Stardust microcrater residue compositional groups

Conference or Workshop Item

STARDUST MICROCRATER RESIDUE COMPOSITIONAL GROUPS. J. C. Bridges[1], I. A. Franchi[2] and S. F. Green[2], [1]Space Research Centre, Dept. of Physics and Astronomy, University of Leicester, Leicester LE1 7RH UK, [2]Planetary and Space Sciences Research Institute, Open University, Milton Keynes MK7 6AA UK.

Introduction: Impact craters found across the *Stardust* foils have preserved residue from the cometary grains [1, 2, 3]. It has been successfully demonstrated that stoichiometric mineral compositions have been preserved in larger craters (e.g. >15 μm) including identified mineral grains [4]. Techniques used have been SEM EDS analyses of residue *in-situ* within craters and TEM EDS analyses of wafers extracted with FIB-SEM techniques [4]. Here we describe a project to extend quantitative mineral analyses to the more abundant smaller craters (e.g. 1-10 μm D_c). This has the potential to provide a representative dataset of quantitative *Stardust* mineral compositions.

Samples and Techniques: Over 260 craters within *Stardust* Al foils were analysed as part of the Preliminary Examination Team work [1, 2, 3]. A subset of this (105 craters with EDS analyses on foils C2008N, C2051N, C2054N, C2060N) was used in this study. Initial EDS analyses were made by SEM EDS with 15 kV, 0.5 nA beam current and acquisition times of 75 s. Analysing the residue in small craters (e.g. <10 μm D_c) is limited to qualitative results by the blocking effects of the crater walls and small residue volumes. We have used these results to define compositional groupings. Selected craters illustrating these compositional groups were then used for FIB-SEM extraction. Extracting craters and residue from out of the craters offers the chance to remove blocking effects and use EDS systems on SEM and TEM instruments. Most samples were analysed by EDS on SEM and TEM instruments in such a way to help EDS analysis rather than enhance imaging of the residue.

An FEI Quanta 200 3D dual Focussed Ion Beam/SEM was used. In order to preserve the <100 nm thick residues within the craters during analyses, Pt was deposited with the electron beam over the craters. Electron beam deposition is performed at low accelerating voltage and so damage to the underlying residue is minimised. Following that a thicker layer (~1 μm) of Pt was deposited over this using ion beam deposition. Wafers (≤ 1μm thick) were cut down through the Pt caps and craters using the FEI AutoFIB software control with parameters adjusted to minimize beam damage. Beam currents of ≤3.0 nA were found to provide a sufficient sputter yield within the Al foil. Wafers were extracted using an Ascend Instruments extraction system with Mo end effectors to hold ≤1 μm thick extracted wafers in the SEM and TEM. EDS analyses on the extracted crater with residue were then performed at 15-20 kV, 0.5-0.6 nA with a variety of acquisition times on the SEM. An INCA analytical system with appropriate mineral standards were used for these analyses. The wafers attached to Mo end effectors were also analysed by TEM (Jeol 200 kV with a sample holder made of Be to minimize the contribution of X-rays from it) using a standardless EDAX routine. TEM EDS with a small working distance allows a relatively high X-ray count rate on some of the smallest volumes of residue analysed. TEM EDS calibration was checked using silicate minerals extracted from polished blocks (e.g. see Results section below). A double tilt sample holder allowed optimum count rates, allowing sample orientations in the sample chamber that avoid the blocking of X-rays by the adjacent Mo end effector. The EDS analyses were used to determine the atomic proportions. This technique has been tested with residue-bearing impact craters prepared by light gas gun shots (prepared by M. Burchell, Kent University and A. Kearsley, NHM) and the full results will be reported in a subsequent publication.

Results: *FIB-SEM extraction of mineral standards and light gas gun craters.* Minerals from an equilibrated L-chondrite were analysed with this technique. Results accurate to 1-2 Mg# units were produced on olivine grains in sections of varying thickness, we regard this as a guide to the potential accuracy of this technique. Residue from pyroxene craters produced by a light gas gun [2] was $(MgCaFe)_{21}Si_{19}O_{60}$ showing that stoichiometry can be preserved during impact. Further analyses are underway to determine an accurate range of compositions from practice crater residues.

Table 1. Frequency of compositional groups in 1-15 μm D_c craters.

Residue	Frequency	%
Fe S	18	17.1
Fe Ni S	2	1.9
Silicate + sulphide	24	22.9
Cr-bearing	1	1.0
Mg Fe silicate	38	36.2
Mg Fe Ca silicate	7	6.7
K, Na silicates (+ sulphide)	3	2.9
Cl, Fe-bearing contamination	3	2.9
None/trace Si or Fe	8	7.6
Ti-bearing	1	1.0
Total	105	100

Residue compositional groups. The compositional groupings defined by the preliminary analyses of craters at the OU are given in Table 1. Most craters of >1 μm D_c have preserved some residue and at least 20% have a mixture of different phases e.g. sulphide and Mg-Fe silicate. One crater out of 105 was found to have a refractory, Ti-rich residue and this frequency is consistent with *Stardust* samples within the aerogel collectors [5].

