

CLEANING STUDY OF GENESIS SAMPLE 60487. K. R. Kuhlman¹, M. C. Rodriguez², C. P. Gonzalez², J. H. Allton², D. S. Burnett³; ¹Planetary Science Institute, 1700 East Fort Lowell, Suite 106, Tucson, AZ 85719; kim@psi.edu, ²NASA/Johnson Space Center, Houston, TX 77058; ³California Institute of Technology, Pasadena, CA.

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 were stopped 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. Particles of Genesis wafers, brine from the Utah Testing Range and an organic film have deleterious effects on many of the high-resolution instruments that have been developed to analyze the implanted solar wind.

We have conducted a correlative microscopic study of the efficacy of cleaning Genesis samples with megasonically activated ultrapure water [1] and UV/ozone cleaning [2]. Sample 60487, the study sample, is a piece of float-zone silicon from the B/C array approximately 4.995mm x 4.145 mm in size (Figure 1).

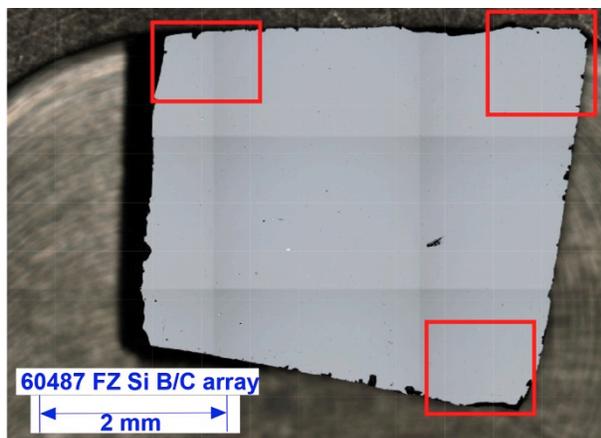


Figure 1. 5X Optical image of Genesis sample 60487 with regions scanned at higher resolution indicated. The region analyzed in this study is at Position 1, the upper left hand corner of the sample.

Initial UPW Cleaning. Sample 60487 was cleaned using the UPW method for 15 minutes at 40C and 3000RPM at NASA JSC. It was then divided into 3 regions for high resolution (50X) optical microscopic scanning (Figure 2). Each individual image is 221.6 μm across.

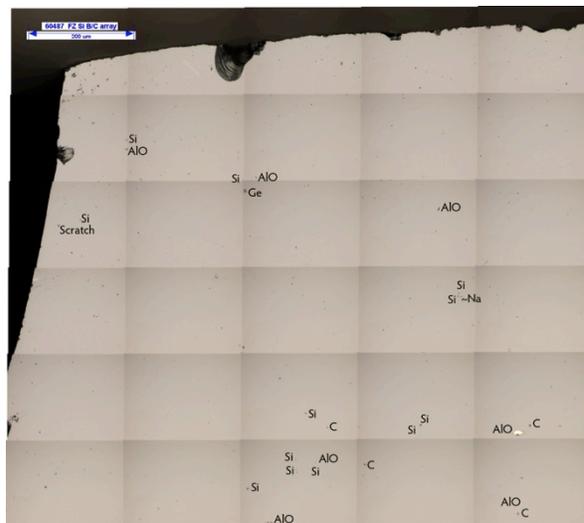


Figure 2. 50X Optical micrograph consisting of multiple high-resolution images of sample 60487. Particles that could be analyzed with low energy EDX are indicated.

Energy dispersive X-ray spectroscopy (EDX) study of remaining particles. The sample was then shipped to Dr. Kuhlman at the University of Wisconsin for scanning electron microscope (SEM) and energy dispersive X-ray (EDX) analysis using the SEM on a Zeiss CrossBeam microscope with an in-lens detector that has been shown to reveal much greater degrees of damage and particulate contamination on the Genesis samples than can be seen with a secondary ion detector. EDX analysis was performed with a 3 keV electron beam to minimize the electron interaction with the substrate material. By definition, this eliminated those elements with X-ray peaks above 3 keV. However, the 3 keV beam gives a qualitative analysis of the contaminants present. A list of the particles and their constituent elements is shown in Table 1.

One set of particles of interest are the white ovals seen in both the 5X and 50X optical images. Qualitative EDX indicated that these particles are composed of Al and O that appears to have wetted the sample upon deposition (Figure 3). The source of the Al and O is still unknown.

