CHARACTERIZATION OF REFRACTORY PRESOLAR GRAIN ANALOGUES SHOT INTO AL FOIL UNDER STARDUST-LIKE CONDITIONS.
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Introduction: Once properly calibrated, presolar SiC abundance determinations from Stardust Al foils will provide a good quantitative measure of the degree of primitiveness of Wild 2 material [1]. That presolar SiC can survive collisions with Al-foils under Stardust-like conditions has already been demonstrated [2]. In an effort to better understand the survival probabilities of various refractory grains under such conditions, we have carried out experimental test shots into Al foil using a mixture of refractory acid-insoluble phases. We report here preliminary results from a coordinated examination of both the raw projectile material and surviving residues on Al foil target with TEM, SEM-EDXS and Auger spectroscopy.

Experimental Methods: A mixture of refractory materials (alumina, SiC, Si_3N_4, TiC, TiN, diamond and olivine stuck together with WN acrylate) was shot at Al 1100 foil with the two stage light gas gun at the University of Kent [3]. The impact speed was 6.05 km/s. The resulting cratered Al foil was then examined with a JEOL 840a equipped with a NORAN Energy Dispersive X-ray Spectrometer (EDXS) and NSS spectral imaging software. Debris on both the crater rims and bottoms was also examined with the PHI 700 Auger Nanoprobe, which allows chemical measurements with a finer spatial resolution. Raw test shot material was also deposited on carbon-coated copper TEM grids and examined in a JEOL 2000FX analytical TEM equipped with a NORAN EDXS.

Results and Discussion: To remove uncertainty in the interpretation of analyses of crater residues, the raw projectile material was first examined in the TEM. A typical cluster of material is show in Fig. 1a. The phases present in the fragment were identified primarily based on their chemical compositions with EDXS. The color-coded phase map (Fig. 1b) indicates that the material is a fine-scale mixture dominated by diamond, TiN and Si_3N_4. TEM analysis of ~60 grains indicates that the material consists of 40% TiN, 35% diamond, 20% Si_3N_4, and minor amounts of TiC and olivine (percentages defined by areal fraction). Phase identification was confirmed on selected grains using diffraction to identify their crystal structures. Multiple diffraction patterns from representatives of each phase agreed with the known crystal structures, with patterns corresponding to TiN (FCC, 4.4Å), TiC (FCC, 4.4Å; isostructural with TiN), Si_3N_4 (hexagonal, a=7.8Å, c=5.6Å) and diamond (cubic, a=3.6Å). The average geometrical mean diameters of the grains were 1.7, 1.4, 1.0, 0.7 and 0.6 microns for olivine, TiN, diamond, Si_3N_4, and TiC, respectively. Given our interest in the survival probability of presolar SiCs and their condition after impact [2], it is unfortunate that no SiCs were found among the ~60 grains examined in TEM.

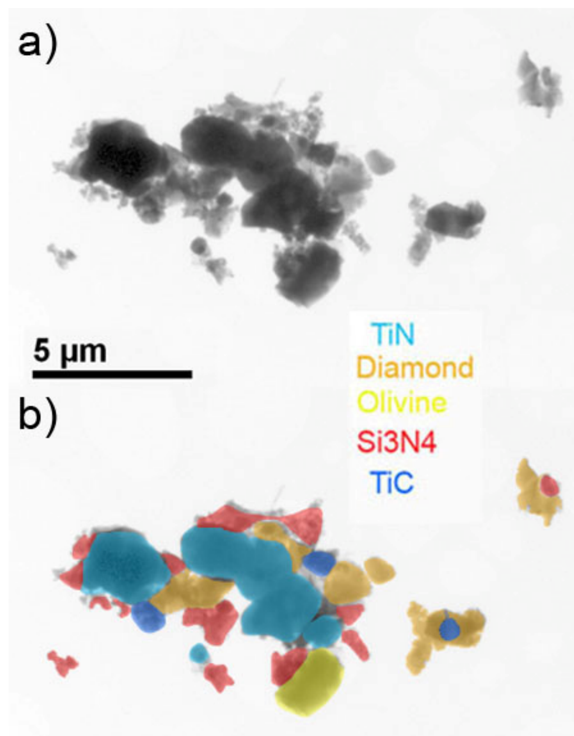

Fig.1. a) Bright-field image of typical fragment from the refractory grain mix projectile material and b) same fragment with phases identified with color code.

In order to better define the capabilities and limitations of SEM-EDXS spectral imaging and the Auger Nanoprobe, the same grains from the TEM grid were subsequently examined in these other instruments. EDXS spectral images of the TEM-studied grains and hundreds of additional ones were consistent with the projectile material predominantly consisting of TiN, Si_3N_4 and diamond. Grains with a composition consistent with alumina were also found, as were more olivine grains. Unfortunately smaller Si_3N_4 fragments attached to larger diamond grains appear compositionally similar to what is expected for SiC, so SiC in this mixture cannot be reliably identified solely with SEM-EDXS. Auger spectra of the Si_3N_4 and TiN grains from the projectile mix (e.g. Fig. 4b) confirm our ability to identify these phases, although variable amounts of carbonaceous coatings were seen on the grains. Measurements of more isolated grains are needed to obtain accurate sensitivity factors for N and Ti, which are inherently difficult in TiN grains due to unresolvable overlap of the Ti and N peaks.

