DETERMINATION OF Fe AND Mg FLUENCES IN GENESIS SoS WAFER FRAGMENTS. S. Huang[1], M. Humayun[1], D. Burnett[2], and A. J. G. Jurewicz[3], [1]National High Magnetic Field Laboratory & Dept. of Geological Sciences, Florida State University, 1800 E. Paul Dirac Dr., Tallahassee, FL 32310, huang@magnet.fsu.edu, [2]Division of Geological and Planetary Sciences, California Institute of Technology, Pasadena, CA 91125, [3]Center for Meteorite Studies, Arizona State University, Tempe, Arizona, AZ 85287.

Introduction: During the NASA GENESIS mission, solar wind samples were collected by exposing ultraclean wafers to the solar wind outside the Earth's magnetosphere for 884 days. However, a design error caused the sample canister to crashed-land into the Utah desert on return to Earth. This accident violated the ultraclean environment of the sample canister. The wafers were broken into small fragments and were seriously contaminated by Utah desert dust/mud, debris from the sample canister, and powdered wafers. Since the solar wind was implanted into the wafers ~100 nm below the surface, careful cleaning of the wafer surface to remove the surficial contamination will make it possible to recover the chemical abundances of the implanted solar wind tracers. Following Huang et al. [1], we applied step-cleaning procedures on two flight Silicon-on-Sapphire (SoS) wafer fragments, 60297 and 60298, and determined the Mg and Fe fluences by Laser Ablation Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS).

Experiments: LA-ICP-MS analyses were performed on a Thermo Finnigan Element XR™ ICP-MS with a New Wave™ UP 213 Nd-YAG (213 nm) laser system at the Plasma Analytical Facility at National High Magnetic Field Lab, Florida State University. The laser was used in line scan mode: 250 μm spot size, scan rate of 50 μm/s, 1-2 mm length tracks, laser repetition rate 10Hz, 40% output. The energy output of laser was 0.15-0.20 mJ. Elemental analyses were conducted at medium resolution (M/ΔM=4400) using ^{24}Mg, ^{25}Mg, ^{27}Al, ^{29}Si, ^{52}Cr and ^{56}Fe. Glass standard NIST SRM 612 and a piece of ^{25}Mg SoS implant were used to calibrate the Fe and Mg fluences in the wafer fragments. It is important to mention that our recently installed Element XR™ ICP-MS has significantly higher sensitivity than the instrument used in our previous experiments [1]. For example, we obtained a signal/noise ratio of 300-400 on the ^{25}Mg peak at medium resolution using the ^{25}Mg SoS implant which has a nominal ^{25}Mg fluence of 1.1E13 atoms/cm^2.

The flight SoS wafer fragments provided were for testing cleaning procedures. Each fragment was 2-3 mm in size.

To remove surficial contamination, we applied step-cleaning procedures [1] on flight wafer fragments 60297 and 60298, which had been pre-cleaned using the ultra-pure water cleaning procedure at NASA JSC Curatorial Facility. Prior to cleaning, the wafer surfaces were analyzed by laser ablation ICP-MS, and found to have Fe-Cr particulates which were not found previously on Si wafers [1]. The wafers were then cleaned by: 1) Ultrasonication in de-ionized water (DI) for 10 minutes with the mirror-like surface facing down; 2) Ultrasonication in 10% NH_4OH for 10 minutes to remove adhering particles followed by rinsing with DI water; 3) Ultrasonication with 50% aqua regia for 10 minutes to remove Fe-Cr metal followed by rinsing with DI water; 4) Ultrasonication with 5% HNO_3 for 10 minutes to remove chemical residues, followed by rinsing with DI water.

Results: The surfaces of flight SoS wafer fragments 60299, which was not pre-cleaned with ultra-pure water at NASA, and 60296, which was pre-cleaned with ultra-pure water at NASA, were analyzed by LA-ICP-MS. Coupled spikes of ^{52}Cr and ^{56}Fe were observed in both fragments, suggesting the presence of stainless steel particles on the wafer surface [2]. Since the laser tracks trapped solutions during subsequent cleaning steps, contributing to cross-contamination, two fresh fragments, 60297 and 60298, were put through the full cleaning procedure. The surface of flight SoS wafer fragment 60297 was analyzed afer the aqua regia bath and the final HNO_3 bath, separately, and the surface of flight SoS wafer fragment 60298 was analyzed after the final HNO_3 bath. Conversion of measured ion intensities to fluences were performed for Mg using the ^{25}Mg implant in SoS but a similar implant was not available for Fe. So the Fe/Mg atomic ratio was determined using a ratio of sensitivity factors determined on the NIST SRM 612 glass. The result is shown in Figure 1. The four analyses of GENESIS flight SoS wafer fragments form a hyperbolic curve in the Mg fluence-Fe/Mg plot, and they plot along a mixing line between the the canonical solar wind composition and the Utah Desert Dust. The canonical solar wind Mg fluence was taken from [3] and the Fe/Mg atomic ratio (0.838) from [4]. Data for the UTTR dust were collected previously by solution ICP-MS at FSU, and Fe/Mg was found to be 0.11.

