Extraction and analysis of microcrater residues using focused ion beam microscopy

Conference or Workshop Item

EXTRACTION AND ANALYSIS OF MICROCRATER RESIDUES USING FOCUSED ION BEAM MICROSCOPY. J. C. Bridges[1] , I. A. Franchi[1] and S. F. Green[1], [1]Planetary and Space Sciences Research Institute, Open University, Milton Keynes, UK, j.bridges@open.ac.uk.

Introduction: Microcraters within *Stardust* aluminium foils have the potential to trap impact residues – probably molten or partially vapourised – of comet Wild 2 material [1]. Thus, in addition to the cometary particles captured by aerogel, microcraters may also reveal compositional information. In anticipation of this we have prepared a technique using focused ion beam microscopy to extract and analyse such residues.

Samples and Techniques: Dual focused ion and scanning electron beam microscopy (FIB/SEM) was performed using an FEI Quanta 200 3D. This technique combines analytical SEM with the ability to use a Ga^+ ion beam to mill material in a controlled way or deposit Pt metal strips from a Pt-bearing gas source. Tilting the stage at a fixed working distance allows both EDS analyses and ion beam milling to be performed on the same sample and facilitates extraction of wafers.

The cratered Al foils used in this work were donated by F. Horz (JSC-NASA) who performed the light gas gun experiments in which glass beads of known size distribution [1] and composition were fired at the 100 µm thick foils. We have studied foils which have experienced multiple shots (6 km/s) from 23 µm, 36 µm, 49 µm diameter glass beads. The foil (#2414) with the 49 µm bead shots was found to contain recognizable molten impact residues in some of the craters. Craters range up to 400 µm diameter and their size distribution is described in [1].

The glass spheres used in the light gas gun experiments have a distinctive Na-rich composition (11.9 wt% Na_2O) and thus the resultant impact residues can provide some constraints on the remobilisation or loss of volatiles during the impact. Their composition, with which we compare our results, was obtained from [2].

Silicate residues were identified within some of the craters by back-scattered and secondary electron imaging. The residue was milled using the Ga^+ ion beam at 30 kV accelerating voltage and beam currents which are reduced from 7 to 0.1 nA as 10 µm long, 4-5 µm deep, 1.5 µm thick wafers are progressively cut. Cutting was performed using a combination of the microscope's Auto-FIB routine and making individual milling trenches as necessary [3]. On the basis of experimenting with the extraction from microcraters and other materials, this thickness was found to be a good compromise between volume of sample desirable for subsequent energy dispersive analyses (EDS) and succcessful extraction. A tungsten needle, controlled with a Kleindiek nanomanipulator, was attached to the samples by deposition of a 1 µm thick strip of Pt. Once welded to the needle the wafers are raised from the crater and placed on the surrounding Al foil. The samples are then progressively milled at 0.1 nA beam current to provide smooth surfaces with reduced Ga and Pt contamination.

Energy dispersive analyses were performed at 20 kV accelerating voltage, ~1 nA beam current and a range of spot and raster sizes. A P&H Developments Geo Mark II mineral standard block was used for calibrations. In order to remove the effect of Al in the surrounding foil and Ga and Pt from the focused ion beam and Pt-deposition, residue analyses were normalized to 100% after subtraction of these elements.

Results: An example of an impact residue within a microcrater is shown in Fig. 1a. Subsequent stages in the technique are shown in 1b-c where the residue has been milled prior to extraction, is attached to a W needle and then placed on the adjacent Al foil for EDS analyses. We also place extracted wafers on Cu grids for transmission electron microscopy techniques but prefer to use the Al foil for EDS analyses as it provides a suitable flat substrate.

The impact residues identified in this study show signs of having been molten e.g. the smooth, lobate forms of the residue in Fig. 1a. Other impact residues have been placed on Cu grids in preparation for scanning transmission electron microscopy.

We are preparing secondary minerals standards which will be prepared to the same thickness as the extracted crater residues. This will be useful in assessing the effect of relatively small volumes (ie <2 mm thickness) on the quantification of our EDS analyses.

Composition of glass bead residues. Fig. 2 shows a comparison between the accepted composition of the glass beads and our analyses of residue in one of the craters (Fig. 1). The normalized EDS analysis is an average of 11 points taken at 4.5 µm spot size. The Na-rich glass composition has been preserved despite the effects of impact and melting.

The beads also contain 1.0 wt% Al_2O_3, 0.25 wt% SO_3 , 0.1 wt% Fe_2O_3 and 0.3 wt% K_2O [2]. These elements were not detected in the residue, except one analysis point which contained 1.1 wt% K_2O. The overall range of Na_2O contents 9.7 – 16.4 wt% and

MgO 6.5 – 10.8 wt% in the melted residue shows the variability in individual analyses that can be expected with this technique. Some of this variation may be due to element diffusion within the glassy residue. This is consistent with shock processes in chondrites where K is remobilized and can be enriched in some glassy mesostasis [4]. SiO_2 and CaO show smaller compositional ranges 70.6 – 76.9 wt% and 5.0 – 6.7 wt%.

