CLEANING OF GENESIS SAPPHIRE TARGET 61530 FOR SOLAR WIND CHLORINE, COBALT AND IRIDIUM MEASUREMENTS. K. C. Welten[1], A. J. Bixler[1], K. Nishiizumi[1], M. W. Caffee[2], A. J. G. Jurewicz[3], D.S. Woolum[4], and D. S. Burnett[5], [1]Space Sciences Laboratory, University of California, Berkeley, CA 94720, USA (kcwelten@ berkeley.edu), [2]PRIME Laboratory, Purdue University, West Lafayette, IN 47907, USA; [3]School of Earth & Space Exploration, Arizona State University, Tempe, AZ 85287, USA; [4]Dept. of Physics, California State University, Fullerton, CA 92834, USA; [5]Div. Geological & Planetary Sciences, California Institute of Technology, Pasadena, CA 91125, USA.

Introduction: Knowledge of the bulk composition of the Sun (comprising more than 99% of the total mass in the solar system) is an important goal for cosmochemistry and planetary science. Solar elemental abundances are derived from spectroscopic measurements of the solar photosphere. Except for the volatile elements, they show good agreement with elemental abundances in CI chondrites. The uncertainties in solar abundances are large, but can be improved by analysis of the solar wind (SW) composition from Genesis samples. The Genesis mission exposed various high-purity collector materials to the Sun for two years. These collector materials captured the solar wind and were returned to Earth for detailed elemental and isotopic analysis [1]. Upon return, the sample return capsule made an unexpected hard landing in the Utah desert, shattering many of the collector materials and contaminating them with Utah dirt and with pieces of spacecraft and collector materials.

Our main goal is to analyze solar wind Cl in a Genesis Si or sapphire target, using a new RNAA-AMS method, which is a combination of high-fluence neutron irradiation at the National Institute of Science and Technology (NIST), followed by radiochemical separation and analysis of ^{36}Cl by accelerator mass spectrometry (AMS). In the same neutron irradiation, other elements such as Co and Ir produce short-lived isotopes that can be determined by low-level gamma-ray counting. To measure these SW elements in the sample, we have to remove any contaminants on the surface of the Genesis target both before and after performing the neutron irradiations. Based on expected SW abundances, we have to reduce the contamination level to <0.02 pg of Cl, <0.04 pg of Co and <0.04 fg of Ir for ~1 cm^2 of target. These cleaning methods are not only necessary for the SW Cl, Co and Ir measurements, but will also be beneficial for other Genesis SW measurements, especially those that require the analysis of areas of >10 mm^2.

The major contaminants on Genesis collector samples are Utah dirt, accretionary Si, Ge, Al_2O_3, Au, Al, stainless steel (ss), white paint, and C. Our previous cleaning study of a Genesis flight Si target (61229) showed that the Si contamination is difficult to remove without using strong acid (HF-HNO_3) or ammonia, which would damage the Si target itself [4]. Although the Si contamination itself does not interfere with our SW Cl, Co and Ir analyses, it contains small (µm - sized) ss particles that appear to have co-deposited with the Si [4]. It also may shield Utah dirt contamination. Since then, most of our cleaning efforts have focused on a Genesis sapphire target, 61530 (Fig. 1).

Genesis sample: We examined a damaged piece of Genesis sapphire target 61530, which measures 6.8 mm x 3.6 mm (Fig. 1). This target was cleaned at the JSC Curatorial Facility using the megasonic UPW cleaner system [2, 3], which removed the majority of Utah salt and dirt components.

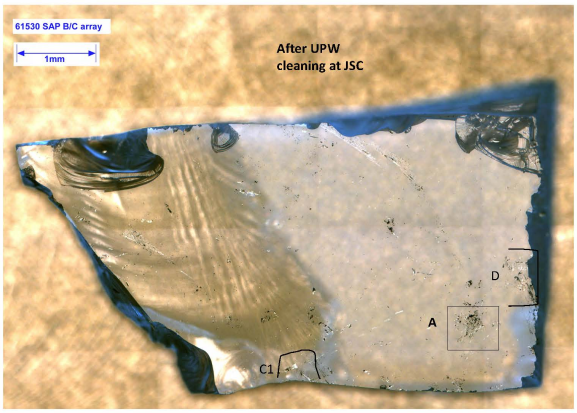

Fig. 1. Genesis sapphire sample 61530 after UPW cleaning at JSC. Most SEM studies focused on areas C1 and D.

Methods: Our cleaning strategy combines the following methods: (a) transmitted and reflected light optical microscopy, (b) SEM backscattered electron (BSE) imaging to identify contamination and energy dispersive X-ray spectroscopy (EDS) for chemical analysis of contamination, including our "low vacuum" mode (10 Pa N_2) for sapphire, (c) chemical etching with high-purity reagents, including HNO_3, NH_4OH and HF, using agitation in a megasonic tank (1 MHz, 1200 W) cleaner, and (d) ICP analyses of cleaning solutions, both optical emission spectroscopy (ICP-OES) and mass spectrometry (ICP-MS). A benefit of doing all these steps in the same lab is a very large gain in efficiency, and the elimination of possible contamination during transport between multiple labs. Two 2-day sessions (December 2017 and June 2018) at UC Berkeley resulted in significant progress. The main results of the first session

showed that megasonic cleaning in dilute HNO_3 removed a large portion of 1-10 μm particles (including Ge and ss), but was less effective in removing accretionary Si and many ss particles associated with accretionary Si [4].

