CATASTROPHIC IMPACT OF SILICON ON SILICON: UNRAVELING THE GENESIS IMPACT USING SAMPLE 61881. K. R. Kuhlman[1], H. Kim[2], A. J. G. Jurewicz[3], C. P. Gonzalez[4], K. K. Allums[5]; [1]Planetary Science Institute, 1700 East Fort Lowell, Suite 106, Tucson, AZ 85719; kim@psi.edu, [2]Microscopy and Imaging Center, Texas A&M University, 2257 TAMU, College Station, TX 77843-2257, [3]Arizona State University, Tempe, AZ, [4]Jacobs, NASA Johnson Space Center, Houston, TX 77058, USA, [5]HX5, LLC – Jacobs JETS Contract, NASA Johnson Space Center, Houston, TX 77058.

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.

The current work is motivated by the need to understand the interaction of the Genesis payload with contamination during the crash in the Utah desert. Silicon contamination has been found to be notoriously difficult to remove from silicon samples despite multiple cleanings with multiple techniques [1]. However, the question has been posed, "Does the silicon really need to be removed for large area analyses? [2]." If the recalcitrant silicon contamination is all pure silicon from fractured collectors, only a very tiny fraction of that bulk material will contain solar wind, which could skew the analyses. This could be complicated if the silicon trapped other materials and/or gases as it impacted the surface.

Sample selection and preparation for scanning transmission electron microscopy (STEM): Small samples of Genesis-flown CZ silicon that are not considered useful for any other purpose (< 2 mm) were requested from the Genesis curator. Three samples, 61881, 61883 and 61885 were cleaned at NASA JSC using isopropyl alcohol (IPA) sonicated for 5 min. and ultrapure water (UPW) sonicated for 5 min. according to previously published protocols [3, 4]. Sample 61881 was found to have several areas of silicon contamination in addition to some germanium contamination using energy dispersive X-ray spectroscopy (EDS) in a Tescan Lyra focused ion beam (FIB). A strip of electron beam assisted deposition (EBAD) platinum was deposited across the silicon mound shown in Figure 1 to protect the surface without introducing any mixing. A strip of ion beam assisted depositon (IBAD) platinum was then deposited on top of the EBAD layer to increase conductivity and further protect the surface. The FIB was then used to fabricate and lift out a section approximately 15 microns long and 10 microns deep. This section was then thinned to less than about 100 microns in the FIB. The sample was further thinned in a Fischione NanoMill using 900 eV argon at 5 degrees tilt for 10 min. on each side. The finished TEM sample is shown in Figure 2.

STEM and EDX Analyses: Sample characterization was performed on a TECNAI F20 Super Twin STEM equipped with a Schottky field emission gun, a high angle annular dark field (HAADF) detector, and an EDAX Instruments ultrathin window EDS detector. Convergent beam electron diffraction (CBED) patterns were collected with around a 1 nm electron probe and a 200 mm camera length after moving the probe to an area of interest and tilting the sample to the 001 zone axis. The electron beam energy was 200 keV.

Upon sectioning, it was evident that the silicon contamination was heavily damaged. However, initial TEM observations showed that the underlying CZ silicon is undamaged, except for two small conchoidal fractures (Figure 2). Further imaging clearly showed the solar wind implanted surface of the original Genesis collector, approximately 75 nm deep (Figure 3). We also observed a heavily shocked layer of silicon that is partially amorphous and partially polycrystalline with varying crystal orientations as shown by diffraction (Figure 3). A very distinct layer was seen at the interface between the original collector and the silicon contamination. This layer appears to vary gradually in thickness up to 10 nm, thicker than expected for the native SiO_2 on Genesis flight silicon [5]. In some places, this layer seems to disappear completely. We investigated the interface further using an EDS line scan beginning in an area of shocked polycrystalline silicon and ending at the edge of the solar wind implanted layer (Figure 3). The oxygen profile is shown as an overlay in Figure 3. This profile appears to be elevated in the interfacial layer as well as in the top 5 nm of the collector. Other than a small signal from the copper grid, no other elements were observed. It may be that the native SiO_2 layer on the collector is imaging as brightly as the silicon because it has been subject to solar wind irradiation damage.

Next Steps: The next step in our work is to fabricate samples of this type of contamination for atom probe tomography (APT). This technique will allow us to further investigate the composition and structure of the interfacial layer at the atomic scale to see if other elements are present at the interface, which could skew large area analyses. This layer could conceivably be "brown stain [6]," but no carbon was observed using EDS. If the interface is pure SiO_2, it is likely that the silicon contamination need not be removed.

