

SMALL PARTICULATE CONTAMINATION STUDY OF GENESIS FLIGHT SAMPLE 61423. K. R. Kuhlman¹, M. Schmeling², C. P. Gonzalez³, K. K. Allums⁴, J. H. Allton⁵, and D. S. Burnett⁶; ¹Planetary Science Institute, 1700 East Fort Lowell, Suite 106, Tucson, AZ 85719; kim@psi.edu, ²Loyola University Chicago, 408 Flanner Hall, 60660, ³Jacobs, NASA Johnson Space Center, Houston, TX 77058, USA, ⁴HX5, LLC – Jacobs JETS Contract, NASA Johnson Space Center, Houston, TX 77058, ⁵NASA/Johnson Space Center, Houston, TX 77058; ⁶California Institute of Technology, Pasadena, CA, 91125.

Introduction: The Genesis mission collected solar wind and brought it back to Earth in order to provide precise knowledge of solar isotopic and elemental compositions. The ions in the solar wind stop in the collectors at depths on the order of 10 to a few hundred nanometers. This shallow implantation layer is critical for scientific analysis of the composition of the solar wind and must be preserved throughout sample handling, cleaning, processing, distribution, preparation and analysis.

We continue to work with the community of scientists analyzing Genesis samples using our unique laboratory facilities -- and, where needed, our unique cleaning techniques -- to significantly enhance the science return from the Genesis mission. This work is motivated by the need to understand the submicron contamination on the collectors in the Genesis payload as recovered from the crash site in the Utah desert, and -- perhaps more importantly -- how to remove it. We continue to evaluate the effectiveness of the wet-chemical “cleaning” steps used by various investigators, to enable them to design improved methods of stripping spacecraft and terrestrial contamination from surfaces while still leaving the solar-wind signal intact.

Cleaning History of Genesis Sample 61423: We have performed an extensive iterative study of cleaning procedures on Genesis sample 61423, a piece of CZ silicon from the B/C array. Optical imaging was performed at NASA JSC following each step. The sample was cleaned at NASA JSC prior to each analysis [1, 2]. After TRXRF analysis, the sample was cleaned using two cellulose acetate extraction replicas, as described previously [3]. The sample was returned to Loyola University for a post-replica analysis using TRXRF. The sample was then extensively imaged using secondary electron microscopy (SEM) with energy dispersive X-ray spectroscopy (EDS), with particular emphasis on finding small particles remaining in areas that appear “clean” under optical microscopy. A final analysis was performed using TRXRF.

TRXRF Analyses: We performed TRXRF analyses to determine if the cellulose acetate extraction replicas leave any residue that might interfere with large area solar wind analyses. TXRF is a surface sensitive analytical method with detection limits in the lower ppb to upper ppt range. Its non-destructive nature make it an ideal tool for repeated surface observation of Genesis samples, e.g. after different cleaning

procedures were applied to the samples. In TXRF an incident X-ray beam strikes the mirrorlike surface of the sample at an extremely small angle and is totally reflected. Trace contaminants on the surface will generate fluorescence, which is recorded with a Si-drift fluorescence detector. Elements from aluminum onward are accessible with this method.

Analyses before and after the application of the replicas are shown in Figure 1. The third spectrum taken after further IPA and UPW cleaning is shown in Figure 2 for comparison. Two observations can be made. First, it does not appear that the extraction replicas leave contamination on the surface that would skew large area analyses. Second, multiple cleaning steps appear to reduce the germanium contamination on the surface.

Analysis of Extraction Replicas: The extraction replicas were examined using SEM and EDX to see what types of particles were removed from the surface of 61423. The SEM image of an example of a silicon feature that was removed is shown in Figure 3. EDS mapping shows that the replica has removed a great deal of silicon from the feature, which apparently incorporated particles of aluminum, germanium, gallium and calcium during the crash.

Small Particle Survey: An area of the sample that appeared “clean” using optical microscopy was selected to see what types of very small particles remain after multiple cleaning steps (Figure 4). 53 particles were analyzed using EDS with a 10 keV electron beam. The results are shown in Figure 5 along with the results obtained for silicon 60205 and 60206 by Allton, et al. (2009) [4]. Silicon is the most predominant contaminant remaining.

Results: We have shown that extraction replicas can efficiently clean Genesis samples that cannot be exposed to water. Despite many cleaning steps, a number of very small particles remain. However, most of these are silicon, which may not drastically affect large area analyses because they are likely ultrapure materials from pulverized collectors.

References: [1] Calaway, M. J., et al. (2009) LPSC XL Abstract #1183. [2] Allums, K. K., et al. (2015) LPSC XLVI, Abstract #2014. [3] Kuhlman, K. R., et al. (2014) LPSC XLV, Abstract #2030. [4] Allton, J. H., et al. (2007) LPSC XXVIII, Abstract #2138.

