Genesis: Removing Contamination from Sample Collectors. H.V. Lauer[1], K.M. McNamara[2], Andrew Westphal[3], A. L. Butterworth[3], D.S. Burnett[4], A. Jurewicz[4], D. Woolum[4]; J.H. Allton[1]; [1]Lockheed Martin c/o NASA/Johnson Space Center, Houston, TX 77058NASA, [2]NASA - Johnson Space Center, Houston, TX 77058; karen.m.mcnamara@nasa.gov;[1] ; [3]University of California at Berkeley, Berkeley. CA 84720; [4]California Institute of Technology, Pasadena, CA 91125;

Introduction: The Genesis mission returned to Earth on September 8, 2004, experiencing a non-nominal reentry. The parachutes which were supposed to slow and stabilize the capsule throughout the return failed to deploy, causing the capsule to impact the desert floor at a speed of nearly 200 MPH

Both the science canister and the major components of the SRC were returned before nightfall on September 8 to the prestaged cleanroom at UTTR , avoiding prolonged exposure or pending weather changes which might further contaminate the samples. The majority of the contaminants introduced as a result of the anomalous landing were in the form of particulates, including UTTR dust and soil, carbon-carbon heat shield material, and shattered collector dust (primarily silicon and germanium).[1]

Condition of the ArrayCollectors: The array collectors suffered severely in the impact. More than 10,000 individual fragments were retrieved from the science canister.[2] Additional fragments were collected on the ground in the field during recovery and embedded in the spacecraft hardware. Contamination issues range from light dust to heavily cemented UTTR soil. Fortunately, the latter is extremely uncommon.

Sample Analogs: Since the Genesis solar wind collector material is too valuable to consume in testing, cleaning techniques are developed using contaminated sample analogs Actual soil obtained from the landing site at UTTR was combined with dust generated by cleaving silicon wafers. The material was applied to surfaces of collector analogs in one of three ways: as a light coating of dry dust, resembling many of the collectors retrieved from the arrays; as a heavier coating of dry material including silicon, resembling material closer to the impact side of the canister; and as reconstituted UTTR soil slurry, resembling materials found outside of the canister and spacecraft. The soil slurry was produced by freeze-drying, grinding and reconstituting UTTR soil with water to resemble the consistency at the landing site. Because UTTR had experienced substantial rains the week before reentry, the ground at the recovery site was wet.

Physical Removal of Loose Particulates: Flowing Inert Gas: The primary contamination concern for the majority of the recovered collectors is that of loose particulates as described above. "Loose" refers to the fact that the particles are held in place by forces such as Van der Waals and electrostatic attraction rather than by chemical bond. The term, however, is misleading in that these forces can be quite difficult to overcome, particularly for particles <1 μm in size. This is confirmed by the observation that flowing inert gas such as nitrogen or argon, fails to remove substantial material (Figure 1). This was observed using both nitrogen and argon gas stream at UTTR and at JSC. Even the addition of surface vibration and ionization did not significantly improve the efficiency of particle removal.

Mechanical Removal: In addition to physical removal with flowing gas, mechanical removal using sable brushes was also investigated. The results for UTTR soil on silicon are shown in Figure 1C. These results are encouraging, however, additional evaluation is required since the actual collectors are contaminated not only with UTTR soil, but also carbon-carbon fibers and collector fragments. For many of the coated collectors (ie. AuOS), these materials will be substantiall more abrasive than on silicon and may cause scratching and removal of the solar wind implant. The technique may, however, have merit for hard solid collectors like sapphire and silicon.

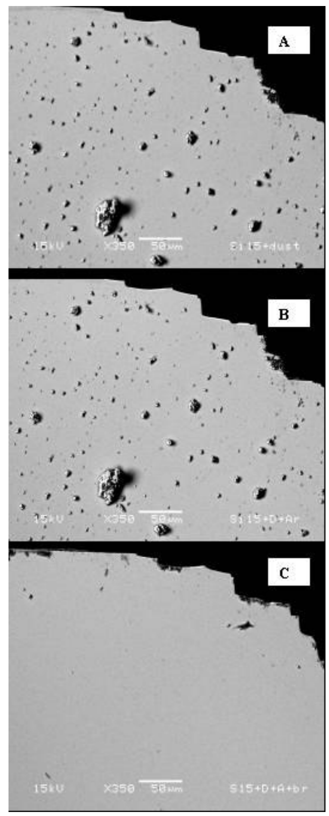

Figure 1: 350X backscattered electron images showing the results of successive cleanings, (**A**) dusty starting wafer, (**B**) blown on with argon gas stream. and (**C**) brushed with a fine soft hair brush.

Removal using Cryogenic "Snow" Techniques: Both Ar/N_2 and CO_2 cryogenic cleaning have been used to remove particulates from surfaces in the semiconductor industry. CO_2 snow tests were performed at Berkeley without success. Ar/N2 cleaning is currently being explored at FSI International, Inc. in Minnesota.

