SURVIVAL AND CONDITION OF MICRON-SCALE REFRACTORY GRAINS IN STARDUST-ANALOG AL FOIL CRATERS.

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Introduction: There are now numerous published studies of silicate and sulfide mineral capture during hypervelocity impact on Al-foils [e.g. [1]), but relatively little is known about how refractory carbides and nitrides survive in micrometer-scale craters. This has been due to difficulties in both experimental impact methods and sample preparation technique prior to the advent of focused ion beam lift-out (FIB) techniques. Here, we present FIB-TEM results from STARDUST-analog test shots of µm-scale refractory grains.

Samples and Experimental Methods: A mixture of refractory materials (including SiC, Si_3N_4, TiC, and TiN) was shot at Al 1100 foil in the two stage light gas gun at the University of Kent [2] with an impact speed of 6.05 km/s (simulating the STARDUST-Wild 2 encounter). The resulting Al foil craters were characterized with SEM-EDXS, and FIB sections were successfully made from 3 Si-rich and 8 Ti-rich craters (including 4 presented earlier [3]). Transmission electron microscopy (TEM) was then used to characterize the Al crater properties as well as the crystal structures and morphologies of the surviving grains. Crater/impactor diameter (D_c/D_p) calibration [4] for µm-scale silicate particles (density 2 – 2.6 g/cm^3) suggests $D_c/D_p \approx 1.6$. Only limited D_c/D_p calibration data is available [5] for the higher density range appropriate for Si/Ti carbides and nitrides (3.2-5.4 g/cm^3). By extrapolation, we predict their craters to have diameters ~1.7- 2.1x larger than the projectile. The < 5 µm size range would indicates craters formed by impactors < 3 µm in size.

Results: Table 1 summarizes results from 11 craters, including crater diameter and the number, size (or size range), and phase of the surviving crystals found at the crater bottom. No grains were found in raised crater rims, a few were found in sidewalls. Diffraction patterns from surviving Ti-rich grains always showed a 4.2-4.3Å FCC phase, consistent with either TiN or TiC. In some cases slight differences in the lattice parameter and low energy EDXS peaks (of C and/or N) indicated one phase over the other.

Si-rich craters: Prior FIB-TEM studies of craters Si1 and Si2 [3] demonstrated the survival of crystalline SiC and the less-refractory Si_3N_4 phase after impacting Al foils under STARDUST-like conditions. However, the SiCs from crater Si1 were fragmented and only two ≈200 nm diameter fragments were observed, less material than expected given the crater's size. In contrast, the 0.9 µm diameter Si3 crater contained a single 0.5 µm crystalline SiC, a grain that is quite large relative to its crater size. Like the surviving Si_3N_4 grain from crater Si2 [3], the SiC is still faceted, and its size suggests no material was lost on impact.

Table 1. Summary of FIB-TEM results from STARDUST analog Al foil craters.

Name	Crater Diam. (µm)	# crystals	Crystal size (µm)	Phase
Si1	4.2	2	0.20-0.25	SiC
Si2	1.4	1	0.51	Si_3N_4
Si3	0.9	1	0.53	SiC
Ti1	5.0	5	0.12-0.43	TiN
Ti3	1.4	2	0.27 -0.33	TiC or TiN
Ti4	1.0	1	0.33	TiN
Ti5	2.1	3	0.08-0.40	TiN
Ti6	4.2	1*	0.44*	TiC or TiN
Ti7	4.2	5	0.24-0.58	TiC
Ti8	5.2	2	0.52-0.88	TiC or TiN
Ti9	4.6	3	0.58-0.72	TiC or TiN

* Uncertain value due to problem with FIB section

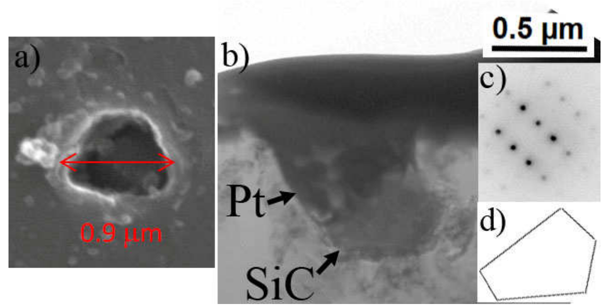

Fig.1. a) SE image of Si- crater Si3 b) Bright-field TEM image of FIB cross-section from Si3 showing 0.65 x 0.43 µm SiC grain at bottom (crater is filled with darker Pt) c) SAD of 3C-SiC at the [112] orientation d) Schematic (drawn to scale) showing shape of crystalline SiC.

