CLEANING STRATEGIES AND DEPTH PROFILING OF GENESIS 60130 SILICON. Ian C. Lyon[1], Kimberley R. Kuhlman[2], and Donald S. Burnett[3], [1]The University of Manchester, Manchester, M13 9PL, UK (Ian.Lyon@manchester.ac.uk); [2]Planetary Science Institute, 1700 East Fort Lowell Blvd., Suite 106, Tucson, AZ 85719, (kim@psi.edu), [3]Division of Geological and Planetary Sciences, 100-23, California Institute of Technology, Pasadena, CA 91125 (burnett@gps.caltech.edu).

Introduction: We have previously reported on the analysis of fragments of the Genesis solar wind collector materials for contamination [1]. The crash landing of the Genesis collector capsule on its return to Earth shattered the ultra-pure collectors and coated them with Utah desert sands and mud. Strategies have had to be evolved to clean the contaminants from the surfaces of the fragmented collector pieces without compromising or contaminating the implanted solar wind atoms. At LPSC 2010 we reported on a study of particulate contamination of silicon sample 60130 using a Zeiss 1530 SEM with the unique capability of an in-lens detector and a TOFSIMS (time-of-flight secondary ion mass spectrometry) elemental analysis of the same areas to gauge the relative merits of each analytical technique and a measure of the particulate contamination remaining upon the detector material after cleaning.

The initial cleaning strategy involved megasonification in ultra-pure water, ozone treatment followed by HF/HCl. Micrometer scale particulates and nanometer-thin films containing O, Fe and Ca were detected by both techniques although TOFSIMS further identified particulates and films embedded into the sample surface.

Further sample cleaning and analysis

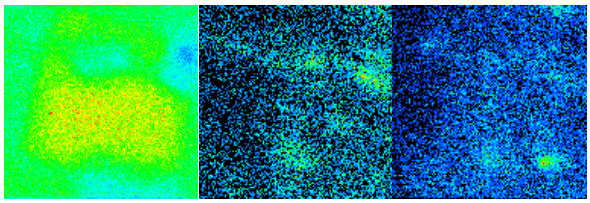
Following this discovery, sample 60130 was further cleaned at Caltech using HCl and then in hot xylene to remove any organic contamination. Analysis by the Wisconsin Zeiss SEM detected no particulate contamination in the previously analyzed areas.

The sample was then analyzed using TOFSIMS at the University of Manchester using a focused pulsed gold ion beam (spot size ~ 1 micrometer) to generate secondary ions for time of flight mass analysis.

TOFSIMS analysis

TOFSIMS analyses of the previously analyzed areas were found to still display thin film and particulate contamination, often buried within the sample surface at depths of a few nm.

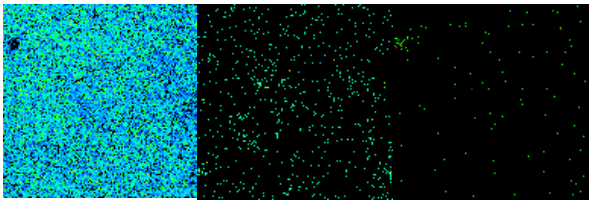
Firstly the area was analyzed with no pre-sputtering by the primary ion beam. Elemental images of a 152 micrometer sized area encompassing the previously analyzed areas is shown below. (The areas previously analyzed were sputtered and here show higher yields of Si as surface contamination and a covering oxide layer were removed during the previous analysis.)



Si Na Ca

Figure 1. Secondary ion images of the previously analyzed area reported in LPSC 2010 #1822 (shows as yellow in the Si image). Field of view 152 micrometers.

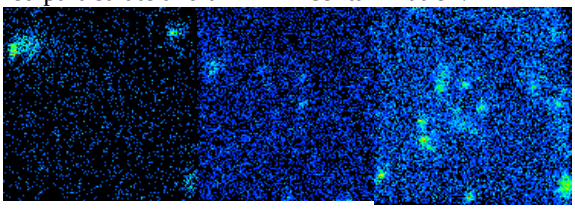
Analyses were also obtained of an adjacent area which had previously been unanalyzed.



Si Ca Al

Figure 2. Adjacent 200micrometer area showing little contamination on the surface.

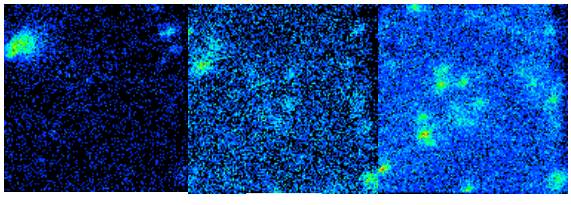
Subsequent depth profiling of the area exposed buried particulate and thin-film contamination.



