

MEASURING MAGNESIUM ISOTOPES IN GENESIS SILICON COLLECTORS USING RASTERED ION IMAGING. E. C. Koeman-Shields¹, G. R. Huss¹, A. J. Westphal², R. C. Ogliore³, A. J. G. Jurewicz⁴, and D. S. Burnett⁵, K. Nagashima¹, ¹HIGP, Univ. of Hawai'i at Mānoa, 1680 East-West Road, Honolulu, HI 96822 (ekoeman@hawaii.edu), ²Space Sciences Laboratory, Univ. of California at Berkeley, Berkeley CA 94720, ³Dept. of Physics, Washington Univ. in St. Louis, One Brookings Drive, St. Louis, MO 63130 ⁴CMS, ASU, Tempe, AZ 85287-1404, ⁵Div. of Geol. and Planet. Sci., MC 100-23, Caltech, Pasadena, CA 91125.

Introduction: An accurate and precise measurement of the Mg isotopic composition for the solar wind is a high priority in both heliophysics and planetary science. Because Mg isotopes are fractionated by <1‰ among planetary materials (except CAIs), any significant fractionation in Mg isotopes in the Genesis samples probably reflects fractionation of solar matter during acceleration of the solar wind. That fractionation is not only a fingerprint of solar processes, but the data will certainly be used to refine Genesis measurements of other isotopic systems (eg., C, N, O, Li, S).

Measuring Mg isotopes in Genesis collectors is challenging. When the return capsule crashed on return to Earth, collectors shattered, their surfaces were damaged, and Utah dirt, brine and spacecraft debris was introduced as contamination on their surfaces. Large collector surfaces are no longer available for large area analytical techniques; moreover, the surface contamination is ubiquitous and the dirt provides orders of magnitude more Mg than the solar wind. Attempts at large area analysis by cleaning and piecing together small samples have been made [1]. However, secondary ion mass spectrometry (SIMS) is now the technique of choice because it can avoid residual areas of surface contamination.

Early SIMS work concentrated on the diamond-like carbon on silicon (DOS) collectors [e.g., 2, 3], but this material has inconsistent material properties that make quantitative SIMS measurements difficult [4]. Heber et al. [5] developed a backside profiling technique to measure Mg isotopes in Si collectors, which have consistent material properties. We have developed an improved method to measure solar wind Mg in Si collectors using backside ion imaging. This method removes the requirement for a perfectly flat thinned sample and allows for elimination of pixels that are contaminated by terrestrial materials [6]. Following [7], a Mg-ion implant put in from the backside is used to standardize the measurement internally, and the implant is calibrated by simultaneously implanting into a piece of NIST SRM 614 glass, which has a known amount of Mg [8].

Experimental Methods: A Genesis float zone Si collector chip from the B/C array (# 60635) was embedded in epoxy, solar-wind-exposed side down, in a one-inch Al round. The sample was ground and polished into a wedge with a ~0.2° angle. A piece of SRM

614 glass was also prepared. To ensure a good implant, both mounts were coated with ~100 angstroms of carbon. Both the glass and sample were partially masked to produce implanted and non-implanted areas. Then the mount was implanted (Leonard Kroko, Inc) with ²⁴Mg and ²⁶Mg at a nominal fluence of 5×10¹² atoms/cm² for each at an energy of 90 keV. Because the standard implant is put in from the backside of the chip, the wedged B/C chip ensures that there will be regions with adequate separation between the implant and the solar wind.

SIMS measurements were made using the Cameca ims 1280 at the University of Hawai'i. Prior to the measurements, ~200 angstroms of carbon was added to both mounts. The implanted SRM glass was measured as a standard depth profile with a ~4 nA O⁻ primary beam and a 50×50 μm raster. The B/C chip with its internal standard implant was analyzed using a 300 pA O₂⁺ primary beam focused to a 3 μm spot and an impact energy of 10 keV. Raster images (256×256 pixels) were collected over a 50 μm² area. We simultaneously collected images of ²⁴Mg, ²⁵Mg, and ²⁶Mg using multi-collector electron multipliers, with field jumps for ¹H and ³⁰Si. The measurement was made ~100 microns from the edge of the wedge in the implanted region; this thickness allowed ~300 cycles between measurement of the internal standard implant and that of the solar wind. The images were aligned in depth using the Si breakthrough into the epoxy (Fig. 1). After align-

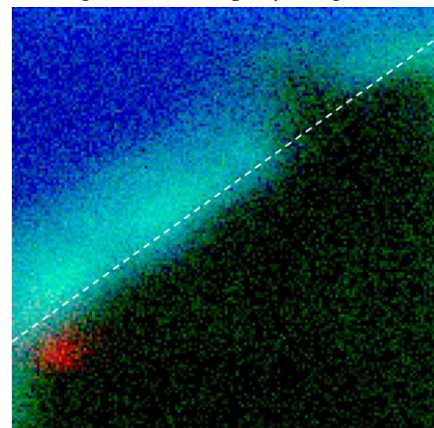


Fig. 1: Frame from the depth profile (Si = Blue, H = Green, Red = Mg). The white dashed line represents the Si breakthrough into the epoxy and was used to align the pixels by depth. The pixels in the Mg "hot spot" in the lower left hand corner were removed.