Solvent Cleaning: Wet chemical methods for removing particulates and encrusted soil were investigated as well.

Such methods are often used in the semiconductor industry for particulate removal.[3] The primary concern with wet chemical methods is the potential for reaction with the collector material, either in solution, at surfaces or at edges and defects. Many of the solvents used in semiconductor cleaning achieve high cleaning efficiencies by etching and removing several surface layers. Because the solar wind implant is shallow, these techniques may not be optimal for Genesis collectors.

KOH is often used in the semiconductor industry to remove contamination.[3] Figure 2 shows a comparison of the cleaning effectiveness of room temperature water and 30% KOH with sonication. The upper photo is a 1000X back scattered image of a dusty wafer that was ultrasonically cleaned at room temperature in distilled water for three minutes and then rinsed in three separate water baths. The dark grains in the photo are dust particles that were not removed in the water cleaning process. The stripe in the middle of the photo is a laser scribe location mark. The bottom photo is the image after similar cleaning with 30% KOH. Note that the particles were removed. Careful comparison of the scribe mark and very fine crack next to the scribe indicates that the KOH solution did not noticeably etch the wafer surface.

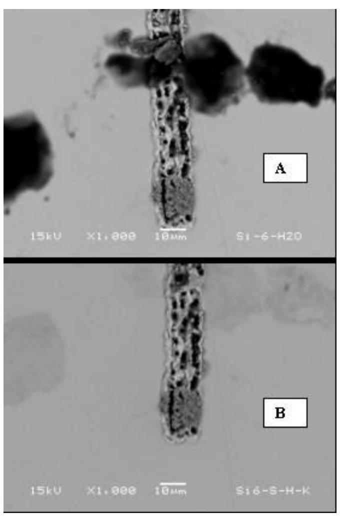

Figure 2: 1000X backscattered electron images showing the results of successive wet cleanings:(**A**) ultrasonic cleaning in distilled water for 3 minutes plus 3 successive rinses in fresh distilled water and (**B**) 1 minute additional ultrasonic cleaning in 30% KOH solution plus 3 successive rinses in distilled water.

Replicate Cleanning Process: Replicate cleaning is a process sometimes used in art restoration to remove contaminant films from objects and paintings. A polymer material suspended in a solvent is applied to the surface much like paint. As the solvent evaporates, the polymer forms a continuous film. The material is selected such that its adhesion to the surface is minimal. Patriculates on the surface become suspended in the liquid polymer which entrapps them upon drying. The particles are peeled away along with the film. The primary drawback of this technique is the use of organic solvents and the likely potential to leave behind molecular contamination.

The material tested here is known as Formvar and has very low adhesion to polished silicon. The cleaning process was as follows:

Formavar replica peel (repeated twice)
Toluene-acetone-methanol rinse (air dry)
Toluene-acetone-methanol rinse with sonication (air dry)
Methanol sonication (air dry)
Methanol sonication (blow dry)
Water + micro-90 rinse

Surface particle densities were measured using optical microscopy after each step. The results for each step are shown in Figure 3, where we plot integral particle density as a function of cutoff size for particle detection. Interestingly, there are cases where subsequent steps increase particle density indicating one of the difficulties of wet chemical processes – obtaining "clean" solvent materials.

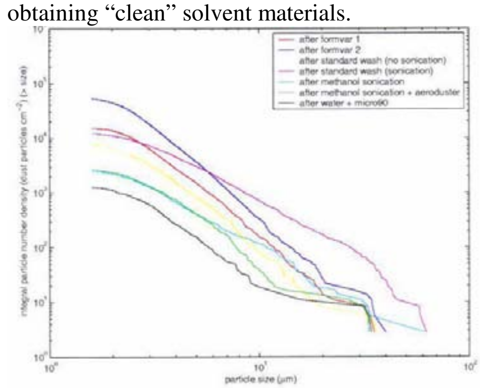

Figure 3. Integral Particle Density as a function of particle cutoff size for Replicate process cleaning steps.

Defocused Laser Ablation: While loose particulates are the primary contamination concern for the Genesis collector fragments, there are samples which encountered wet soil which has subsequently cemented to the surface. These materials may require more aggressive removal techniques such as defocused laser ablation which is being investigated at JSC and is also used in art restoration to remove surface contaminants.

Hydrogen Plasma Cleaning: Likewise, though molecular contamination has not been observed on collector surfaces to date, there may be some instances where removal becomes necessary. By far the most widely used technique for the removal of this type of molecular contamination from Genesis-like semiconductor surfaces is exposure to an oxygen plasma. However, there are several issues with the exposure of Genesis materials oxygen plasmas. Hydrogen plasma cleaning is being investigated as an alternative.

References: [1] K.M. McNamara, LPSCXXXVI, this volume, [2]J. H. Allton, LPSCXXXVI, this volume; [3] *Properties of Elemental and Compound Semiconductors* Edited by Harry C. Gatos **5** (1959) Interscience Publishers, Boston Ma.