During imaging and analysis of the particles, the electron beam deposited a fair amount of hydrocarbons on the surface of the sample (Figure 4).

Table 1. Summary of qualitative EDX analysis of the particles remaining on Genesis sample 60487 after UPW cleaning.

| Particle Constituents | Number of Particles |
|-----------------------|---------------------|
| Si | 12 |
| AlO | 7 |
| C | 4 |
| Ge | 1 |

UV/Ozone cleaning. Sample 60487 was returned to NASA JSC for UV/O₃ cleaning [2] to check the efficacy of this technique to remove the hydrocarbons deposited by the SEM during imaging and analysis. It was exposed to UV/O₃ for 30 min.

SEM study of remaining residue. Sample 60487 was returned to the Univ. of Wisconsin for further SEM study. A series of SEM images was quickly taken to avoid adding more hydrocarbons to the sample. One of these images from the in-lens detector is shown in Figure 5. While subtle, the rectangles made during the first SEM analysis are still visible, indicating that the UV/O₃ cleaning was only partially effective.

References:

[1] Allton, J. H., et al. (2007) LPS XXXVIII, Abstract 2138. [2] Calaway, M. J., et al. (2007) LPS XXXVIII, Abstract 1627.

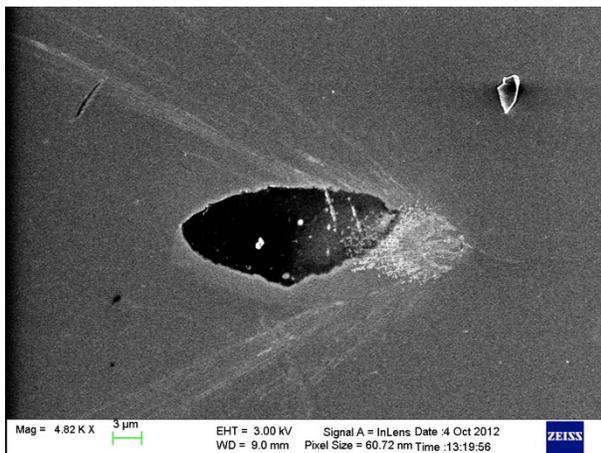


Figure 3. Al and O contamination of surface of 60487. The impact-like feature is unexplained as it does not occur with other particles of Al and O on this sample.

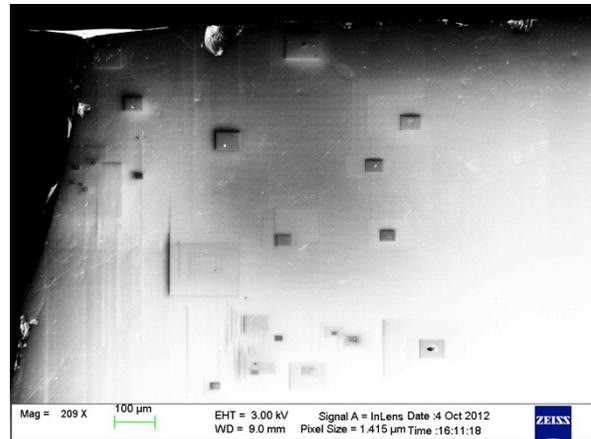


Figure 4. In-lens image of 60487 after SEM and EDX analysis. Note the distinct rectangles of hydrocarbons deposited by the electron beam during imaging and analysis.

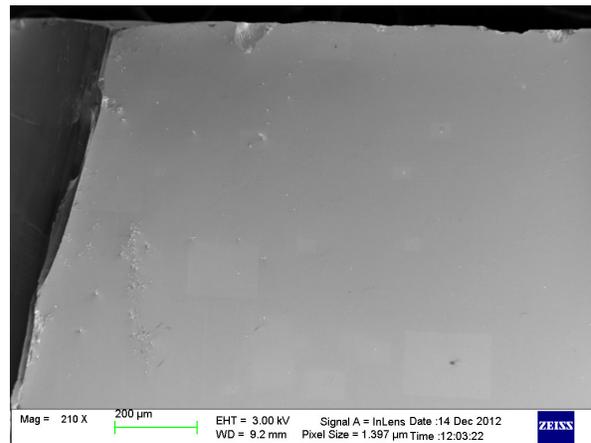


Figure 5. In-lens micrograph of 60487 after UV/O₃ cleaning. The rectangular boxes deposited by the electron beam during the first SEM analysis are more subtle, but can still be seen using the in-lens detector.