

GENESIS SOLAR WIND COLLECTOR CLEANING ASSESSMENT: UPDATE ON 60336 SAMPLE CASE STUDY. Y.S. Goreva¹, K. K. Allums², C.P. Gonzalez², A.J. Jurewicz³, D.S. Burnett⁴, J.H. Allton⁵, K.R. Kuhlman⁶, D. Woolum⁷. ¹Smithsonian Institution, Washington, DC, *gorevay@si.edu*, ²Jacobs-JETS at NASA/J, Houston, TX, ³Arizona State University, Tempe, AZ, ⁴California Institute of Technology, Pasadena, CA, ⁵NASA/JSC, Houston, TX, ⁶Planetary Science Institute, Tucson, AZ, ⁷California State University, Fullerton, CA.

Introduction: To maximize the scientific return of Genesis Solar Wind return mission it is necessary to characterize and remove a crash-derived particle and thin film surface contamination. A small subset of Genesis mission collector fragments are being subjected to extensive study via various techniques [1-6]. Here we present an update on the 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 the table below): UPW spin wash, aggressive chemical cleanings (including aqua regia, hot xylene and RCA1), 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
11/12/2014	Imaged using DM6000M at JSC
11/24/2014	10 min RCA1 cleaning at Dartmouth
12/2/2014	25 min RCA1 cleaning at Dartmouth
12/4/2014	Imaged using DM6000M at JSC
12/4/2014	UPW clean 5min, 40C at JSC
12/4/2014	Imaged using DM6000M at JSC
12/18/2014	ToF SIMS analysis at Smithsonian

Results: Contamination appeared on the surface of 60336 after the initial 2007 UPW cleaning. Aqua regia and hot xylene treatment (8/13/2013) did little to remove contaminants [7]. The sample was UPW cleaned for the third time and imaged (9/16/13). 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. 1b) appeared largely intact, resisting all previous cleaning efforts. These features were likely from mobilized adhesive, derived from the Post-It notes used to stabilize samples for transport from Utah after the hard landing. To remove this contamination, an RCA step 1 organic cleaning (RCA1) was employed.

The RCA is a standard semi-conductor procedure for removing contaminants from silicon wafers. Step 1 removes organic residue and films, using H₂O₂-NH₄OH-H₂O. NH₄OH removes organics; H₂O₂ keeps the silicon from dissolving in the NH₄OH.

An initial 10 min treatment did not remove the flounder, but, possibly, made it thinner. A further 25 min treatment visibly removed the Flounder (except for imbedded grains/pits), as well as a number of previously described contaminants ([7], Figs 1, 2, 5): CMgBr grain in position 2 is gone, some (if not all) AIOs and many Si particles are gone.

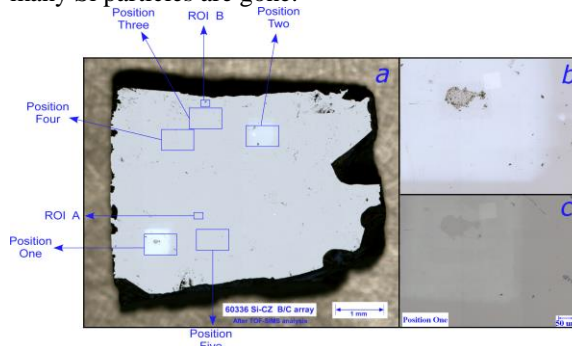


Fig 1. Sample 60336 with labeled JSC-monitored areas a) Optical imaging on 11/12/2014; b) Position One on 11/12/2014; c) Position One on 12/4/2014 after RCA1 treatment

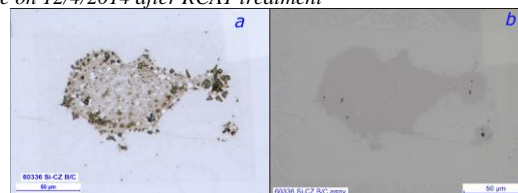


Fig 2. Optical images of the “Flounder” a) before, b) after RCA1.

Figures 3 and 4 are positive ion images of Position One area before and after RCA1 treatment (note different magnification in Fig 3 and 4). The prominent square around the Flounder in Fig 3 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. RCA1 treatment removed the Flounder, most of Ca-rich halo around it, AIO (2 particles in Fig3), and a K-Na particle. Remaining particles within the Flounder are likely embedded Si (Fig8a,b). Figures 6 and 7 are ion images of Position Two (Fig 5). Here we can see the removal of smaller AIO and Na-K particulates as well, however, two big-

ger features remain (although did become smaller). These features are not pits as evidenced by SEM SE imaging (Fig 8 c, d) but, surprisingly still carry a “brown stain” chemical signature (SiC₃H₉⁺).

Conclusions: Although we are still uncertain on the nature of the Flounder and why it is resistant to UPW and aqua regia/hot xylene treatment, we have found RCA1 to be suitable for its removal. It is likely that the glue from sticky pads used during collector recovery may have been a source for resistant organic contamination [9]; however [8] shows that UPW reaction with crash-derived organic contamination does not make particle removal more difficult.