Some of the difference between our analyses and the standard values (e.g. lower CaO contents) may be related to the small volume analysed by EDS. Preparation of mineral standards to the same thickness is being done to assess this issue.

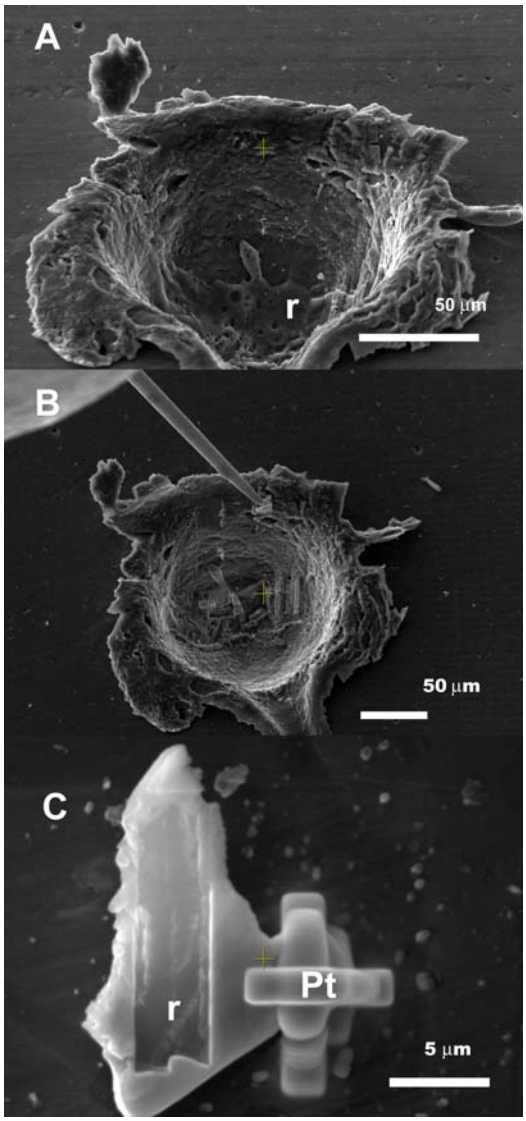

Figure 1. Secondary electron images of an example of microcrater residue extraction. A: Residue r can seen in the base of the crater. The texture indicates that it has been molten. This sample has been tilted at 52°. B: Extraction of a milled part of the residue on a W needle. Milled trenches can be seen in the residue. C: Residue (with Al and Pt contamination from foil and extraction process) attached with Pt straps to the Al foil adjacent to the microcrater. The area r contained the highest proportion of residue. The surface was milled with Ga^+ ion beam at low beam current to produce a surface suitable for EDS analyses.

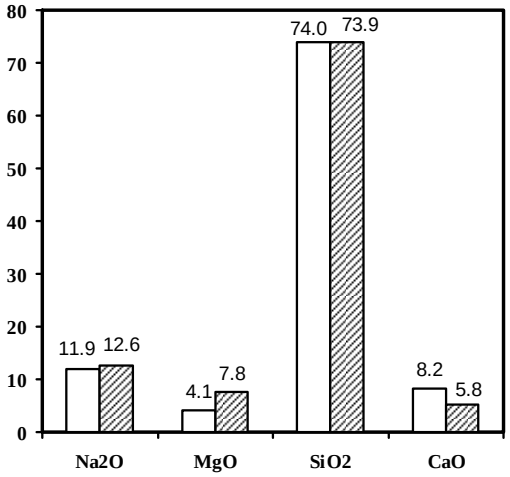

Figure 2. Mean composition (wt% oxide) of impact residue (grey columns) compared to standard value of pre-shot glass beads (white columns) [2]. Residue compositions are the normalized mean of 11 analyses in a residue-rich part of the extracted wafer (Fig. 1c). Al, Ga and Pt contamination has been subtracted from the analyses before normalisation.

Conclusions: By using a FIB/SEM technique we can extract and analyse microcrater residues from Al foils. The impact residue shows a variation in alkali contents but average compositions are similar to the glass bead impactor suggesting that this technique will be useful for analysing *Stardust* residues. We are using similar techniques to analyse particles separated from aerogel and produce samples mounted on Cu grids for transmission electron microscopy techniques.

References: [1] Horz F. et al. (2006) *LPS, XXXVII*. [2] www.whitehousescientific.com [3] Giannuzzi L. A. et al. (2005) in *Introduction to Focused Ion Beams*, Giannuzzi L. A. and Stevie F. A. (eds) 201-228. [4] Rubin A. E. (1985) *Rev. Geophys. 23*, 277-300.