Plan for Subdividing Genesis Mission Diamond-on-Silicon 60000 Solar Wind Collector

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Background: NASA's Genesis solar wind sample return mission experienced an off nominal landing resulting in broken, albeit useful collectors [1]. Sample 60000 from the collector (Figure 1) is comprised of diamond-like-carbon film on a float zone (FZ) silicon wafer substrate (DOS), and is highly prized for its higher concentration of solar wind (SW) atoms. A team of scientist at the Johnson Space Center was charged with determining the best, nondestructive and noncontaminating method to subdivide the specimen that would result in a 1 cm^2 subsample for allocation and analysis [2]. Previous work included imaging of the SW side of 60000, identifying the crystallographic orientation of adjacent fragments, and devising an initial cutting plan.

Approach: 1) Determine the analog wafer material that most closely approximates sample 60000. **2)** Verify crystallographic orientation of the sample. **3)** Scribe the backside to a depth that would assure cleaving along the desired path. **4)** Devise techniques for cleaving along scribe lines. **5)** Recommend mounting and handing techniques. **6)** Recommend appropriate materials contacting the SW surface. **7)** Consider methods of minimizing further contamination. **8)** Research particle removal and/or sample cleaning techniques. **9)** Detail the logistics and procedures of cutting two lines to produce three subsamples.

Results: 1) Although the production of 60000 was unique, we used similar non-flight FZ-Si wafers as our analog material to develop the optimal strategy for subdividing the flight sample. **2)** Previous investigations primarily used Czochralski (CZ) silicon wafers. As a consequence of the chemical and physical preparation of both the flight and analog FZ-Si wafers, the uncoated backside displayed a well defined euhedral crystals habit within octahedral crystallographic system (Figure 2).This enabled precise independant determination of crystallographic axis. **3)** We investigated the use of focused ion beam (FIB) milling a deep scribe line in the wafer material, but this proved impractical at centimeter scales The single-line multiple-pass (i.e., 100) standard approach of laser scribing resulted in a maximum cut depth of 110 µm; while the thickness of 60000 was 564 µm including ~1 µm diamond coating on the SW side. To increase penetration depth, after many experimental runs, a systematic approach was devised. This consisted of a) laser scribing 31 lines spaced 5 µm apart with 50 passes along crystallographic axis, b) cleaning out the ablated Si fluff generated by laser scribing and removed using an ultra-sonicated vibrating micro needle, c) repeating both steps, and finally d) laser scribing a single line at 100 passes down the center of the channel. We refer to this as the SCSCS (scribe-clean-scribe-clean-scribe) method which results in a channel depth ~35% of the sample thickness (Figure 3). **4)** We determined which side of sample to cleave following backside scribing. We recommended the first cleave of line #1 use the guillotine edged Cleavinator on SW side. The second cleave of line #2 use the sandwich cleave technique between glass which was facilitated by the small sample size. After consideration, we rejected the pressure point cleave common in industry as being too risky. **5)** Maintaining a consistent sample attitude during laser scribing of lines up to ~1 cm in length was challenging due to sample shape, uneven edges, polished edges, and crash artifacts such as chips. The standard sample holder supported the sample in suspension from well placed slotted holders. We considered that the sample would be handled and transported many times during the SCSCS process. The most stable and level configuration achievable was determined to be flat mounting the sample on a non-flight FZ-Si wafer as the substrate. **6)** Stabilizing the sample, against even subtle movement and not adding contaminants, such as tape adhesive, was achieved by bracing the sample with strips of Si wafers and taping them into position. A final bracing piece was taped straddling the sample, but free from the laser path (Figure 4). **7)** No contaminants contacted the SW surface. A 5 mm clean rubber tip and a polyester brush did contact the backside during the scribe channel clean out. All personnel wore nitrile gloves and clean room attire during sample processing and handling. **8)** We determined that vacuuming did remove particles, and was used only on the backside. Although the sample was fixed to a flat mount, Si particles produced during scribing, did adhere to the SW surface. Only clean stainless steel tweezers and containers were used for moving the sample, which was handled to avoid particle smearing or scratching. **9)** A detailed all-encompassing procedure including the cutting plan was written and practiced with end-to-end runs on analog samples until complete confidence ease of operation was achieved (Figure 5).

