REMOVING CONTAMINATION FROM GENESIS SAPPHIRE COLLECTORS BY POLISHING. A. J. G. Jurewicz[1], K. Welten[2], R. Hervig[3], C. P. Gonzalez[4], A. J. Bixler[2], and J. Allton[5], [1]BCMS m/c 6004, ASU, Tempe AZ 85287 (Visiting Scientist Dartmouth College, Hanover NH). [2]SSL, UC Berkeley, Berkeley CA 94720. [3]SESE, ASU, Tempe AZ 85287 [4]Jacobs-JETS II, NASA JSC, Houston TX 77058 [5]NASA JSC, Mail Code XI2, Houston TX 77058

Introduction: The Array Collectors of the Genesis spacecraft collected bulk solar wind (SW), as well as three subsets of SW [1] (fast, slow, and coronal mass ejection) in a variety of materials [2]. In all cases the SW implant peaked within < 0.2 microns of the surface. So, when the sample return capsule crashed on landing, cleaning the surfaces for analysis became an extremely difficult, but imperative, task.

One collector material was "sapphire", commercial single crystal corundum. Only ~7.7% of the Genesis Array collectors were sapphire (SAP) but a much higher percentage survived the crash. In fact, surviving bulk SW sapphire collectors have an average area ~10x that of their silicon counterparts [3] since SAP is physically tough, and hard (MOHS 9, by definition) as well as is chemically inert. So, SAP is a good candidate for analytical techniques for measuring SW that require clean areas larger than a millimeter; e.g., synchrotron TXRF and INAA. It is also possible to measure SW in SAP using laser ablation and secondary ion mass spectrometry (SIMS) and, recently, more researchers are choosing to analyze sapphire. Thus, removing surface contamination from SAP could enable many research projects.

This report extends the task presented in Schmeling et al. ([4]) that used polishing compounds to remove contamination from Genesis sapphire. It focuses on how polishing with their two top candidates (colloidal silica and cerium oxide) affects the SW layer in SAP.

Experimental Procedures:

Pre-Characterization. Two small (<5mm long dimension) test samples were allocated for polishing: 61541 and 62161. These had been chosen in collaboration with Genesis curation for the wide array of crash-related smears and similar contaminants on the surface. They were too small to remove loose debris using JSC's megasonic cleaning process, so they were ultrasonically cleaned in ultra-high purity water at ASU. The "washed" samples were re-photographed and then sent to Berkeley for elemental mapping on a scanning electron microscope using the AZtec software [5].

Cleaning. 61541 was polished with cerium oxide (CeO_2) polish for ~3 minutes, and 62161 was polished with colloidal silica for ~8 minutes. Polishing was done mostly by rubbing with polish on gloved fingers as the small size made standard polishing techniques difficult. Polishing was done stepwise: photos were taken periodically throughout the procedure to document the progress. After rinsing, the polished samples were then returned to Berkeley for a second round of elemental mapping. TXRF was not used, due to small sample size.

61541 looked clean under a low power microscope after polishing and no subsequent procedures were used. Small silicon smears remained on the surface of 62161 after polishing. So, to remove silicon, another short, stepwise polish, was performed, followed by an HF-etch to remove any SiO_2, followed by a dilute ammonia soak to remove any underlying silicon. The sample was photographed to document each step.

Post-Characterization. SIMS measurements using the ASU Cameca IMS 6f were performed to determine if the SW H had been disturbed. To that end, analyses of SW H in the polished samples were compared with SW H from a previously-allocated test sample (50056-1) which had not been polished. Prior to SIMS analyses, the Cameca was baked-out, and samples were Au-coated for electrical conductivity. Throughout the analyses, an e-gun provided charge compensation for a ~2.2 nA Cs+ primary beam rastered over 125 microns. The analyzed area was 32 microns. Other instrumental parameters included a 40 eV energy window, a 400 micron contrast aperture, and 1 sec count times for H and Al.

Results: Both colloidal silica and CeO_2 cleaned the majority of the contamination from the crash. Silicon contaminants remaining on the sample polished with colloidal silica were removed by an HF etch followed by washing in dilute ammonia. The CeO_2 removed at least some (if not all) of the SW layer and, optically, pits seemed to be filled or polished out (Fig. 1). The colloidal silica + HF/Ammonia treatment left the sample clean and the SW layer untouched (Figs. 2, 3). 62161 (colloidal silica) and the control, 50056-1) both chipped/cracked during handling, probably along microfractures caused by the crash.

Discussion: The two polishes have MOHS hardness <7 so both were softer than undamaged SAP. But, both polishes are known to be reactive, which resulted in higher removal rates than harder, non-reactive polishes (e.g., [4]). Unfortunately, CeO_2 reacted with the SW layer of 61541 and should not be used for cleaning of Genesis samples. The colloidal silica showed good results, which were further improved when polishing was followed by etching of the recalcitrant contaminants.

Crack propagation during the handling of 62161 and 50056-1 may have been a positive result since contamination could escape cleaning within these cracks.

