

DEVELOPMENT OF GENESIS SOLAR WIND SAMPLE CLEANLINESS ASSESSMENT: INITIAL REPORT ON SAMPLE 60341 OPTICAL IMAGERY AND ELEMENTAL MAPPING . C.P. Gonzalez¹, Y.S. Goreva², D.S. Burnett³, D. Woolum⁴, A.J. Jurewicz⁵, J.H. Allton⁶, M.C. Rodriguez⁷, and P.J. Burkett¹. ¹Jacobs-JETS at NASA/Johnson Space Center, Houston, TX 77058; carla.p.gonzalez@nasa.gov, ²Smithsonian Institution, Washington, DC, ³California Institute of Technology, Pasadena, CA, ⁴California State University, Fullerton, CA ⁵Arizona State University, Tempe, AZ, ⁶NASA/Johnson Space Center, Houston, TX 77058, ⁷Geocontrol Systems-JETS at NASA/Johnson Space Center, Houston, TX 77058.

Introduction: Since 2005 the Genesis science team has experimented with techniques for removing the contaminant particles and films from the collection surface of the Genesis fragments. A subset of ~40 samples have been designated as “cleaning matrix” samples. These are small samples to which various cleaning approaches are applied and then cleanliness is assessed optically, by TRXRF, SEM, ToF-SIMS, XPS, ellipsometry or other means [1-9]. Most of these samples remain available for allocation, with cleanliness assessment data. This assessment allows evaluation of various cleaning techniques and handling or analytical effects.

Cleaning techniques investigated by the Genesis community include acid/base etching, acetate replica peels, ion beam, and CO₂ snow jet cleaning [10-16]. JSC provides surface cleaning using UV ozone exposure and ultra-pure water (UPW) [17-20]. The UPW rinse is commonly used to clean samples for handling debris between processing by different researchers. Optical microscopic images of the sample taken before and after UPW cleaning show what has been added or removed during the cleaning process.

Sample 60341 optical imaging: Genesis flight sample 60341 is a Czochralski silicon (Si-CZ) wafer from the bulk solar wind array (B/C). Cleaning or assessment processes applied are shown in Table 1. Sample 60341 was implanted with ²⁵Mg (100 kV, 10¹²/cm²) and ⁵⁴Fe (190 kV, 10¹²/cm²). Optical imaging was used to document both removal and addition of contaminants between steps (cf., Figs. 1- 6).

Table 1.

DATE	PROCESS	Step
2/21/2007	UPW	1
6/12/2007	²⁵ Mg implant, SIMS analysis	2
5/1/2011	⁵⁴ Fe implant, SIMS analysis	3
6/22/2011	UV ozone	4
3/11/2013	ToF-SIMS	5
8/21/2013	UPW	6
8/28/2013	HCl, hot xylene	7
9/4/2013	UPW	8

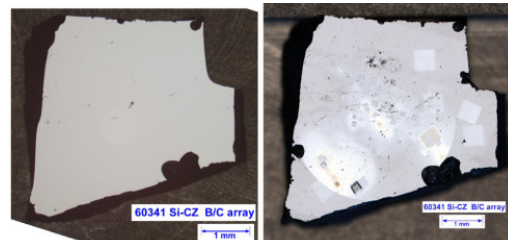


Fig. 1: (left) Image taken after first UPW cleaning - Step 1. (right) Image taken after ToF-SIMS analysis Step 5. Note handling debris.

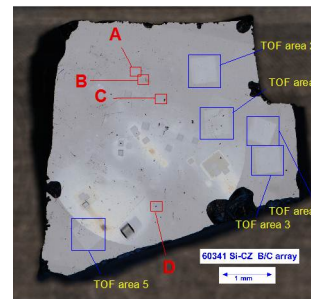


Fig. 2: Location of high magnification optical images and ToF-SIMS scans. Overall image taken after 2nd UPW cleaning- Step 8. The bright areas are oxidation introduced during SIMS analysis.