Conclusions: Experimentation and repeated practices [3] lead to the successful subdividing of the 60000 DOS target fragment as planned. However the actual space exposed sample responded a bit differently from what was witnessed during end-to-end runs or other practices on nonflight analog materials. Space exposure resulted in embrittlement Si wafer evident during clean out of particles following laser scribing, and during cleaving which required more pressure. The cleave of line #1 curved slightly toward one end of the scribe and possibly could be attributed to warping. Details of laser scribing process and instrument are explored in companion abstract.

References: [1] McNamara, K M et.al (2005) LPSC XXXVI Abstract #2403, [2] Rodriguez et.al (2009) LPSC XL Abstract #133, [3] See, T. H, et.al (2012) ESCG-3200-12-ARG-MEMO—0001 "Recommendations to the JSC Curator and Genesis CAPTEM Committee on Subdividing the Genesis DOS 60000 Concentrator Wafer.

Figure 1. Genesis solar wind concentrator target showing triangular-shaped fragment 60000 DOS.

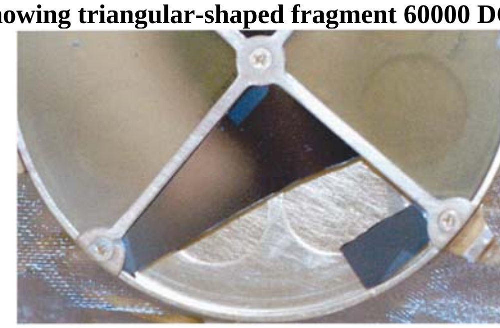

Figure 2. Crystallographic orientation of FZ-Si used for placement of scribe lines. Shown is a non-flight sample.

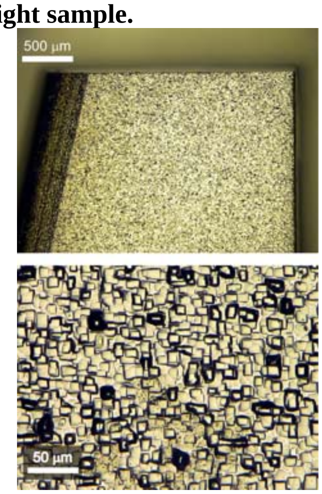

Figure 3 Channel created by SCSCS (left image-plan view) and (right image on-end view) before and after cleaning. Shown is a non-flight sample.

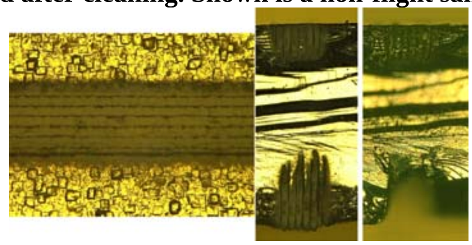

Figure 4 Schematic for mounting and stabilizing sample for laser scribing. Shown is a non-flight sample.

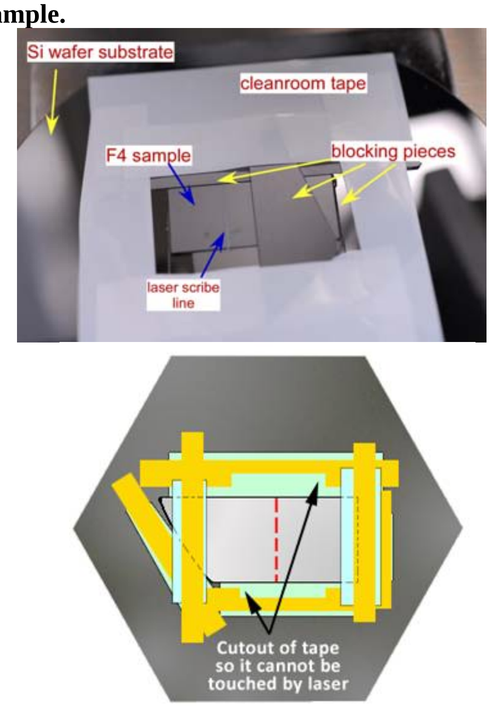

Figure 5 Cutting plan for 60000 DOS. The toe and heel clip marks (shown in white) were transposed from the SW side to verify location of scribe lines.