Summary and Conclusion: Surface contamination can be safely removed from Genesis sapphire collectors if the correct polish is used. This work has determined that colloidal silica polish does a good job of removing welded material from the collector surface without disturbing the SW layer. Any of the remaining contaminants are gently removed by a short HF etch followed by a dilute ammonia soak (which can be repeated, if needed). Some cracking along pre-existing microcrack may occur, but this likely exposes contamination not otherwise removable by either polishing or etching.

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References: [1] Barraclough et al. (2003) SSR **105:** 627–660; [2] Jurewicz et al. (2003) SSR **105:** 535–560; [3] JSC's Genesis catalog (12/2023); [4] Schmeling M. et al. (2021) 52nd LPSC, Abstract #1261; [5] Bixler et al. (2020) 51st LPSC, Abstract #2680.

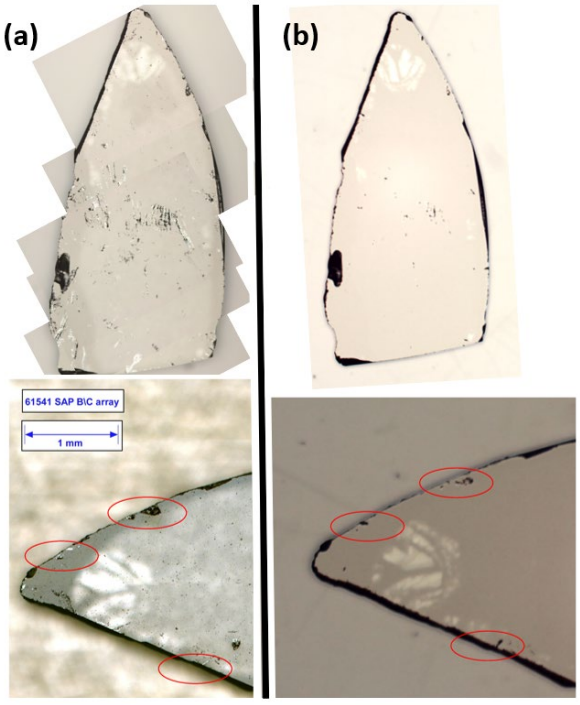

Fig 1. 61541 (a) , (b) are before, after ~ 3 min of polishing with CeO_2 slurry, respectively. Top: overview photos; bottom: closeup, with red circles showing significant changes – cracks/pits with contaminants removed, pits apparently reduced in size. *No SW H was recognized in 61541 after polishing*.

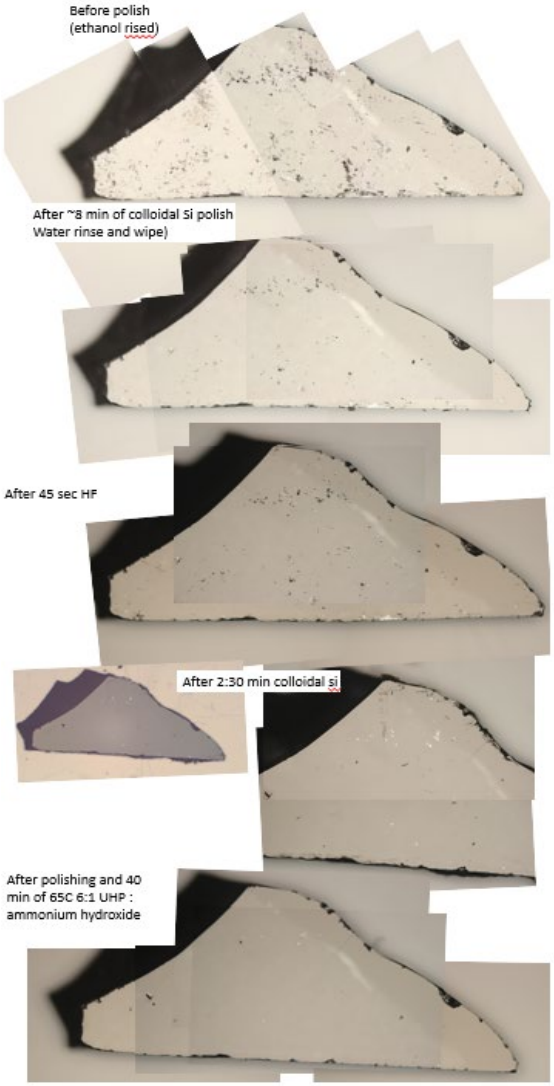

Fig 2. 62161 after each cleaning step (as marked).

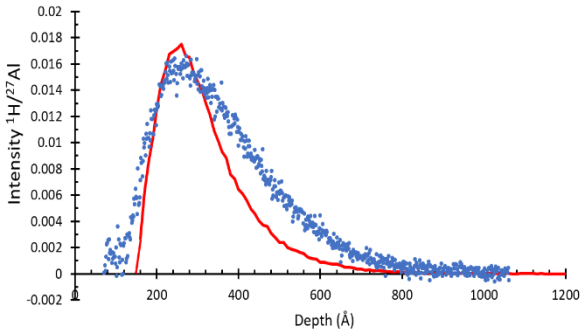

Fig 3. 62161 SW H depth profile (blue dots) after background H correction. SRIM calculation for SW H in perfect Al_2O_3 (red line) fits SW peak, assuming 140Å of Au-coating.