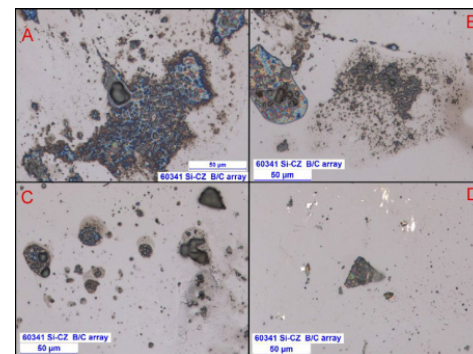


Fig. 3: Sample 60341 at positions A-D (cf., Fig. 2), following ToF-SIMS analysis of an ion implanted sample – Step 5.

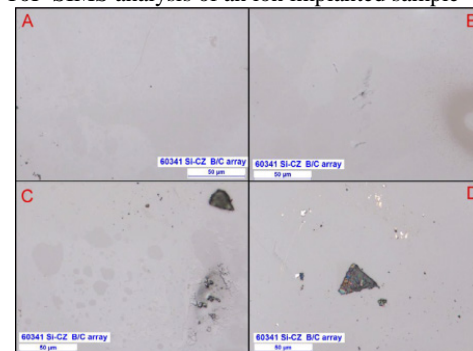


Fig. 4: Sample positions A-D after UPW – Step 6. UPW effectively removed handling debris documented in Fig. 3.

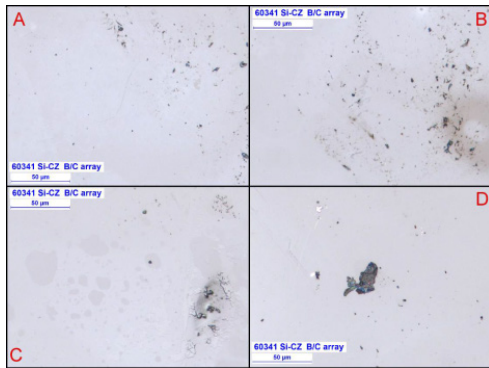


Fig. 5: Sample 60341 at positions A-D following acid cleaning, hot xylene treatment, and ultrasonic cleaning – Step 7 – prior to UPW. Contamination was added to A and B, but removed in C and D (cf., Fig. 4).

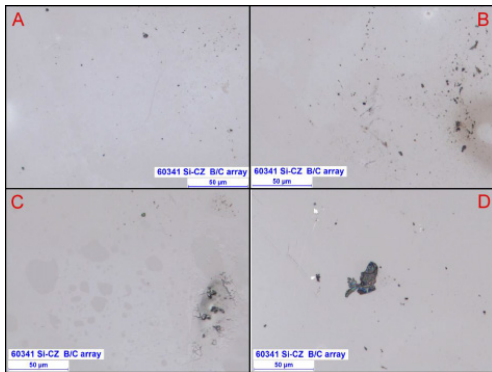


Fig. 6: Sample 60341 at positions A-D after UPW cleaning – Step 8. Contamination from Step 7 handling was partially mitigated in A and B, but there were few visible changes in C and D.

Sample 60341 ToF-SIMS: The field of view in Fig. 7 was evaluated by ToF-SIMS (Area 1, Step 5). After argon sputtering (5 sec, 25nA) to remove hydrocarbons, the elemental mapping shows: 1) a large Na-K-Mg-Ca-Al-Fe-Li-Cl smudge, with some Cs; 2) Fe-Cr scratch, 3) indium streak; 4) several pits filled with silicone; and 5) relatively less particle contamination. This initial set of ToF-SIMS scans is useful for improving the clean handling and for testing correlation between optical images and elemental mapping. A post-UPW-cleaning set of ToF-SIMS scans is in work with a now optically cleaner sample.