Al Na Ca

Figure 3. Same area as in figure 2 but with <1nm depth of the surface removed.

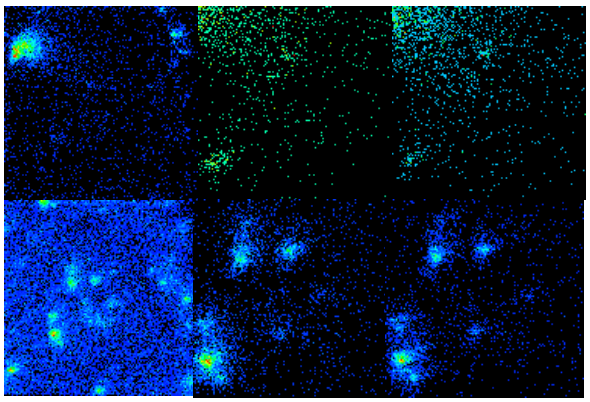
Subsequent depth profiling of the same surface showed that the particulate/thin-film contamination becomes more pronounced with depth.



Al Na Ca

Figure 4. Same area as figure 3 but 0.2nm deeper

The thin-film nature of the contamination can be seen in a sequence of analyses, Al in the first 3 images and Ca in the second 3 images. These follow on in depth from the images shown in figures 3 and 4. The latter 2 images are from a 100micrometer field of view



0.6nm 1.2nm 1.4nm

Figure 5. Successive elemental ... Al first row, Ca second row) with depth into ... ce.

An alternative explanation to the particulates being present at depth within the surface is that they are covered by water and hydrocarbon contamination following exposure to the atmosphere and that this is removed during sputtering. High spatial resolution imaging, selecting regions of interest may avoid particulate contamination but it is clear that particulate contamination is present at the surface.

Solar wind detection by TOFSIMS

TOFSIMS holds the potential for the simultaneous analysis of solar wind depth profiles of different elements. Simultaneous profiles of different solar wind elements would be very valuable as the detailed implantation depths could be directly compared and hence the energies of different elements be determined. However, these contaminant issues have so far prevented the successful unambiguous detection of the solar wind signal in silicon using TOFSIMS. Mg and Fe solar wind profiles have been successfully acquired using ion probes with direct current primary ion beams [2] and the Mg solar wind profile detected by TOFSIMS in a diamond collector sample [3]. However, the silicon collector material is still proving difficult to analyse with TOFSIMS. .

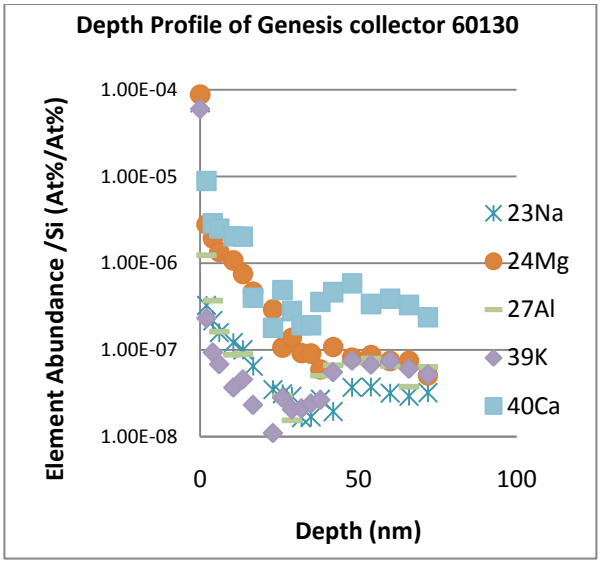


Figure 6. Simultaneous depth profiles into silicon sample 60130 obtained after further HCl cleaning of the surface.

The different elements at <50nm depth are still decreasing in abundance at a level above the known solar wind abundance which should be peaking at that depth. Abundances at depths approaching 100nm are an order of magnitude lower than the expected solar wind abundance for Mg so demonstrating that background levels can be low enough to detect the solar wind abudances but measured levels at depths of <50nm preclude measurement of the solar wind by this technique at present.

References:

[1] Kuhlman et al., 41st LPSC (2010) #1822, [2] Jurewicz et al., LPSC XXXIX (2008) #2272, [3] Claydon et al., LPSC XXXIX (2008) #1727

Acknowledgements

Support from the Science and Technologies Facilities Council (UK) is gratefully acknowledged as is technical help from T. Henkel, D. Rost and D. Blagburn.