9 g cm\(^{-3}\)), spanning the range expected for cometary dust [11]. The findings of our earlier experiments were employed in the initial interpretation of Wild 2 dust fluence [3]. In further experiments, projectiles were selected as size, shape and density analogues for the likely range of cometary dust properties, and possible mineral and glass compositions. We used powders of natural minerals that greatly varied in shape (e.g. Figs. 5 and 6) and novel porous aggregate impactors to help us interpret: (i) crater morphology in three dimensions as a reflection of projectile density, structure and shape; (ii) the extent of alteration/preservation of impactor mineralogical and elemental composition. Monodispersive buckshot and polydispersive powder samples were impacted on flight-grade foil at ca. 6 km s\(^{-1}\) (Table 1), yielding numerous craters. Mafic silicate minerals (and amorphous materials) with density ca. 3.2 g cm\(^{-3}\) produced craters with a top lip diameter that is ca. five times the impactor diameter.

4. Refining the interpretation of crater size and shape measurements

Techniques for the firing of larger (>10 μm) projectile grains of known composition and size under tightly constrained velocity conditions in laboratory experiments are well established and reliable [12]. Experimental impact by grains of 1 μm and smaller grain size, of appropriate physical properties and at tightly constrained velocity, is much more difficult with LGG and is limited to electrically conductive materials in electrostatic Van de Graaff accelerators [16]. Monodispersive spheres of a size range suitable for LGG calibration shots must also yield identifiable impact residue, so that craters made by the nominal projectiles can be distinguished from fine gun-derived debris, precluding many polymer and ferrous metal projectiles. Also, escape of propellant gas through the very fine, porous projectile powder may cause lowering and dispersion of effective impact velocities, hindering precise calibration in the light gas gun by possibly introducing a slightly lower speed tail in the impacts.

As a result of these (and other) limitations, direct experimental calibration for craters < 10 μm, let alone < 1 μm, as a function of projectile properties has not been systematic to date. However, although the tiny Stardust impacts may carry only a very small fraction of the total mass, they reflect the most abundant cometary grains [3], and are very important in providing evidence for the size of pristine cometary dust components. As there is significant difference between fluence measurements for Wild 2 dust based upon in situ dust impact sensors [17] and measured impact features found on the Stardust collector foils, careful calibration of observed experimental crater vs. impactor size for the smallest dust is

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**Fig. 1.** Schematic vertical section through the Stardust cometary dust collector, showing relative positions of aerogel (with penetration tracks) and Al-foil wraps (which served as cratering targets) on the exposed front side of the collector. The aerogel had continuously variable density from some 0.005 g cm\(^{-3}\) at the exposed surface to 0.05 g cm\(^{-3}\) at the rear, thus trapping all particles < 100 μm.

**Fig. 2.** SEM image (SEI) of bowl-shaped craters from a light gas gun shot of diopside (Ca–Mg–Fe silicate) powder, showing measurement of crater diameter by SEM. Note difference between the full ‘top lip’ and a ‘narrow lip’ where thin metal has detached.
Fig. 3. SEM images of small Stardust impact craters on foil C054 W, 1, showing wide range of shape, especially in the width of the crater lip, crater outline, and crater floor topography.

Fig. 4. SEM images of monodispersive projectiles (a) soda lime glass, density ($\rho$) = 2.4 g cm$^{-3}$; (b) soda lime bubble glass, $\rho$ = 0.4 g cm$^{-3}$ (average, very variable); (c) polymethyl methacrylate, $\rho$ = 1.19 g cm$^{-3}$; and (d) hydrous silica, $\rho$ = ca. 2 g cm$^{-3}$. 
particularly important, especially as the impact sensor data are restricted to grains > 3 μm diameter. When fragmented projectiles are excluded, the close linear fit of the larger crater experimental data from Kearsley et al. [10], trending to intercept very close to the origin (Fig. 7), suggests that the calibration constant for the Stardust encounter conditions (crater top lip diameter = 4.63 x impactor diameter, based on soda lime glass sphere impacts) may be a good estimate even for very small particles, although error bars of ±5 μm [10] demonstrate the lack of precision, critical at small sizes.