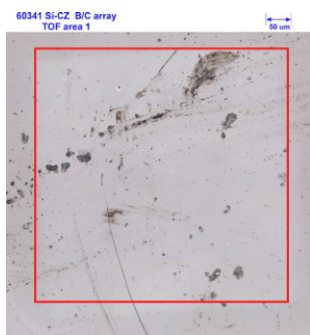


Fig. 7: (left). Optical image of ToF SIMS Area 1. Compare to ToF SIMS composition data in Fig. 8.

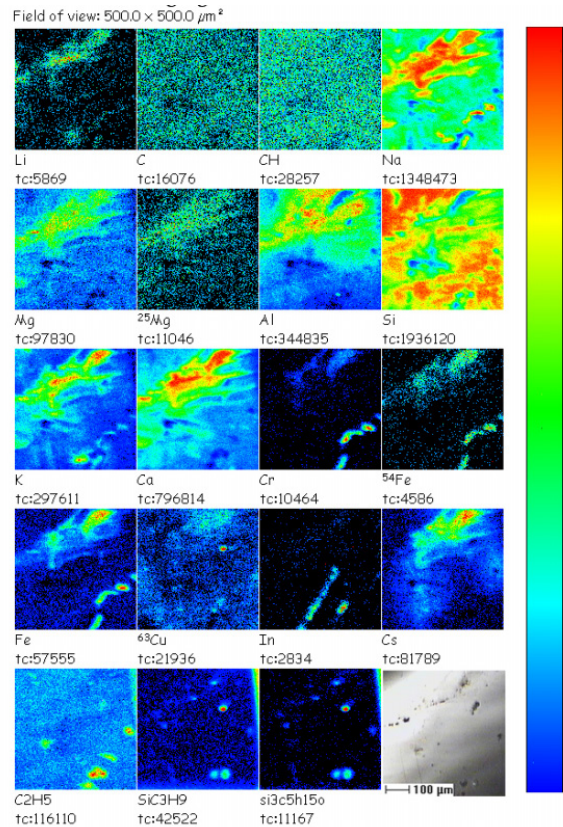


Fig. 8: ToF-SIMS mapping of Area 1 after argon sputtering, positive ion imaging

Conclusion: UPW cleaning, which is effective at removing crash-derived particulates >5 microns, is also useful between more rigorous aqueous cleaning processes and assessment analyses. The ToF-SIMS mapping of the optically cleaner surface, now in progress, should be a benchmark for achievable cleanliness.

References: [1] Brennan S. *et al.* (2006) *LPS XXXVII*, # 2029. [2] Kitts K. *et al.* (2006) *LPS XXXVII*, #1451. [3] Kuhlman K. *et al.* (2010) *LPS 41st*, #1822. [4] Schmeling M. (2010) *LPS 41st*, #1682. [5] Lyon I. *et al.* (2011) *LPS 42nd*, #2528. [6] Schmeling *et al.* (2011) *LPS 42nd*, #2041. [7] Schmeling M. (2012) *LPS 43rd*, #2209. [8] Goreva Y. S. and Burnett D. S. (2013) *LPS 44th*, #2109. [9] Kuhlman K. *et al.* (2012) *LPS 44th*, #2930. [10] Lauer H. *et al.* (2005) *LPS XXXVI*, #2407. [11] Huang S. *et al.* (2006) *LPS XXXVII*, #2440. [12] Kuhlman K. and Burnett D. S. (2007) *LPS XXXVIII*, #1920. [13] King B. *et al.* (2010) *LPS 41st*, #1975. [14] Veryovkin I. *et al.* (2012) *LPS 43rd*, #2732. [15] Veryovkin I. *et al.* (2013) *LPS 44th*, #2970. [16] Schmeling M. *et al.* (2013) *LPS 44th*, #2465. [17] Allton J. *et al.* (2007) *LPS XXXVIII*, #2138. [18] Allton J. *et al.* (2006) *LPS XXXVII*, #2334. [19] Calaway M. J. *et al.* (2007) *LPS XXXVIII*, #1627. [20] Calaway M. J. *et al.* (2009) *LPS 40th*, #1183.