Ti-rich craters: Crater Ti4 again demonstrates survival of a submicron refractory projectile as an intact single crystal (Fig. 2). The surviving TiN grain is elliptical and oriented with its long axis perpendicular to impact, which is strikingly different from FIB-TEM results of less-refractory submicron impactors (e.g., [6]) that often have surviving residues flattened on the crater floor. Crater Ti5 contains two adjacent surviv-

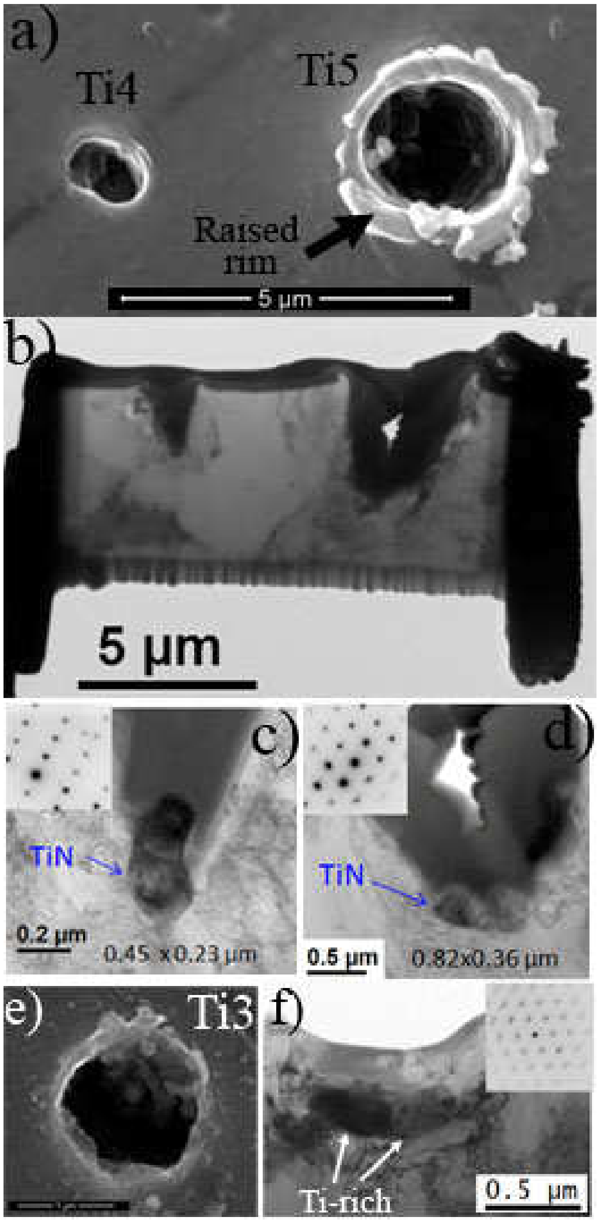

Fig. 2. a) SE images of adjacent Ti-rich craters Ti4 and Ti5 b) FIB cross-section through both Pt-filled craters c) TEM image of surviving TiN grain in crater Ti4 with inset [112] diffraction pattern d) TEM image of TiN grains in crater Ti5 with [011] diffraction pattern e) SE image of Ti3 crater and f) TEM image of two TiN grains from Ti3 with [011] diffraction pattern

ing TiN crystals (another ~0.08 µm diameter TiN fragment is found in the crater sidewall). This crater has the raised bright rim (or lip) that is characteristic of most STARDUST-like hypervelocity impacts [5]. The more carrot-like crater shape of Ti4 compared with Ti5 shows that crater shapes are clearly influenced by the orientation angle at impact of elliptical grains. Crater Ti3 lacks a prominent crater rim and shows two adjacent surviving Ti-rich crystalline grains (4.3Å fcc).

Discussion: Micrometer-scale projectiles show different behavior than larger ones, both in the condition of the surviving grains at the crater bottom and in the craters that they create. These smaller projectiles tend to survive as single intact faceted crystals (e.g. Si3 and Ti4 craters here and Si2 crater in Fig 3. from [3]), rather than the crystalline fragments observed from larger projectiles (e.g., see crater Ti1 shown in Fig. 1b of [3]). Unlike larger projectiles, the grains tend to not be flattened on the crater bottom (the Ti4 elliptical grain is a dramatic example of this), which is a clear indication that they did not melt. Moreover, the craters formed by µm-scale refractory projectiles (e.g., Si3 and Ti4) often lack the bright raised rims characteristic of slightly larger projectiles (e.g., Ti5). This is particularly significant because difference in appearance may cause such craters to be preferentially overlooked or disregarded, especially in automated crater searches. From comparison to the measured size of grains in the projectile powder samples, we can confirm that these craters are also small relative to the projectiles that formed them, with D_c/D_p only 2.4 x (± 0.7). This is in agreement with past studies demonstrating transition in cratering behavior for < 10 µm projectiles [4].

These observations have several important implications for the study of Wild 2 STARDUST samples. When studying Wild 2 cometary fines in Al foil craters, it might be assumed that one could more rapidly survey the bulk of the material by focusing on larger craters that formed by impacts of aggregates containing many fine grains. However, smaller craters formed by the impact of single submicron grains contain better-preserved material (less fragmented, less flattened on impact, etc.), perhaps due to lower peak temperatures and pressures on impact [1], and may therefore provide more accurate information about the original projectile. Moreover, presolar grain abundance estimates based on measurements from larger craters have recently been shown to be artificially low due to alteration or destruction of such grains [7]. Smaller craters may indeed provide more accurate estimates [e.g., 8].

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