CLEANING STUDIES OF GENESIS SAPPHIRE TARGET 61530. K. C. Welten[1], A. J. Bixler[1], K. Nishiizumi[1], M. W. Caffee[2], A. J. G. Jurewicz[3], and D. S. Burnett[4], [1]Space Sciences Laboratory, University of California, Berkeley, CA 94720, USA (kcwelten@ssl.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]Div. Geological & Planetary Sciences, California Institute of Technology, Pasadena, CA 91125, USA.

Introduction: The Genesis mission exposed a variety of 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.

One of our goals 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 followed by radiochemical separation and analysis of ^{36}Cl by accelerator mass spectrometry (AMS). After Cl extraction, we will also measure other SW trace elements with high neutron capture cross sections - such as Co and Ir - in the same target, using low-level gamma-ray counting. To measure these SW elements in the sample, we have to thoroughly remove any contaminants (especially those containing Cl, Co or Ir) on the surface of the Genesis target before performing the neutron irradiations. This work describes preliminary cleaning studies of a Genesis flight sapphire target (61530) using a combination of megasonic cleaning, followed by ICP-MS analysis of the cleaning solution and examination of the target by Scanning Electron Microscopy (SEM), using the BSE mode for imaging and the X-ray mode for chemical analysis.

Genesis sample: We examined a piece of Genesis sapphire target 61530, which measures 6.8 mm x 3.6 mm (Figure 1). This target was cleaned at the JSC Curatorial Facility using the megasonic UPW cleaner system [2.3], which removes the majority of Utah salt and dirt components.

Methods: Before cleaning efforts at UC Berkeley, the target was examined by optical microscopy at 100-1,000x magnification to identify areas with high contamination levels and to estimate the approximate thickness of the contamination. The sample was then examined by SEM using the Tescan Vega3 instrument with a low-vacuum scanning ability and Oxford EDS for chemical identification of the particles. The low-vacuum mode (10Pa N_2 environment) allows imaging of the sapphire sample without the need for graphite coating, which would hinder subsequent cleaning of the target. The sample was visualized at low current to minimize carbon deposition, and current was increased only in select regions for subsequent elemental analysis of the contaminants by X-ray analysis. Since our initial concern was that C deposition during SEM imaging would armor contaminants with graphite coating, thus retarding their subsequent removal by etching reagents, we did not perform elemental mapping of the areas of interest.

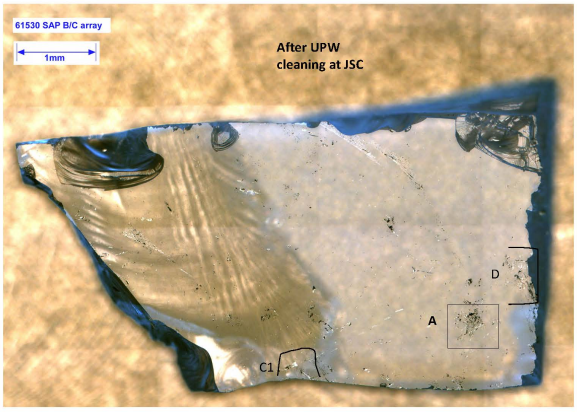

Fig. 1. Genesis sapphire sample 61530 after UPW cleaning at JSC. Most SEM studies focused on areas C1 (bottom) and D (right).

To remove expected contaminants like Si, Al, Ge, and ss, we performed two consecutive cleaning steps by placing the target in a pyrex tube with 3 ml of 1.5N HNO_3 (step-1) for 10 minutes at 38-45 ºC or in 3 ml of 3N NH_4OH (step-2) for 15 minutes at 43-57 ºC. The target was sonicated in a megasonic bath (1MHz, 1200 W) for the full duration of these tests, and subsequently rinsed with UPW (18.2 MΩ). The cleaning solution was subsequently measured by ICP-OES (Thermo iCAP6300 duo) and/or ICP-MS (Agilent 8800) to quantify any dissolved contaminants. The target was analyzed by SEM after each cleaning step. The SEM analyses mainly focused on two 500 µm x 500 µm areas (labeled C1 and D in Figure 1). The contamination in area D is dominated by small fragments (mostly 1-10 µm) of Ge and Si collector material. Area C1 is dominated by 100-200 µm long streaks of relatively porous splattering Si metal with a few bright specks, which are either identified as FeNiCr stainless steel (ss), or Ge. Although no Co was detected by SEM, it is likely that the ss contains up to 100 ppm of Co, so any

ss has to be removed. The X-ray spectra of most of these Si rich areas also show a small Mg peak of unkown origin. The Mg contamination is not from the Si metal itself or unlikely to be from 6061 Al. Although the Mg could be related to Utah soil not removed by UPW cleaning, it is not associated with Na, Ca and Fe, which are generally also present in the soil. While the O/Al ratio of the X-ray spectra of pure sapphire is ~0.8, the sapphire that is covered with a 1-10 µm layer of Si metal yields significantly lower O/Al ratios (as low as 0.5) due to preferential absorption of O X-rays in Si. No pure Al X-ray spectra were found, suggesting that no large (few um) pieces of Al metal were present.