SEM examination of the Al foil target revealed many craters with an average size of 3.5 μm. Thus, the crater sizes are roughly in agreement with prior determinations of the relationship between impactor size and crater size [4]. X-ray spectral images of ~70 craters show that most (~80%) are dominated by Ti-rich debris on the crater floors (Fig. 2). In the Ti-rich craters, Si and N were usually not detected, which may result from difficulties based on the crater topology, as described by [4]. Smaller Ti-rich residue grains were present on the crater rims but were less abundant. A number of Si-rich craters (Fig. 3) were also found although these are less abundant (~10% of population). Mg was also clearly present in one crater along with Si, perhaps indicating an olivine grain as the impactor.

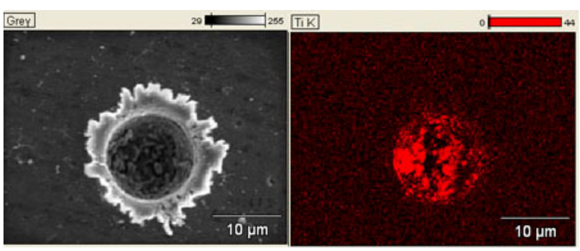

Fig. 2. Image and extracted Ti x-ray map from a typical Ti-rich crater.

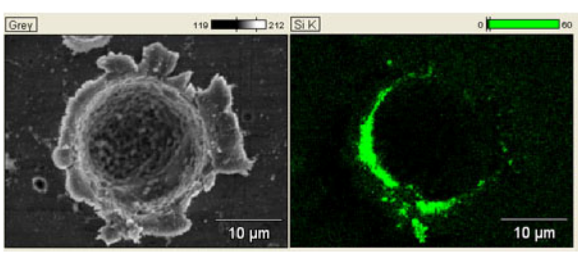

Fig.3. Image and extracted Si x-ray map from a Si-rich crater. Uneven distribution of Si counts on left side is an EDXS count rate artifact (crater wall facing detector has enhanced counts).

Further characterization of these Ti-rich craters with the Auger Nanoprobe was hampered by moderate C contamination from the spectral image acquisitions (35 min at ~2000x mag). Measurements of six craters that had shown Ti x-ray enrichments showed Auger spectra dominated by Al, C and O peaks; Ti was not detected. Auger spectra from uncontaminated craters (those without long SEM exposures) as expected did show lower C signals, but were still dominated by the same peaks and generally lacked the N, Si and Ti peaks expected from surviving residue grains. Fig 4a shows an example of a residual grain on a crater rim that did show Ti and N signals. Comparison of this spectrum to that of a typical raw TiN projectile grain (Fig. 4b), with its stronger Ti and N peaks, demonstrates that thin surface coatings are likely obscuring Auger electrons from the underlying material. This suggests that efforts to remove the top few nm must first be undertaken to allow better Auger characterization of surviving grains on Al craters.

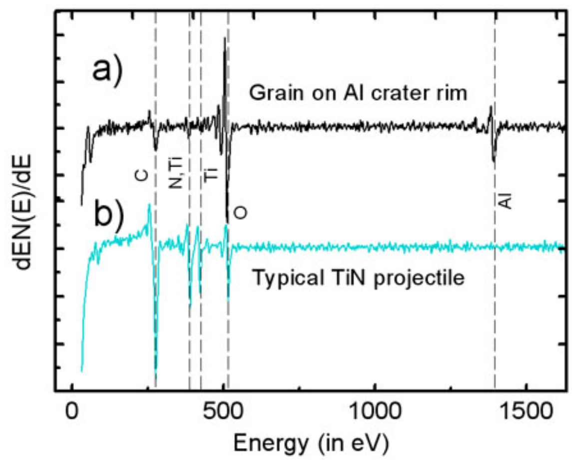

Fig 4. Derivative Auger energy spectra of a) grain on Al crater rim showing N and Ti peaks and b) typical raw TiN projectile showing considerably stronger N and Ti. The strong C peak from the TiN projectile is presumably due to thin surface contamination.

Conclusion: Our results showing the presence of abundant Ti-rich debris on the Al foils suggest that a significant portion of the refractory material (either TiC or TiN) does survive upon impact into Al foils, in agreement with results from [5]. Despite the dearth of SiC in the projectile mix, subsequent TEM studies of this Stardust calibration sample may yet yield relative survival probabilities of similarly refractory phases that are applicable to the survival of presolar SiCs.

References: [1] Stadermann F.J. et al. (2008) *MAPS 43*, 299. [2] Stadermann F.J. et al. (2009) *LPS* XL, *Abstract # 1188*. [3] Burchell M.J. et al. (1999) *Measure. Sci. Tech, 10*, 41. [4] Kearsley A.T. et al. (2007) *MAPS 42*, 191. [5] Leroux H. et al. (2008) *MAPS* 43, 143.