A FIRST LOOK AT OXYGEN IN A GENESIS CONCENTRATOR SAMPLE. K.D. McKeegan[1], G. Jarzebinski[1], A.P. Kallio[1], P.H. Mao[1], C.D. Coath[2], T. Kunihiro[3], R. Wiens[4], J. Allton[5], M. Callaway[5], M. Rodriguez[5], and D.S. Burnett[6] [1] Dept. of Earth & Space Sciences, UCLA, Los Angeles, CA. 90095-1567 USA, [2]Dept. of Earth Sciences, Univ. of Bristol, Bristol, BS8 1RJ, UK, [3]SEI, Okayama Univ., Misasa, Tottori 682-0193 Japan, [4]Los Alamos National Laboratory, Los Alamos, NM 87545 USA, [5]Johnson Space Center, Houston, TX 77058 USA, [6]Div. Geol. & Planetary Sci., Caltech, Pasadena, CA 91125, USA.

Introduction: An accurate and precise determination of the oxygen isotopic composition of the Sun is the highest priority scientific goal of the Genesis Mission [1] as such data would provide a baseline from which one could interpret the oxygen isotopic anomalies found at all spatial scales in inner solar system materials [2]. MegaSIMS [3], a hybrid secondary ion and accelerator mass spectrometer (SIMS/AMS), was designed to measure the solar oxygen and nitrogen isotopic compositions from the Solar Wind (SW) captured by an electrostatic concentrator into SiC target wafers. We are pleased to report that three years after the 'hard-landing' of the sample return capsule, we have made the first oxygen isotopic measurement on the concentrator SiC sample #60001.

Analytical challenges: The primary difficulty in measuring the SW oxygen is essentially the challenge of analyzing a trace element in the face of potentially overwhelming contamination from terrestrial oxygen. The Genesis SiC concentrator samples comprise a total area of 7 cm^2 (for each of two wafers) and the expected mean implantation depth of the SW oxygen is only ~100 nm with a total expected fluence of 3×10^{14} atoms cm^{-2}. The types of terrestrial oxygen that we must contend with are surface particulates, uniformly adsorbed compounds (eg., water), and instrumental background (sources of oxygen not originating from the sample). Surface particulates can be avoided by inspection with a reflected light microscope, and, if necessary, they can be detected with the micron-resolution ion-imaging capability of MegaSIMS [3]. For the past two years, we have concentrated on developing methods to remove adsorbed sample surface contamination while minimizing sample consumption and overall instrumental background. Figure 1 summarizes the improvements we have made in reducing surface and instrumental backgrounds.

Surface contamination: The surface of a clean SiC wafer typically acquires roughly a monolayer of contaminant oxygen, which is significantly more than the SW oxygen we expect to find in the concentrator samples. Therefore it is important to quantitatively remove this material without mixing it into the SW layer implanted shallowly beneath the surface. Additionally, the time between surface cleaning and analysis must be minimized to prevent significant re-absorption of surficial oxygen thus requiring cleaning *in-situ* under ultra-high vacuum conditions. Because of sputter knock-on effects, the inherent depth resolution of SIMS is proportional to the impact energy per unit mass of the analyzing beam on the sample, so for surface cleaning, it is advantageous to minimize the impact energy of the primary ion beam.

Removing 20 nm of the sample surface with Cs^+ at an impact energy of 5 keV (as opposed to 20 keV for analysis) reduces the surface signal by two orders of magnitude and makes the surface contamination negligible with respect to the implant signal by ~30 nm depth into the sample. It also pre-cesiates the sample surface, which reduces transient effects during analysis. With a lower impact energy, and the correspondingly shorter mixing length, we would not have to remove as much material, but the practical considerations of cleaning time and a quick return to analytical conditions limits us to 5 keV impact energy.

Instrumental background: The instrumental background consists of at least two components that contribute to a nearly constant background on the time scale of a measurement. One component is due to oxygen on the secondary ion extraction plate that contaminates the sample via secondary beam sputtering. We clean the extraction plate by sputtering oxygen-free SiC with 80--100 nA of Cs^+ at 20 keV impact for several hours prior to analyses. The efficacy of the cleaning can be monitored by the intensity of ^{16}O in the secondary beam. At pressures below 5×10^{-11} Torr, buildup of oxygen contamination on the extraction plate is not a problem during or between analyses, but the contamination does reestablish itself overnight, making the high-current cleaning procedure a daily task.

The other component of instrumental background is believed to be water migrating from the sample chamber walls to either the sample or the extraction plate, though this explanation is not entirely satisfactory. We have brought this component under control by replacing the pumps on the sample chamber and through frequent baking (at 125 ºC). A stock Cameca IMS-6f has a 280 l/s (N_2) turbo pump in series with a Ti-sublimation pump on the sample chamber and can achieve a base pressure of ~2×10^{-10} Torr. We replaced these pumps with a cryopump (660 l/s N_2, 2200 l/s

H_2O), which reduced the base pressure in the sample chamber to ~1×10^{-11} Torr.