Nitric acid leach. Optical examination of 61530 after nitric acid leaching step showed a relatively clean sample. SEM examination showed that some ss and Ge particles were removed, while a few small sapphire grains were redeposited elsewhere (Fig. 2). A significant fraction of the Ge particles is removed or dissolved, as is concluded from the SEM images after leaching and from the ICP–MS analysis, which shows 100 ng of Ge in the solution. This amount is about 2 orders of magnitude higher than the Ge contamination of ~1 ng found on SoS wafer fragment 50030 [4], confirming previous observations that contamination varies strongly from one target to the next [4]. Although it is likely that some of the ss particles were removed by the nitric acid leach, our blank levels of Fe, Ni and Cr were too high to detect significant ss components in the nitric acid solution. The amount of Co in the nitric acid leach solution was <10 pg. Finally, the SEM X-ray spectra show that the nitric acid leach removes the majority of the Mg contamination.

Ammonia leach. The ammonia leach was performed to attack metallic Si contamination. Optical and SEM examination of 61530 after the leach shows that none of the Si metal streaks were completely removed, but that some Si areas were partly dissolved or removed. Although ammonia does not dissolve ss or Ge, some tiny grains that were associated with the Si "host" were removed by the ammonia leach. About 5 ng of Ge is detected by ICP-MS analysis of the solution. The amount of dissolved Si could not be quantified by ICP, as our NH_4OH blank was too high (~15 µg Si).

Conclusions. The sapphire target shows three major types of contamination, including Si metal, Ge metal and ss, plus some minor contaminants including Mg of unknown source. The Ge and Mg contamination is effectively removed by nitric acid leach, while some of the Si and Si-associated ss is removed by the ammonia leach. In addition, the megasonic agitation seems to help removing micron-sized grains. Future works involves longer leaching steps and more concentrated solutions of nitric and ammonia, as well as other reagents. In addition, physical cleaning methods, such as CO_2 snow will be tested.

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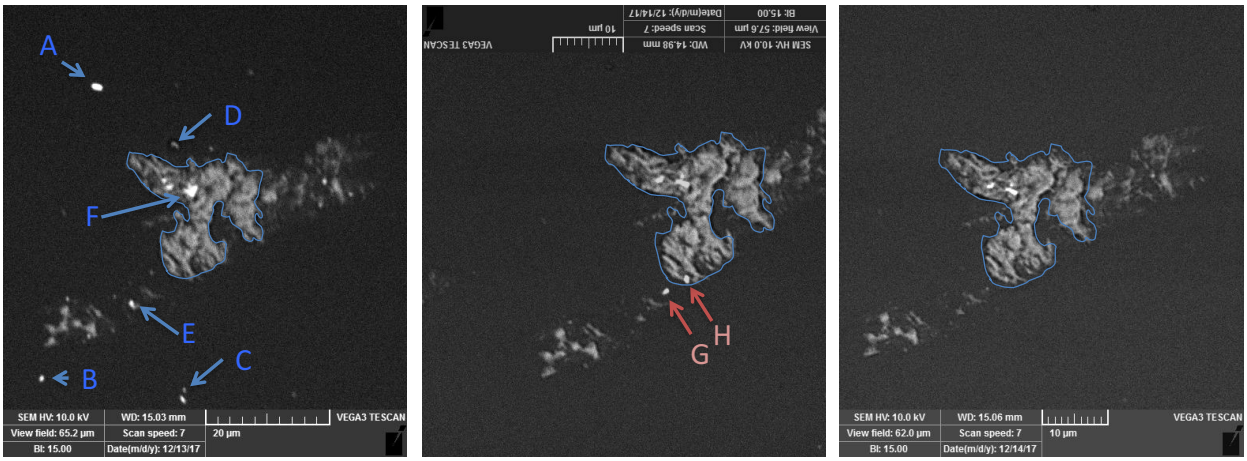

Fig. 2. BSE image of area C1e of 61530, before (left), after HNO_3 (middle) and NH_4OH leach (right). FOV in each image is 65 µm. Particles A-E were removed during HNO_3 leach, while particle F was reduced in size, and G and H were redeposited. An outline of the main Si contaminant is shown before cleaning and copied to each image