C-deposition: A potential problem is the effect of C deposition during SEM analysis, possibly armoring contamination to subsequent etching. Except for areas pre-selected for analysis based on optical examination, the rest of the sample saw only low current electron beams when locating the preselected areas for detailed study. On many occasions, new areas were examined after a given etching step. Many areas studied in detail by SEM prior to etching showed significant contaminant removal. We thus conclude that our SEM examination does not hinder chemical removal of contaminants from Genesis flight samples by C-deposition.

Removal of accretionary Si: The first NH_4OH treatment of 61530 in a pyrex tube showed incomplete removal of Si. Since the Si blanks were too high, we could not quantify the Si removal using ICP. We thus performed the next megasonic treatments (using HF and NH_4OH, respectively) with the sample suspended by a Pt wire in a Pt crucible. Subsequent SEM imaging and ICP analyses showed that both leaching steps further reduced the accretionary Si contamination. ICP analysis of the HF etch solution shows that 1.9 μg of Si was removed, equivalent to an average Si thickness of ~30 nm over the entire target or (more likely) ~1 μm thickness across ~3% of the surface. SEM analyses after the final NH_4OH etch showed additional Si loss (Fig. 2), while ICP analysis indicated that the Si loss in this step was <0.1 μg, an upper limit mostly constrained by the Si blank in NH_4OH. The SEM images and the ICP results clearly give complementary information on the effectiveness of Si removal. The original Si deposits on 61530 were never thick, but thicknesses up to 2-3 μm have been measured. The remaining Si contamination is very thin (<1 μm), as shown by high power (1000x) reflected light observations.

High Z contaminants: The SEM is sensitive for recognizing small ss particles that are a threat to SW analysis. The remaining <1 μm thick Si deposits cannot hide 0.1-1 μm grains of ss. We systematically searched six locations on the post NH_4OH treated 61530 sample for bright areas in the BSE images. We found one 2 μm Ge grain, and three sub-μm GaZn-spinel (paint) grains (Fig. 2), but no ss. These small grains were easily recognized, so we are confident we have not missed any ss grains >0.1 μm in the areas searched, although many of these areas showed abundant ss particles before.

Conclusions: We obtained excellent removal of small particles from 61530 (sapphire), a sample that had already been UPW-cleaned at JSC, with megasonic agitation. With the use of HF and NH_4OH leaching steps, combined with megasonic agitation, we have rendered the accretionary Si harmless, by reducing it to sub-μm thickness where it can no longer hide high Z contaminants such as ss. More detailed chemical analysis of the entire target using SEM with Aztec particle analysis software or using TOF-SIMS is required to obtain a quantitative estimate of the remaining contamination.

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References: [1] Burnett D. S. (2013) *Meteoritics & Planet. Sci., 48,* 2351–2370. [2] Allton J. et al. (2007) *LPS XXXVIII,* Abstract #2138. [3] Calaway M. J. et al. (2009) *LPS XIV,* Abstract #1183. [4] Welten K. C. et al. (2018) *LPS XLIX,* Abstract #2660.

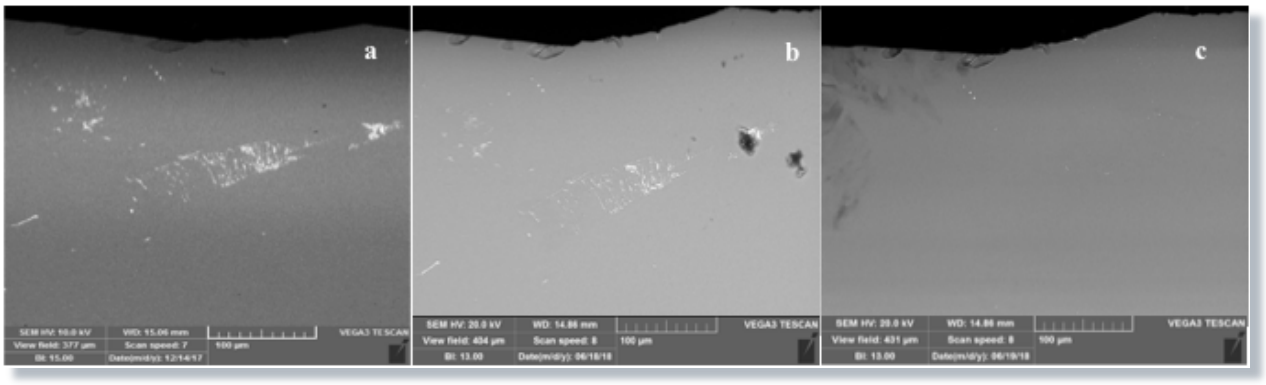

Fig. 2. SEM-BSE images of 61530 area C1 after HNO_3 and NH_4OH etch (a), after HF etch (b) and after final NH_4OH etch (c). Field of view of each image is ~400 μm. The white streaks representing accretionary Si are gradually removed going from panel (a) to (c). The two dark blurs in (b) are Teflon that was removed in the next step.