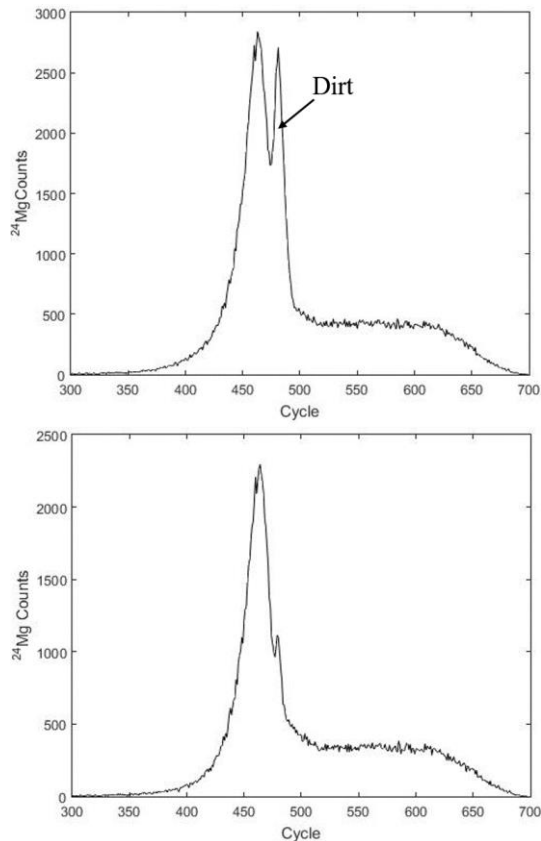


Fig. 2: Depth profile of ^{24}Mg truncated at the collector surface before (top) and after (bottom) dirt pixel correction.

ment, pixels were rejected that showed contamination from the Utah dirt as well as pits from the crash of the spacecraft (Fig. 2).

In Fig. 3, we show the depth corrected Mg profiles for all three isotopes with the dirt and pit pixels removed. After combining all pixels into a single depth profile for each isotope, data reduction includes subtracting the background Mg, integrating the depth profiles, calculating the isotope ratios from the integrated counts of individual profiles, and accounting for instrument mass fractionation (IMF). Because the standard implant is nominally (^{24}Mg , ^{26}Mg), ^{25}Mg gives an independent measure of the background Mg in the silicon and garden-in Mg from the surface. However, the ion implanter cannot separate ^{25}MgH from ^{26}Mg , so there is a small peak at ^{25}Mg that tracks the ^{26}Mg implant. To account for this, we adjusted the background-corrected, integrated profiles for the over-correction of ^{24}Mg and ^{26}Mg due to this peak. The IMF correction was determined from the intrinsic Mg in the SRM 614 glass, which was assumed to have normal terrestrial Mg-isotope composition. The IMF was then applied to the measured $^{26}\text{Mg}/^{24}\text{Mg}$ ratio of the standard implant to determine its “true” isotopic ratio. The measured

ratio of the standard implant in the Genesis chip was compared with this “true” ratio to get the IMF for the solar wind measurement.

Results and Conclusions: Our preliminary measurements are very encouraging. Although we have only been able to obtain one measurement to date, we are able to show that the isotopic ratios in solar wind Mg are within a few percent of the terrestrial values. We have known from the beginning that we will need many more measurements (e.g., 16) to obtain the statistics necessary to determine the solar wind composition precisely. Our single rastered measurement gives $\pm\sim 20\%$ (2σ) statistical uncertainties in measurements of $\delta^{25}\text{Mg}$ and $\delta^{26}\text{Mg}$. There are also systematic uncertainties associated with the IMF correction and the background correction that we currently estimate at $\pm\sim 14\%$ for $\delta^{25}\text{Mg}$ and $\pm\sim 28\%$ for $\delta^{26}\text{Mg}$. Future improvements in our methodology will include implanting ^{24}Mg and ^{26}Mg at higher energy (making the implants deeper) to better separate the implants from surface contamination and increasing the fluence of the implants by a factor of 10 to make them easier to measure in the SRM 614 glass. We also plan to raster a wider area and increase the beam current in order to improve the counting statistics of each depth profile while keeping adequate depth resolution. With these and other modifications, we are confident that we will be able to precisely measure the Mg isotopic composition of the solar wind.

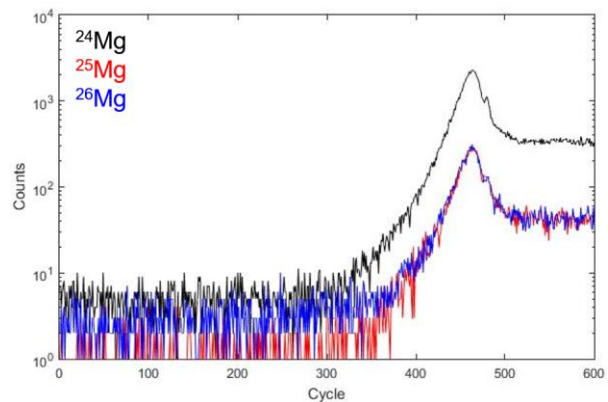


Fig 3: Depth profiles for ^{24}Mg , ^{25}Mg , and ^{26}Mg after corrections.

References: [1] Humayun (2012) 75th Met. Soc. Abstract #5353. [2] Reick et al. (2010) LPSC XLI, Abstract #2391. [3] Jurewicz et al., (2011) LPSC XLII #1917. [4] Jurewicz et al. (2016) LPSC XLVII, Abstract #2350. [5] Heber et al. (2011) 74th Met. Soc. Abstract #5510. [6] Westphal et al. (2014) LPSC XLV, Abstract #2671. [7] Rieck et al. (2014) LPSC XLV, Abstract #1758. [8] Gao et al. (2002) *Geostan. & Geoanal. Res.* 26, 2, 181-196. Supported by NASA Grants NNX14AF25G and NNX17AE73G to GRH. Funds from NNX14AF26G to AJGJ aided this work.