GENESIS SOLAR WIND COLLECTOR CLEANING ASSESSMENT: 60366 SAMPLE CASE STUDY.

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Introduction: In order to recognize, localize, characterize and remove particle and thin film surface contamination, a small subset of Genesis mission collector fragments are being subjected to extensive study via various techniques [1-5]. Here we present preliminary results for sample 60336, a Czochralski silicon (Si-CZ) based wafer from the bulk array (B/C).

History of sample 60336: This sample has undergone multiple cleaning steps (see insert below), including UPW and aggressive chemical cleanings, as well as optical and chemical (EDS, ToF-SIMS) imaging.

2/26/2007	UPW cleaned 5min @40C at JSC
5/14/2013	Imaged using DM6000M at JSC
7/31/2013	SEM analysis at PSI
8/1/2013	Imaged using DM6000M at JSC
8/6/2013	UPW cleaned and imaged at JSC
8/13/2013	Aqua regia and hot xylene at Caltech
9/12/2013	Imaged using DM6000M at JSC
9/16/2013	UPW cleaned and imaged at JSC
10/14/2013	ToF SIMS analysis at Smithsonian
10/21/2013	Optical imaging at Smithsonian
11/12/2013	Low-vacuum nanoSEM at Smithsonian

Results: Contamination appeared on the surface of 60336 after the initial 2007 UPW cleaning, presumably produced by reaction of a crash-derived contaminant with water. Figure 1 shows the sample after aqua regia and xylene treatment which did little to remove contaminants revealed by the PSI SEM examination (7/31/13). The sample was UPW cleaned for the third time and imaged (9/16/13; Fig. 2). The UPW removed the dark stains that were visible on the sample. However, some features, like "the Flounder" (a large, 100 micron feature in Fig. 3) appeared largely intact, resisting all cleaning efforts.

High-vacuum SEM imaging showed that there are numerous feature in the Flounder, including: (1) particles (possibly, non-conductive - charging in Fig. 6b), (2) thin films, and (3) an enrichment in C (Fig. 6c).

ToF-SIMS imaging has been useful of this technique for the characterization of contamination on Genesis samples [1]. Figures 4 and 5 are ToF-SIMS ion images (positive and negative) of the Flounder. The prominent square around the feature is an outline of the area scanned with high-vacuum SEM. From ToF-SIMS analysis it is clear that the SEM deposited various hydrocarbons in the scanned area. There is also an apparent enrichment in F and Cl in the feature, as well as enrichment in Ca around it. Hydrocarbons obscured the speciation of the Ca compound; however, correlation of Ca+ and CaOH+ localizations suggest Ca oxide and/or hydroxide.

Ar sputtering (Fig. 5) removed most hydrocarbons, Na, as well as much of the Ca – suggesting that these

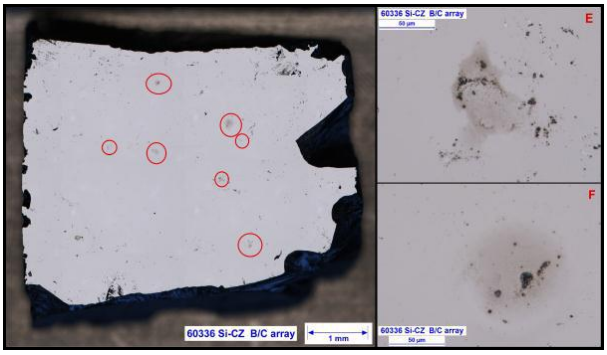

Fig. 1: Sample 60336 after aqua regia and hot xylene treatment (cf., 9/12/2013). The red circles indicate contamination added after the 8/6/13 imaging. Images on the right (E and F) show contamination at two different positions.

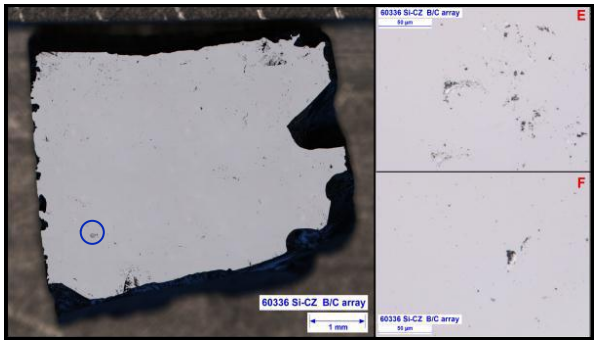

Fig. 2: Sample 60336 after UPW cleaning (9/16/13). Right: images showing the dark stains (Fig.1) are no longer visible.

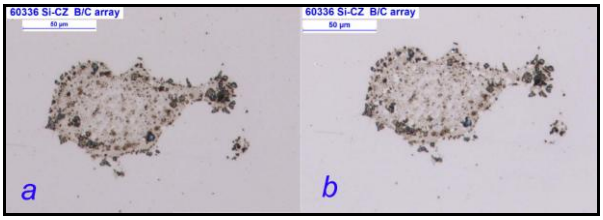

Fig. 3: Images of "the Flounder" contamination possibly added during initial UPW cleaning (Fig. 2 blue circle). (a) after SEM analysis and UPW cleaning; b) after aqua regia + hot xylene treatment, followed by UPW cleaning.

were likely present as a very thin surficial film.