Genesis Concentrator sample #60001: In early December, we were allocated the Concentrator SiC sample #60001 and authorized to analyze 3 mm^2 of the intact sample. The sample was transported from JSC to JPL for ultrasonic cleaning with xylene. Visually, the cleaning step removed much of the particulate surface contamination that resulted from the crash. The sample was then mounted in a custom MegaSIMS quadrant sample holder, transported to UCLA, and installed in the sample chamber. Optical inspection of the sample confirmed that some areas are damaged and/or contaminated but that relatively large areas are free of particles larger than 1µm. Sample #60001 was left in the sample chamber for 5 days during which time the chamber was baked at 125 ºC for 46 hours. Prior to analysis, the extraction plate cleaning described above was run for 4 hours. For this initial measurement, we picked a location close to a defect in the SiC, so as not to consume area that would be valuable for other analyses (r = 20mm, θ = 45º). Final primary beam tune-up and secondary beam alignment was performed on the defect, and the analysis was carried out ~250 µm from the defect. The 5 keV impact cleaning beam was run for 5 minutes at 20 nA to remove ~20 nm of the surface. The analysis was carried out with a 30 nA Cs^+ beam rastered by 130 µm. The field aperture gated the analytical area to 100 µm diameter. A second analysis was carried out in the same area, adjacent to the SiC defect, without low energy Cs^+ cleaning, in order to establish an absolute depth scale.

The $^{16}O^{++}$ signal from these first analyses are shown in Figure 2. Terrestrial background from surface contamination and the instrument were as good as hoped for; the signal from the SW implanted oxygen exceeded expectations. The mean depth of the implanted oxygen appears to be around 80 nm where the $^{16}O^{++}$ signal peaked at ~75,000 cps, and the signal disappeared into instrumental background at around 250 nm.

Conclusions. Our first look at the SW oxygen signal from Genesis Concentrator SiC sample #60001 is very encouraging. The sample is mostly free of particulate contamination, the dreaded and ill-named "brown stain" is effectively removed by our cleaning procedures, and, most importantly, the concentration of oxygen in the sample exceeds pre-flight estimates by up to 50%. We expect to be able to report preliminary oxygen isotope data at the meeting.

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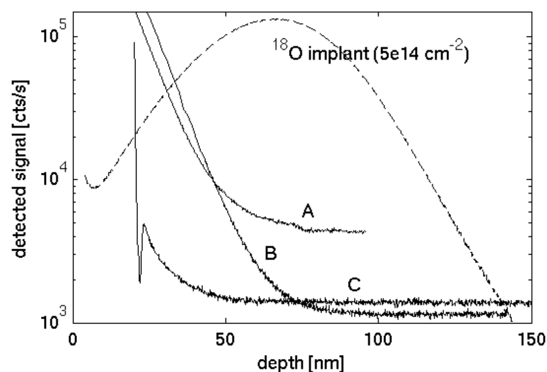

Fig. 1. Oxygen background from SiC flight-spare blanks. The dashed line shows the $^{18}O^{++}$ signal from an implant with fluence near the expected value for SW ^{16}O. (A) Background from SiC blank with original instrument setup. (B) Background with cryo pump, 2 days after baking and after extraction lens cleaning. (C) Similar to B, 5 days after baking, removed 20 nm of surface via low energy Cs^+ sputtering.

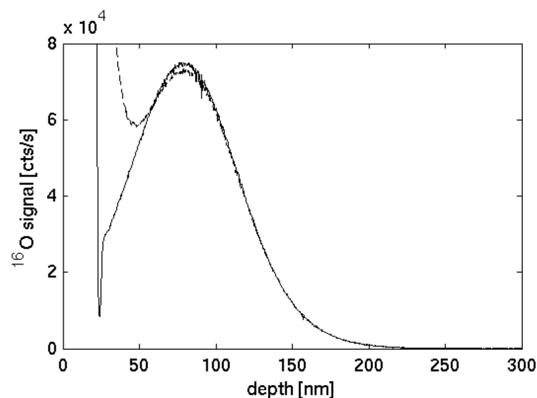

Fig. 2. First two depth analyses of Genesis Concentrator Sample 60001(SiC). Instrumental background subtracted $^{16}O^{++}$ signal with (solid line) and without (dashed line) 5 keV impact Cs^+ precleaning. The precleaning removed 21 nm from the sample surface.

References:[1] D.S. Burnett et al. (2003) Space Sci. Rev. 105: 509-534. [2] K.D. McKeegan and L.A. Leshin (2001) Rev. in Mineralogy & Geochemistry 43: 279-318. [3] P.H. Mao et al. (2006) 37th Lunar and Planetary Science Conference, Abstract #2153.