FIB-SEM analyses of crater residue. Sections across craters have so far been successfully prepared and extracted from foils C2051N, C2054N, C2060N.

Mg-Fe type residue was extracted from a crater (9 μm D_c) in foil C2060N and found to be Fo99. No other residue composition was identified from this crater on the FIB section although qualitative spectra taken when the crater was *in-situ* indicate that Fe-S is also present in parts of the residue not sampled in the extracted section.

In foil C2054N both olivine (Fo77) and low-Ca pyroxene En95 were identified within one 1.7 μm D_c crater. Some analyses from this crater residue have non-stoichiometric (Mg+Fe)/Si ratios indicating that at the scale of EDS analyses on the extracted wafers there is a mixture of olivine and low-Ca pyroxene compositions.

Discussion: Our results indicate that FIB-SEM extraction technique can be successfully applied to <10 μm D_c craters and provide accurate atomic proportions of the major elements. The FIB-SEM results show that the Mg-Fe silicate compositional group defined in the initial EDS survey is composed of forsteritic olivine and low-Ca pyroxene or impact glass with those stoichiometric compositions. The presence of mineral grains within the residue of larger craters [4] suggests that at least some stoichiometric mineral analyses do represent minerals rather than glass.

The presence of identifiably different residue compositions (i.e. olivine and low-Ca pyroxene) within the same crater residue is consistent with the results of the entire *Stardust* crater qualitative EDS survey performed during the Preliminary Examination Team phase. That study suggested smaller craters are more likely than the largest craters to have more than one mineral type or glass composition within them [1].

The analyses determined so far are consistent with those shown from samples extracted from aerogel. For instance olivine compositions from aerogel samples range from Fo4-100 with a peak at Fo99 [5]. The peak of Fo99 is consistent with our crater analysis on foil C2060N. Low-Ca pyroxene analyses from the aerogel collector samples are from En52-100 with a peak at En95. The sulphide analyses summarised in [5] are also consistent with the predominance of Ni-poor Fe-S residue compositions in Table 1.

As we prepare and analyse more samples this technique should allow an accurate breakdown of the proportions of different phases in the smaller size range craters together with their compositions. This will provide another dataset of Comet Wild-2's mineral composition.

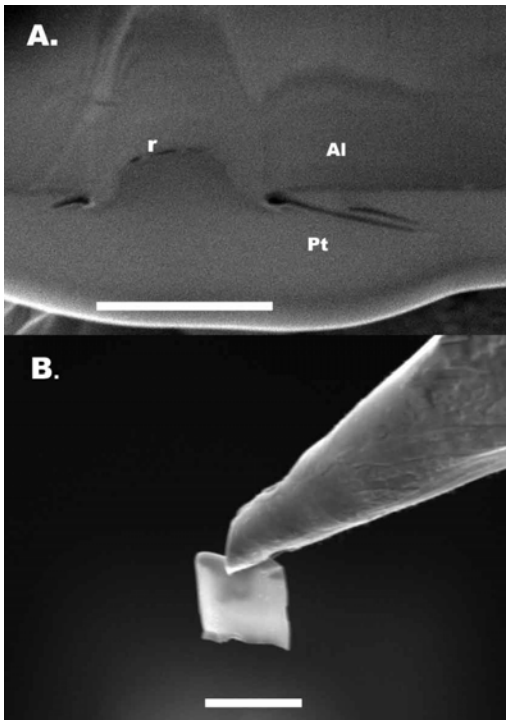

Fig. 1 Extraction of residue-bearing craters by FIB-SEM. A. Ion beam generated secondary electron image (SEI) of 0.8 μm thick wafer cut through 1.7 μm D_c crater in Al foil C2054N. Layers of Pt deposited by electron and ion beam deposition cover the residue-bearing layer (r) on the base of the crater. This residue contains Fo77 and En95 compositions. Scale bar 1.7 μm. B. SEI image of section through 9 μm D_c crater in foil C2060N showing the crater outline between deposited Pt and surrounding Al foil. The Mo end effector is holding the sample. Scale bar 10 μm.

References: [1] Borg J. et al. (2007) *LPS XXXVIII*, #1592. [2] Kearsley A et al. (2007) *Meteoritics & Planet. Sci.*, in prep.. [3] Hörz F. et al. (2006) *Science, 314*, 1716-1719. [4] Leroux H. et al. (2007) *Meteoritics & Planet. Sci.*, in prep.. [5] Zolensky M. E. et al. (2006) *Science, 314* , 1735.