Discussion: The cleaning procedure removed significant amounts of surficial contamination, and minimized but did not completely eliminate the Fe-Cr spikes observed by laser ablation. Since Cr was below detection limit except for surficial contamination, Cr provided an excellent screening tool for such particulates. We noted that a small background of Cr was observed in each line scan. Correlation of this Cr was found with Al (by varying laser power, etc.), and both were inferred to be derived from the underlying sapphire substrate. A correction for Cr from the substrate was performed for the flight wafers. Fe-Cr spikes were not included in the data used to calculate the fluences shown in Figure 1. Similarly, Mg spikes were evident in laser profiles on uncleaned flight samples, but fewer and smaller spikes were observed on the cleaned samples. Fluences for Mg were calculated from portions of the line scan that exhibited constant ^{24}Mg and ^{25}Mg profiles. The ^{25}Mg fluence in the implant is not known precisely (±10-20%) at present, and this must be factored into any interpretation of Figure 1.

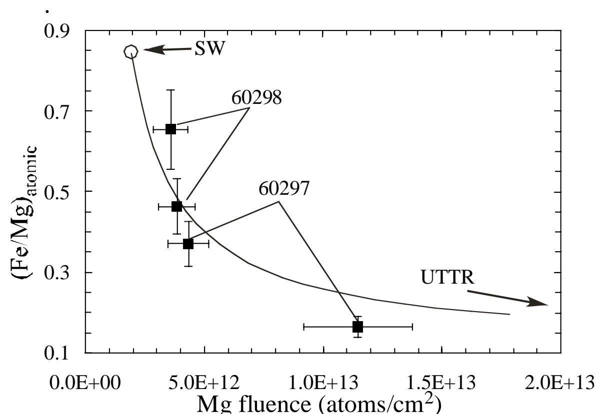

Fig. 1 Estimated Mg fluence and atomic Fe/Mg in cleaned GENESIS flight SoS wafer fragments 60297 and 60298 with 1 σ uncertainty plotted.

For 60297, two line scans were taken. The point showing the highest Mg fluence was taken after aqua regia cleaning but before 5% HNO_3 cleaning. The 60297 point taken after both cleaning steps has a fluence of about 4E12 atoms/cm^2. The difference of a factor of three decrease in Mg surface density is the justification for the 5% HNO_3 cleaning step. The two analyses taken on 60298 are from two separate line scans measured on different portions of the surface of the wafer after all cleaning steps. The Mg fluence estimated from both is 3.6-3.8 E12 atoms/cm^2. Although these fluences are higher than the pre-flight estimate of 2E12 atoms/cm^2 [3], most subsequent measurements of Mg fluence have been 20-40% higher than the pre-flight estimate. The Fe/Mg ratio may be a better indicator of the contamination level than noting that the measured Mg surfaces densities are systematically higher than pure SW values. A time-series of cleaning steps may better indicate the exact amount of effort required to remove all the contamination.

These data are neither sufficiently precise nor accurate to replace current estimates of the solar wind composition determined by other methods [4], but the data are of sufficient quality to advise us on the efficacy of the cleaning procedures we have adopted. These cleaning procedures will be applied to a larger flight SoS wafer fragment recently obtained from JSC Curatorial facility. The analysis of that sample will be performed by solution ICP-MS in which better standardization can be performed.

The recovery of a mixing line between canonical solar wind and UTTR dust/mud compositions is of significant importance. It was never clear that the contamination would produce a simple mixing line. This was because single large embedded mineral grains, not represented by the UTTR bulk analysis, might dominate the composition of the contaminants. Scanning electron microscopy [1] had revealed that the particulates > 10 μm were all either Si or Ge wafer fragments, with a few exceptions, and not mineral fragments. However, laser scanning of the surfaces of wafer fragments had revealed the presence of a ubiquitous layer of submicron contamination [1]. If this layer had formed by vapor deposition, it is not clear the bulk UTTR dust/mud would be an adequate representation. Thus, for Fe and Mg, we now know experimentally that simply mixing appears to be applicable. This test can then be applied to subsequent measurements made by solution ICP-MS to determine if UTTR contaminants might compromise those more precise and carefully calibrated measurements.

References:

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