SURVIVAL AND CONDITION OF SUBMICRON REFRACTORY GRAINS IN AL-FOIL CRATERS.

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Introduction: Although extensive studies of Al-foil captured samples have been made previously (such as those from the LDEF mission [1]), little attention was paid to submicron craters due to sample preparation difficulties prior to the advent of focused ion beam lift-out (FIB) techniques. Here, we present FIB-TEM results from STARDUST-analog test shots of submicron refractory grains that illustrate the utility of small Al foil craters (≤ 1µm) for the study of the Wild 2 fine cometary fraction.

Samples and Experimental Methods: A mixture of refractory materials (including SiC, Si_3N_4, TiC, and TiN) was shot at Al 1100 foil with 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 then characterized with SEM-EDXS, and FIB sections were successfully made from 3 Si-rich and 9 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. For the density range of Si/Ti carbides and nitrides (3.2-5.4 g/cm3), [4] predicts craters with diameters ~5-6x larger than the projectile, so the <5µm size range indicates craters formed by single submicron impactors.

Results: Table 1 summarizes results from the FIB-TEM studies on 12 craters that were successfully extracted, including the Al foil crater diameter and the number, size (or size range), and phase of the surviving crystals found at the crater bottom. No crystals were found in raised crater rims, but a few were occasionally found in the sidewall. Diffraction patterns from surviving Ti-rich grains always showed a 4.2-4.3Å FCC phase (which is 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 0.9µm 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 the Si2 crater [3], the SiC is still faceted, and its size suggests that 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	0.8	1	0.53	SiC
Ti1	5.0	5	0.12-0.43	TiN
Ti2	5.1	n/a*	n/a*	n/a*
Ti3	1.4	2	0.27 -0.33	TiC
Ti4	1.0	1	325	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

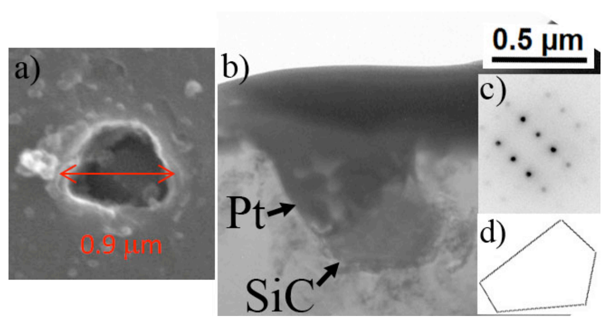

Fig.1. a) SEM image of Si-rich Si3 crater b) Bright-field TEM image of FIB cross-section from Si3 crater showing 0.65 x 0.43 µm SiC grain at bottom (crater filled with darker Pt) c) SAD of 3C-SiC at the [112] orientation d) Schematic drawn to scale showing shape of crystalline SiC.

Ti-rich craters: The Ti4 crater again demonstrates survival of a submicron refractory projectile as an intact single crystal. 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., [5]) that often have surviving residues flattened on the crater floor. The Ti5 crater shows two adjacent surviving TiN crystals (another ~0.08 µm diameter TiN fragment is found in the crater sidewall). Its crater has the raised bright rim (or lip) that is characteristic of most STARDUST-like Al foil impacts [4]. The more carrot-like crater shape of Ti4 (in comparison with Ti5 formed by grain of the same phase into same Al target grain) shows that the crater shapes are clearly influenced by the orientation angle at impact of elliptical grains.