Cl may be a compositional part of this contamination as ion images show mobilization of Cl in the areas of high-resolution SEM x-ray mapping (tail of the Flounder). Signal from CN- is likely an artifact, but indicates the area of overall C-enrichment. No Ca or CaOH was detected outside of the Flounder.

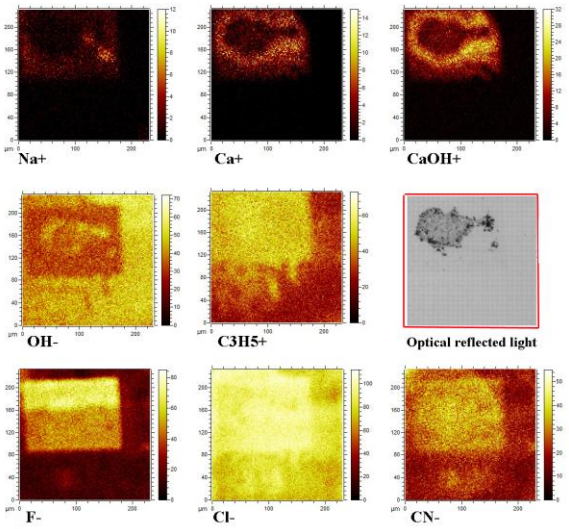

Fig. 4 ToF-SIMS images of "Flounder" before Ar sputtering.

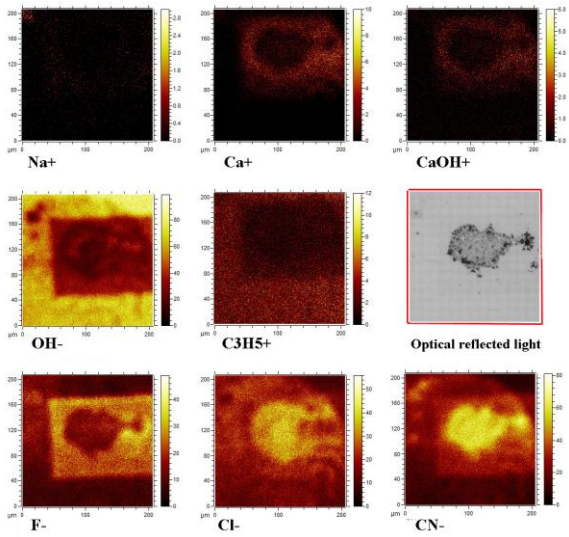

Fig. 5: ToF-SIMS images of "Flounder" after Ar sputtering.

Low-vacuum SEM analyses of individual features within Flounder are presented in Table 1. Si values are high because the EDS excitation volume (3kV beam) is significantly larger than thickness of the contamination. Some compositional correlations with surface features can be observed: Points 2, 13 lay outside of Flounder; "white" specs (points 5, 10) have low C/O ratio, possibly Ca oxide or hydroxide; "black" specs (points 3, 9, 6, 12) have high C/O ratio, possibly Ca carbonate.

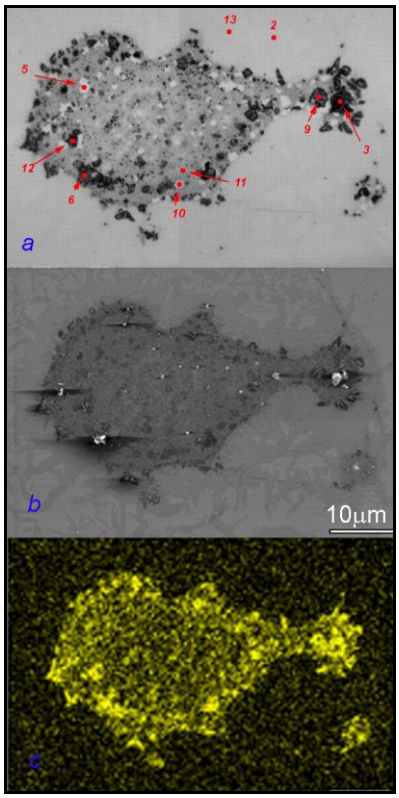

Fig. 6: a) Optical image of Flounder. b) SEM BSE (High vacuum) image. c) High-vacuum EDX X-ray map of Carbon.

Table 1.

Point	Element Wt. %					
	C	O	Al	Si	Ca	Total
2	2.23	0.63	0	96.6	0.54	100
13	1.3	1.68	0	94.55	2.46	100
5	1.22	1.58	0	96.98	0.22	100
10	1.33	1.28	0.67	94.82	1.89	100
3	17.81	5.06	0	76.98	0.15	100
6	20.93	4.19	0	74.72	0.16	100
9	10.31	2.45	0	87.24	0	100
12	8.88	2.9	0	87.59	0.63	100

Conclusions: UPW and aqua regia/hot xylene treatment were not especially effective in removing surface contamination from 60336. It is conceivable that hydrocarbons deposited during high-vacuum SEM x-ray mapping may shield some of the contamination from acid treatment. Test experiments are under way.

References: [1] Goreva Y. S. and Burnett D. S. (2013) *LPSC 44*, Abstract #2109. [2] Kuhlman K. R. and Burnett D. S. (2007) *LPSC 38*, Abstract #1920. [3] Schmeling M. et al. (2013) *LPSC 44*, Abstract #2465. [4] Calaway M. J. et al. (2007) *LPSC 38*, Ab-stract #1627. [5] Calaway M. J. et al. (2009) *LPSC 40*, Abstract #1183.