In our recent experiments using sintered silica microsphere aggregates for analogues of fractal dust, as simulated by Blum and Wurm [18], many aggregates disintegrated during flight and impacted the Stardust foil target at 5.94 km s⁻¹ as small individual spheres (Fig. 4d), creating large numbers of very small craters. The spheres have a tight monodispersive size range, with an average of 1.55 μm and standard deviation of only 30 nm (Fig. 8). Their impact features show an average diameter of 3.42 μm with standard deviation of 0.29 μm (Fig. 8). From this single monodispersive sample we might suggest that an appropriate relationship is:

\[
\text{Crater diameter} = 2.2 \times \text{projectile diameter} \quad (1)
\]

This is a much smaller calibration factor than for larger soda lime glass impacts (4.63). Although the crater size distribution is of low dispersion, suggesting that little sphere fragmentation had occurred, this measurement is at a single impactor size, not a calibration line fit such as in Kearsley et al. [10], and ideally this experiment should be repeated with a range of particle sizes, from less than 1 μm up to 10 μm. To make direct comparison to the larger crater soda lime glass particle calibration, several factors must be considered. The silica is both porous (ca. 20%) and hydrous, with density ca. 2 g cm⁻³ (significantly less than for soda lime glass) and hence the particles might be expected to yield a narrower crater

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**Fig. 5.** SEM images of polydispersive mineral projectiles: (a) olivine, \( \rho \approx 3.2 \text{ g cm}^{-3} \); (b) diopside, \( \rho \approx 3.2 \text{ g cm}^{-3} \); (c) pyrrhotite, \( \rho \approx 4.6 \text{ g cm}^{-3} \); and (d) wollastonite, \( \rho \approx 2.8 \text{ g cm}^{-3} \).

**Fig. 6.** (a) SEM image of Stardust Al alloy target after LGG shot of wollastonite powder; (b) top left region of (a) showing diverse crater shapes, reflecting variable yaw of rod-shaped impactors; and (c) detail of an elongate wollastonite impactor crater.
Table 1

Hypervelocity impact experiments described in this paper

<table>
<thead>
<tr>
<th>Shot</th>
<th>Projectiles (NHM sample)</th>
<th>Density (g cm(^{-3}))</th>
<th>Projectile size and shape</th>
<th>Impact speed (km s(^{-1})) ± 2%</th>
<th>Comments/contaminants</th>
</tr>
</thead>
<tbody>
<tr>
<td>G260405#1</td>
<td>Olivine (BM 1950-337)</td>
<td>3.2</td>
<td>38–53 (\mu)m irregular</td>
<td>6.07</td>
<td>Minor Fe-sulfide</td>
</tr>
<tr>
<td>G210306#2</td>
<td>Enstatite (BM.2005,M318)</td>
<td>3.2</td>
<td>&lt;38 (\mu)m irregular</td>
<td>5.91</td>
<td>Minor Talc inclusions</td>
</tr>
<tr>
<td>G270405#1</td>
<td>Diopside (BM.2005,M310)</td>
<td>3.2</td>
<td>Polydispersive irregular</td>
<td>6.01</td>
<td>Minor inclusions</td>
</tr>
<tr>
<td>G190706#2</td>
<td>Bytownite (BM.2005,M312)</td>
<td>2.6</td>
<td>Polydispersive needles</td>
<td>5.93</td>
<td>K-feldspar inclusions</td>
</tr>
<tr>
<td>G210306#1</td>
<td>Pyrrhotite (BM.2005,M317)</td>
<td>4.6</td>
<td>Irregular</td>
<td>5.92</td>
<td>Minor Chlorite inclusions</td>
</tr>
<tr>
<td>G130106#2</td>
<td>Soda lime glass spheres</td>
<td>2.4</td>
<td>Monodispersive sphere samples</td>
<td>5.93–6.21</td>
<td>Variable Na, K and Ca</td>
</tr>
<tr>
<td>G100506#1</td>
<td>Polymethyl methacrylate (PMMA) spheres</td>
<td>1.19</td>
<td>57 (\mu)m</td>
<td>5.97</td>
<td>Source: Sigma Aldrich</td>
</tr>
<tr>
<td>G090506#1</td>
<td>Monodispersive polystyrene latex spheres</td>
<td>1.05</td>
<td>31.62 ± 1.56 (\mu)m</td>
<td>5.94</td>
<td></td>
</tr>
<tr>
<td>G040406#2</td>
<td>Stainless steel spheroids</td>
<td>7.9</td>
<td>87.9 ± 5.5 (\mu)m</td>
<td>5.96</td>
<td>Slightly oblate</td>
</tr>
<tr>
<td>G200207#1</td>
<td>Artificial aggregates of olivine, pyroxene and pyrrhotite</td>
<td>1.6–2.4</td>
<td>Polydispersive, porous, but break in gun flight?</td>
<td>4.63</td>
<td>2 mm Al 6000 series targets</td>
</tr>
<tr>
<td>G200207#2</td>
<td>Hydrous silica spheres</td>
<td>ca. 2</td>
<td>1.5 ± 0.03 (\mu)m</td>
<td>5.01</td>
<td>2 mm Al 6000</td>
</tr>
<tr>
<td>G240407#2</td>
<td></td>
<td>1.5 ± 0.03 (\mu)m</td>
<td></td>
<td>5.94</td>
<td>Al100 foil</td>
</tr>
</tbody>
</table>

All performed on Stardust foil and similar substrates using the technique of Burchell et al. [12]. Density data from Refs. [13–15].