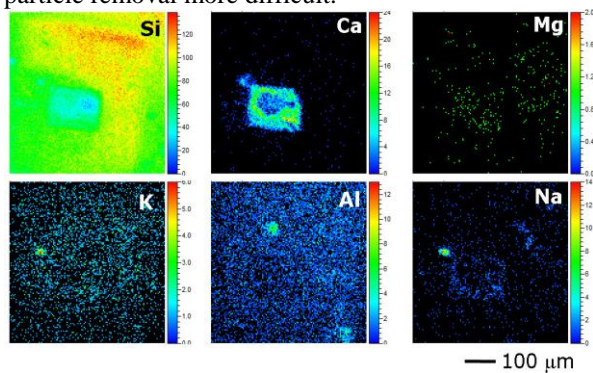


Fig 3. ToF-SIMS ion imaging of Position One before RCA1 treatment. Field of view 500μm. Color scale reflects relative intensity from low (blue) to high (red)

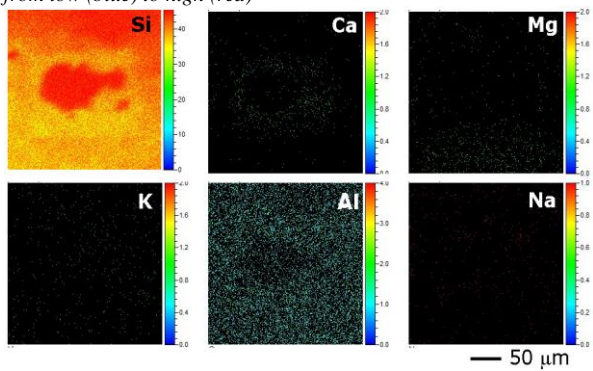


Fig 4. ToF-SIMS ion imaging of Position One after RCA1.

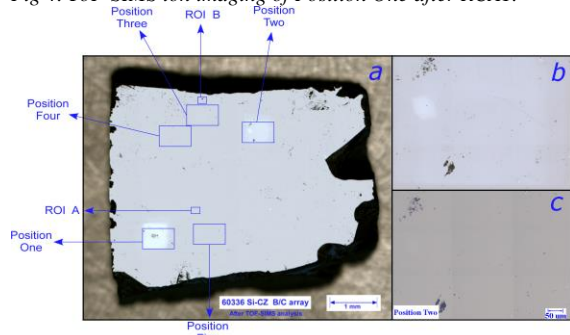


Fig 5 Sample 60336 with labeled JSC-monitored areas a) Optical imaging on 11/12/2014; b) Position Two on 11/12/2014; c) Position One on 12/4/2014 after RCA1 treatment

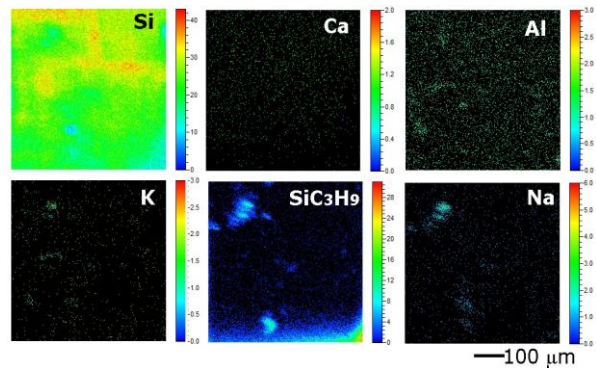


Fig 6 ToF-SIMS ion imaging of Position Two before RCA1.

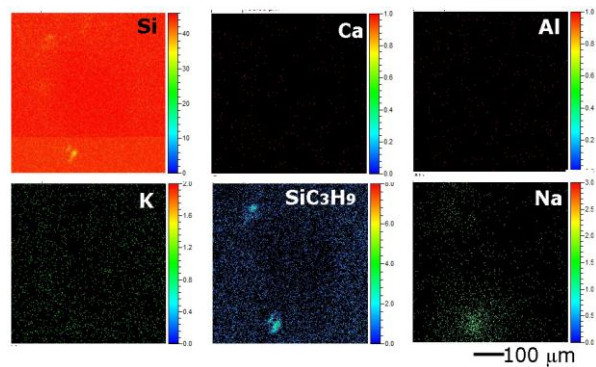


Fig 7. ToF-SIMS ion imaging of Position Two after RCA1.

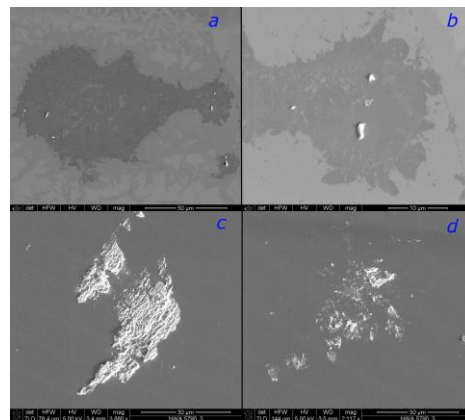


Fig 8. Post-RCA SEM SE imaging of a) Flounder, FOV 120μm, b)close-up of the Flounder's tail, FOV 45 μm, c) Lower large feature in Fig 5b,c, FOV 80 μm d) upper feature in Fig 5b, FOV 120 μm.

References: [1] Goreva Y. S. and Burnett D. S. (2013) *LPS* 44, 2109. [2] Kuhlman K. R. and Burnett D. S. (2007) *LPS* 38, 1920. [3] Schmeling M. et al. (2013) *LPS* 44, 2465. [4] Calaway M. J. et al. (2007) *LPS* 38, 1627. [5] Calaway M. J. et al. (2009) *LPS* 40, 1183. [6] Goreva Y. et al., (2014) *LPS* 45, 2568. [7] Goreva Y. et al., (2014) *LPS* 45, 2127. [8] Allton J. et al, (2015) *LPS* 46, 1896. [9] Allums et al (2015) *LPS* 46