References: [1] Kuhlman, K. R. (2016) LPSC XLVII, Abstract TBD. [2] Jurewicz, A.J.G., Personal Communication. [3] Allums, K. K. (2015) LPSC XLVI, Abstract #2014. [4] Calaway, M. J. (2009) LPSC XL Abstract #1183. [5] Calaway, M. J., E. K. Stansbery and L. P. Keller (2009) *Nuclear Instruments & Methods in Physics Research Section B*, **267**, 1101-1108. [6] Burnett, D. S. (2005) AGU Fall Meeting, Abstract #SH32A-01.

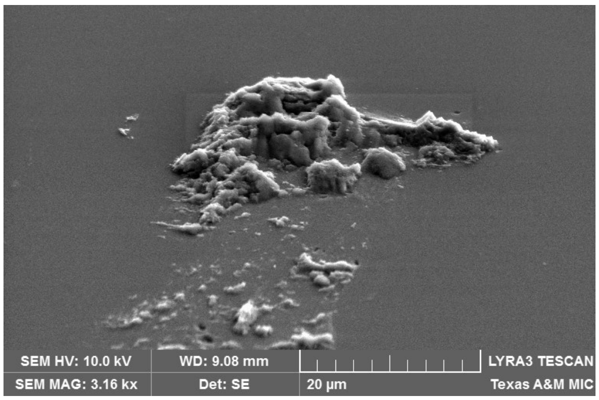

Figure 1. Silicon contamination remaining on Genesis CZ silicon sample 61881 after UPW and IPA cleaning. The sample has been tilted 55 degrees from normal in a Tescan Lyra FIB. A faint electron beam-deposited layer of Pt can be seen across the upper edge. This layer was then coated with a 2 micrometer-thick strip of ion beam-deposited Pt.

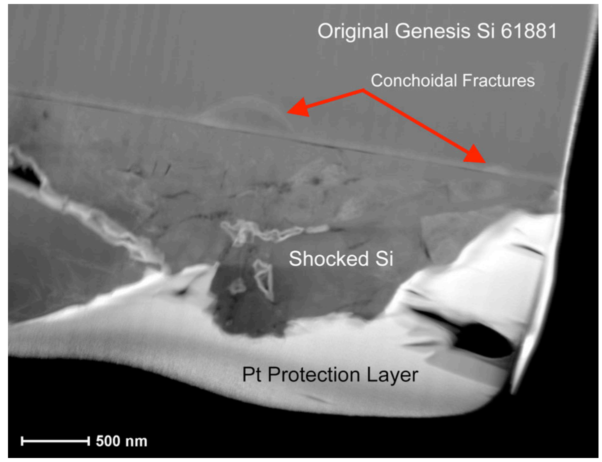

Figure 2. Dark field TEM image of finished silicon on silicon sample extracted from Genesis sample 61881 using a FIB. Note the conchoidal fractures at the surface of the silicon collector material.

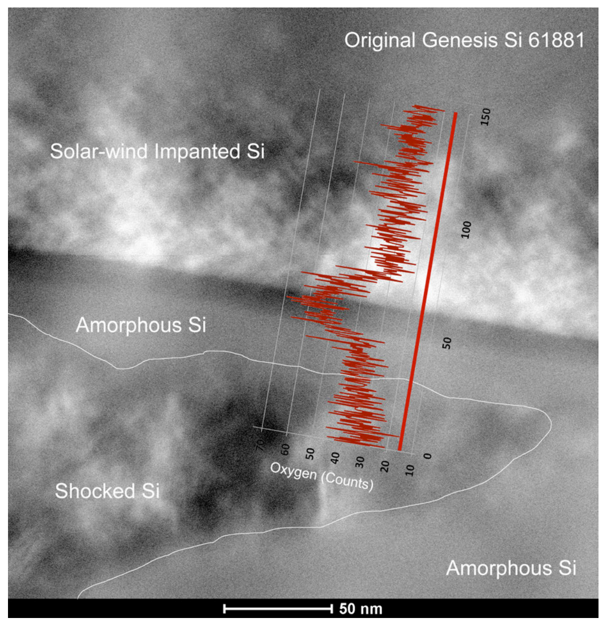

Figure 3. HAADF STEM image of the interface between the silicon contamination in Figure 1 and the Genesis silicon collector material. The line profile is the EDS measurement of oxygen (counts). Note the elevated oxygen in what appears to be the collector silicon.