Fig. 7. Crater top lip vs. projectile diameter for soda lime glass spheres, \(\rho = 2.4 \text{ g cm}^{-3}\). All shots close to 6 km s\(^{-1}\). Raw data in grey. Note clustering of bead size, with a small number of larger projectiles, and smaller craters due to fragmentation during acceleration. To remove this experimental artefact, craters < 4 times average sphere diameter are excluded from data shown in black. Average values (with standard deviation error bars) yield an excellent fit to a linear trend, with intercept very close to the origin.
diameter [11], implying a slightly larger correction factor (2.54) for a soda lime glass particle of the same size. Also, as is often seen in smaller Stardust craters (Fig. 3), the feature available for measurement is usually not a full top lip, but a narrow lip, reflecting detachment of the very thin outer part of the displaced aluminum lip during crater excavation (Fig. 9). This difference can be addressed by a correction derived from craters which have both partial top lip and partial narrow lip form, showing that the narrow lip diameter is approximately 0.78 times the top lip diameter. Taking both density and a correction for the true top lip diameter into account, the calculated calibration factor rises to ca. 3.26 for a soda lime sphere of equivalent size, lower than the value derived from larger craters (4.63, see Fig. 7). Until we are able to create a better calibration by shooting a wider range of projectiles < 10 μm at the appropriate velocity, we suggest that the current calibration factor (4.63) may be the most appropriate value to employ for craters as small as 15 μm diameter, with 3.26 as a minimum value for smaller craters if a full lip is preserved, and 2.54 if only a thin lip developed. If the lower calibration figure is subsequently confirmed, it would imply that the particle masses responsible for some of the smallest Stardust craters reported in Hörz et al. [3] may have been underestimated by a factor of about 2.9 (i.e. (4.63/3.26)3).

The craters preserved on the Stardust foils also include many within the sub-micrometer size range, for which experimental verification of dimensional scaling from laboratory analogue simulations utilizing LGG may ultimately prove impractical. We are therefore also using computational simulations to investigate the cratering process at this nanometer scale.

5. The three-dimensional shape of craters

The three-dimensional shape of impact features should yield important information about the density and internal structure of cometary dust grains [4,11]. Previous non-destructive studies of crater morphometry have mainly relied upon optical microscope methods, with depth below the ambient (pre-impact) foil surface determined by focusing consecutively on the undisturbed foil, and then on the crater floor, with the depth difference measured on a calibrated Z-axis micrometer. Crater diameter can also be measured by moving the specimen on the horizontal axes, for which the most easily recognized feature is the internal crater diameter, determined at the height of the ambient plane (i.e. following focusing on the foil external to the crater). For recognition of the correct height to be unambiguous, this technique requires optics with a very shallow depth of focus, and is necessarily limited to relatively large craters (>30 μm) due to the spatial resolution of the microscope optics, and often poor precision in the mechanical stage movement. Ironically, although SEM has the necessary spatial resolution to make precise lateral measurements on craters of less than 1 μm diameter, the inherent (and usually desirable) great depth of focus makes it almost impossible to track the ambient pre-impact plane into the crater interior. SEM is thus not suited to direct measurement of the inner crater diameter on the ambient plane, or absolute crater depth. However, new image processing programs (such as MeX from Alicona) enable three-dimensional reconstruction of shape from SEM stereo pair images [11], including precise location and measurement of features on the ambient plane, maximum crater excavation depth below the ambient plane, and detailed depth profiling. We have used MeX to measure crater depth/diameter ratios for different experimental impactors (Fig. 10), and we infer two differing controls: (1) impactor density; and (2) crater shape. The dispersion of depth/diameter ratios for each type of impactor reflects the range of possible grain aspects on impact. Robust steel and polymer spheres each show a narrow range of depth/diameter values; fragile soda lime glass spheres (some of which fragment into elongate shards during flight) and irregular mafic silicate grains yield a broader range: whilst elongate wollastonite needles yield enormous variation. Nevertheless, the linear relationship between depth/diameter and impactor density seen clearly from spherical impactors (Fig. 10) is replicated in data from craters produced by irregular impactors, albeit with much greater dispersion. An unexpectedly deep shape for one large Stardust crater containing residue from olivine was attributed by Kearsley et al. [4] to a ‘rod-penetration’ impact by an elongate cometary grain.