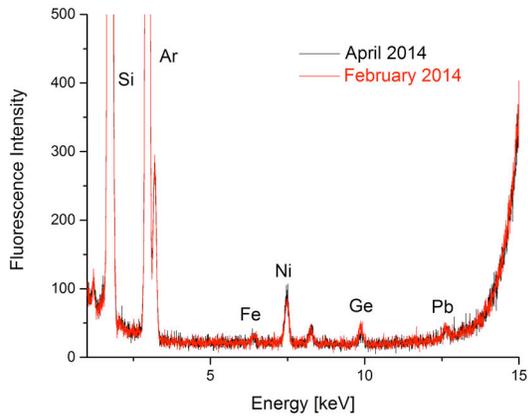


Figure 1. TRXRF analyses of sample 61423 before (red) and after (black) cellulose acetate replica cleaning.

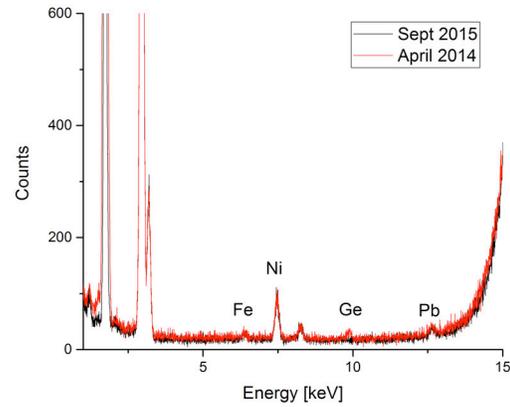


Figure 2. TRXRF analyses of sample 61423 before (red) and after (black) additional UPW and IPA cleaning steps. Note the reduction of Ge contamination.

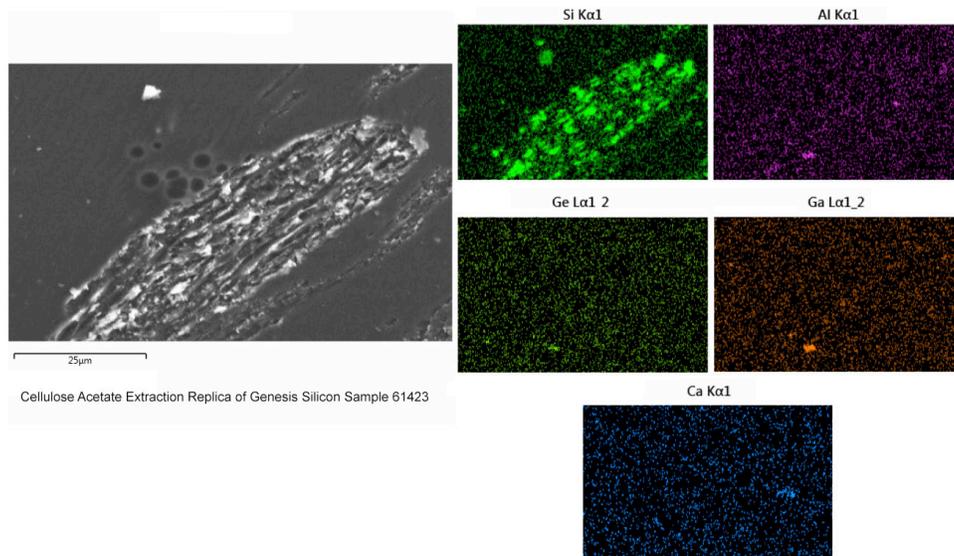


Figure 3. SEM image of a cellulose acetate extraction replica of Genesis sample 61423 and corresponding EDS maps for silicon, aluminum, germanium, gallium and calcium.

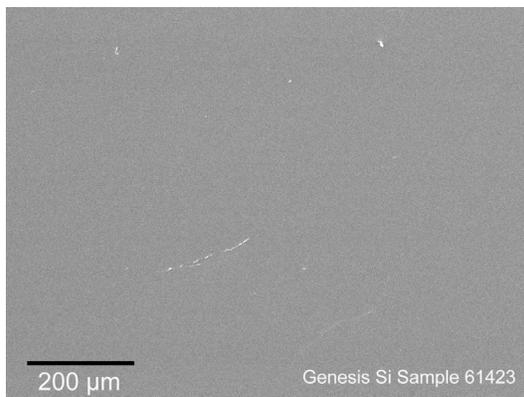


Figure 4. SEM image of nominally clean area of silicon sample 61423 for the small particle survey.

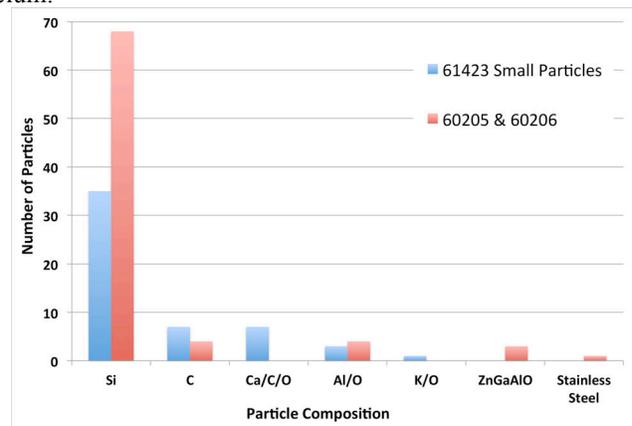


Figure 5. Composition of the 53 particles analyzed in the small particle survey of Genesis sample 61423 and the 80 particles on 60205 & 60206 compiled by Allton, et al. (2007) for comparison.