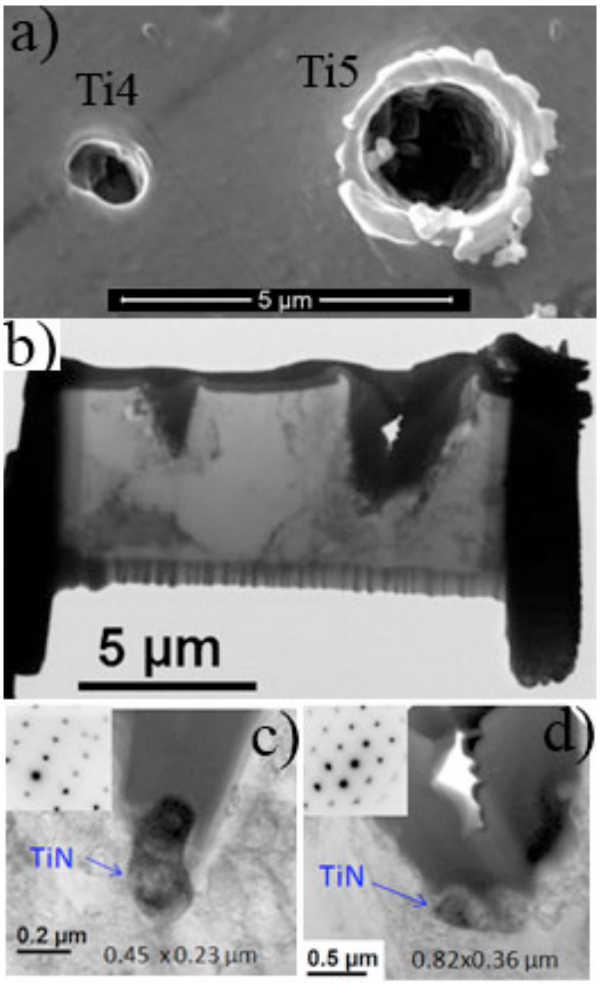

Fig. 2. a) SEM images of adjacent Ti-rich Ti4 and Ti5 craters b) FIB cross-section through both Pt-filled craters c) TEM image of surviving TiN grain at Ti4 crater bottom with inset [112] diffraction pattern and d) TEM image of TiN grain at Ti5 bottom with [011] diffraction pattern.

Discussion: Submicron projectiles (which form Al craters ≤ 1µm) show different behavior than larger ones, both in the condition of the surviving grains at the crater bottom and in the Al craters that they create. The submicron projectiles often survive as a single intact faceted crystals (e.g. Si3 and Ti4 craters here and Si2 crater in Fig 3. from [3]), not the crystalline fragments observed from larger projectiles (for an example of this fragmentation behavior see Ti1 crater Fig. 1b from [3]). Unlike larger projectiles, the grains tend to not be flattened on the crater bottom (e.g. Ti4 elliptical grain taller than it is wide is a dramatic example of this), which is a clear indication that they did not melt. The Al craters formed by submicron refractory projectiles (e.g. Si3 and Ti4) often lack the bright raised rims characteristic of slightly larger projectiles (e.g. Ti5). Their different appearance may cause such craters to be preferentially missed or disregarded, especially in automated STARDUST Al crater searches. The Al craters are also smaller relative to the submicron projectiles that formed them, with crater diameters 2.4x (± 0.7) larger than the projectile diameter (as opposed to 5-6x larger values predicted by [4] for impactors of this density). This trend is in agreement with [6], which found such a transition in the crater behavior for < 10µm projectiles.

These observed behaviors have several consequences for the study of Wild 2 STARDUST samples. When studying Wild 2 cometary fines in Al foil craters, one could more rapidly survey the material by focusing on larger craters that were formed by impacts of aggregates containing many fine grains. However, smaller craters formed by impact of single submicron grains contain better-preserved material (less fragmented, less flattened on impact, etc.), perhaps due to lower peak temperatures and peak pressures on impact [7]. Also presolar grain abundance estimates based on measurements from larger craters [8] will be artificial low due to the more altered condition of such grains.

References: [1] McDonnell J.A.M. et al. (1992) *LPSC XXII*, 185. [2] Burchell M.J. et al. (1999) *Measure. Sci. Tech, 10*, 41. [3] Croat et al. (2013) *LPSC XLIV, Abstract # 2625.* [4] Kearsley et al. (2007) *Met. Planet. Sci. 42,* 191. [5] Leroux et al. (2008) *Met. Planet. Sci. 43,* 97. [6] Price et al. (2010) *Met. Planet. Sci. 45,* 1409. [7] Wozniakiewicz et al.. (2012) *Met. Planet. Sci. 47,* 708. [8] Floss et al.. (2012) *Ap. J. 763,* 140.