6. Complex impact features

Our experiments also help to explain compound Stardust features, through comparison to laboratory impacts by porous aggregates (Fig. 11). Projectiles were made by impregnation of a mixed polydisperse mineral powder (olivine, enstatite, diopside and pyrrhotite of grain sizes from <5 to >100 μm), gently agitated whilst exposed to aerosol droplets of polymer spray, followed by gentle heating to promote hardening of the polymer. Polished cross sections of aggregates reveal a very porous internal structure, with polymer and pore space occupying 25–50% of the volume, resulting in low overall density (ca. 1.6–2.4 g cm−3). LGG shots of aggregates cemented by acrylic and polyvinyl acetate aerosols were successful in generating compound impact features on polished Al alloy substrates, although larger grains (ca. 400 μm) broke into smaller aggregate fragments before impact.

Diameter measurements for these distinctive, shallow and irregular structures should not be compared directly to calibrations
based upon simple but relatively deep bowl shapes that were created by single, dense mineral grains. If an inappropriate deep bowl crater calibration is applied to a shallow compound feature, it will not reflect internal impactor porosity and leads to overestimation of the impactor mass. Estimation of size and mass of internal aggregate components was attempted for a compound Stardust crater with complex depth profile by Kearsley et al. [4], but with a simplistic model of a porous, low-density aggregate impactor structure that cannot be verified. A mass estimate was obtained from size of overlapped minor bowl craters, and summation of the sub-grain impactor masses.

The Stardust mass fluence plot for smaller craters in Hörz et al. [3] was derived from the ‘simple-bowl’ size calibration of Kearsley et al. [10], using linear extrapolation of the crater/impactor size relationship downward from craters of >40 μm top lip diameter. However, many smaller Stardust craters (ca. micrometer scale) are shallow, overlapped features of complex three-dimensional shape. In such compound features, excavated by impact of porous aggregates [4], the external lip diameter does not simply reflect the size of a single dense impactor, but also the distance between sub-grain mass centres within the aggregate. For these craters, comparison of external top lip diameter to a simple bowl calibration results in overestimation of mass, possibly by as much as a factor of 4 [4].

MeX can also be used to determine the total amount of metal displaced (i.e. internal crater volume below the ambient plane), to give a more consistent comparison between craters of differing shape. This may provide a measure of the kinetic energy (and hence mass) for both calibration shots and Stardust particles, although

Fig. 9. (a–c) SEM images from LGG shot of silica aggregates on Stardust foil: (a) numerous small craters from disaggregated 1.55 μm spheres; (b) larger aggregate crater with faint, thin uplifted lip (between arrows) and ragged edge of broader top lip; (c) enlargement of three small crater with thin lips; and (d) SEI of a impact on Stardust foil C011 N; showing a thin lip with remnants of a broader top lip.

Fig. 10. Depth/diameter for laboratory impacts plotted against projectile density, all shots at ca. 6 km s⁻¹: (a) spherical projectiles only; (b) all projectiles, including minerals of irregular shape. Note similar gradient, but much greater dispersion for irregular grains (low R²), and a few fragmented soda lime glass projectiles.
efficiency of material displacement by synchronous, closely spaced impactor centres is likely to be less than for a single dense impactor of equal mass, as was recognized by Schultz and Gault [19] and Hörz et al. [20]. Due to the laborious nature of high quality stereo image collection from very small craters, extraction of digital elevation models, depth profiles and volumes, this process was not more widely applied during the brief Stardust PE, but should prove valuable for future research.

7. Conclusions

It is now possible to make routine, non-destructive quantitative comparisons of size and shape of craters on Al-foil at scales down to below 1 \( \mu m \). New image processing techniques allow three-dimensional crater reconstruction and measurement from SEM data, permitting estimates of impactor dimensions, density, mass and internal structure. As the Stardust encounter velocity and impact angle remained invariant throughout the encounter with comet Wild 2, high fidelity simulations can be pursued in the laboratory, especially for grains of 10 \( \mu m \) and larger. These provide a reliable basis for interpretation of the craters observed on the Stardust foils. Although the existing calibrations are suitable for use with craters larger than 40 \( \mu m \) diameter, our recent experiments suggest substantial strength-scaling effects on crater morphology may occur at smaller sizes, especially for craters < 1 \( \mu m \). We suggest that the current estimate of mass fluence for Stardust grains of less than 5 \( \mu m \) diameter should not be considered to be more precise than the potential errors from inconsistencies in crater diameter measurement (up to threefold mass underestimation) or comparison to simple experimental crater morphology (up to fourfold mass overestimation). More experiments are required for better interpretation of micrometer and smaller impact features, and we are investigating appropriate, well-characterised projectiles and launch facilities for such work.

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References


