

DUAL BEAM SPUTTER DEPTH PROFILING OF GENESIS SOLAR WIND COLLECTORS BY RIMS.

I. V. Veryovkin¹, S. V. Baryshev¹, D. S. Burnett², M. J. Pellin¹, C. E. Tripa¹, and A. V. Zinovev¹, ¹Materials Science Division, Argonne National Laboratory, Argonne, IL 60439, USA, verigo@anl.gov, ² Division of Geological and Planetary Sciences, California Institute of Technology, Pasadena, CA 91125, USA

Introduction: Solar wind (SW) samples collected by the NASA Genesis Mission are an example of extreme analytical challenge. It is particularly true for analyses aiming at determination of elemental abundances of various metals present in the SW samples at trace concentrations. These SW implants are buried under a “blanket” of terrestrial contamination, which has the same elements with orders of magnitude higher abundances. The outcome of trace analyses in this case depends not only on capabilities of a particular piece of analytical equipment but also on the ability of its operators to develop “finely tuned” protocols for sample preparation and analyses. At Argonne National Laboratory, we have developed uniquely sensitive Resonance Ionization Mass Spectrometry (RIMS) instrument for analysis of Genesis samples, SARISA. After the landing of the Genesis spacecraft, we have been developing and testing optimized approaches to the analysis of returned SW samples with RIMS. Finally, we have developed our own variant of sputter depth profiling method, which has ultra-high depth resolution (sub-nanometers) needed for unambiguous distinction between the sputtered and photo-ionized surface species belonging to the contamination and those from the implanted in Genesis collectors SW matter.

Experimental: In this contribution, we will report results of our multi-element RIMS measurements of elemental abundances in four Genesis samples, #60758 and #60428 (bulk SW), #60307 (slow SW, interstream, IS) and #60776 (fast SW from coronal holes, CH). Measurements of elemental abundances corresponding to different SW regimes can reveal the corresponding specific elemental fractionations [1]. Particularly, slow solar wind is considered to be more variable and biased in elements with low first ionization potentials (FIPs). Uniquely high sensitivity of the SARISA RIMS instrument makes it the perfect tool for such studies of SW elemental abundances. For the experiments reported here, we chose Ca, Cr and Mg (FIPs = 6.113 eV, 6.766 eV and 7.646 eV, respectively). The recent major upgrade of the SARISA instrument allowed us to use four newly designed and constructed Q-switched tunable Ti-sapphire lasers to simultaneously perform Resonantly-Enhanced Multi-Photon Ionization (REMPI) of these elements by optimized two-photon schemes [2]. Neutral atoms sputtered by a pulsed 5 keV Ar⁺ ion beam (500 ns pulse length) were converted into photo-ions for time-of-flight mass spectrometry (TOF MS). Concentration versus depth profiles of SW

implants were obtained using two dedicated ion sources by a sequence of alternating sessions of ion milling with a raster scanned normally incident primary ion beam in continuous mode, and TOF MS analysis with scanned pulsed primary ion beam with 60° incidence angle. The ultra-high sub-nm depth resolution of such analyses was enabled by the normal incidence geometry of the ion milling, which allowed us to reduce the beam energy from 5 keV of extraction from the ion source to 250 eV on impact. This made the ion milling process extremely gentle and essentially eliminated ion mixing (“gardening”) artifacts that in the past did not allow us to detect shallow concentration peaks of SW implants. Another key contributor to the reduction of artifacts is the quasi-in-situ CO₂ snow jet cleaning method we implemented last year [3]. The synergy of this combination of sample preparation and analysis procedures allowed us to greatly improve the reproducibility of our RIMS analyses of Genesis SW samples.

Results: The analyzed in this work Genesis samples were not subjected to other surface treatment except for usual megasonication in ultra-pure water. One exception was the sample #60428, which was partially irradiated by Gas Cluster Ion Beam to evaluate the efficiency of this method for sample cleaning. On sample #60758, we conducted five analyses, two on each half of #60428, and one on each #60776 and #60307. The profiles are illustrated and discussed in Figures 1 and 2. For the SW fluences summarized in Table 1, one should keep in mind that we did not use any advanced depth profile integration algorithms capable of improving signal-to-noise ratio and better separating SW from contamination artifacts. The integrals were obtained by visually choosing the range between “saddle points” separating SW implants from the surface contamination on the left and from the background baseline on the right. At the Conference, we will present results of a more sophisticated data processing. By that time, we also will be able to conduct more measurements on fast and slow SW regimes samples.

Conclusion: In this work, we have demonstrated a breakthrough improvement in RIMS analysis of SW samples and for the first time were able to study different SW regimes and estimate elemental fluences for Mg, Ca and Cr..

References: [1] N. A. Schwadron et al. (1999) *ApJ* 521, 859, [2] I. V. Veryovkin et al., (2010) LPSC XLI, Abstract #2579, [3] I. V. Veryovkin et al., (2011) LPSC XLII, Abstract #2308.

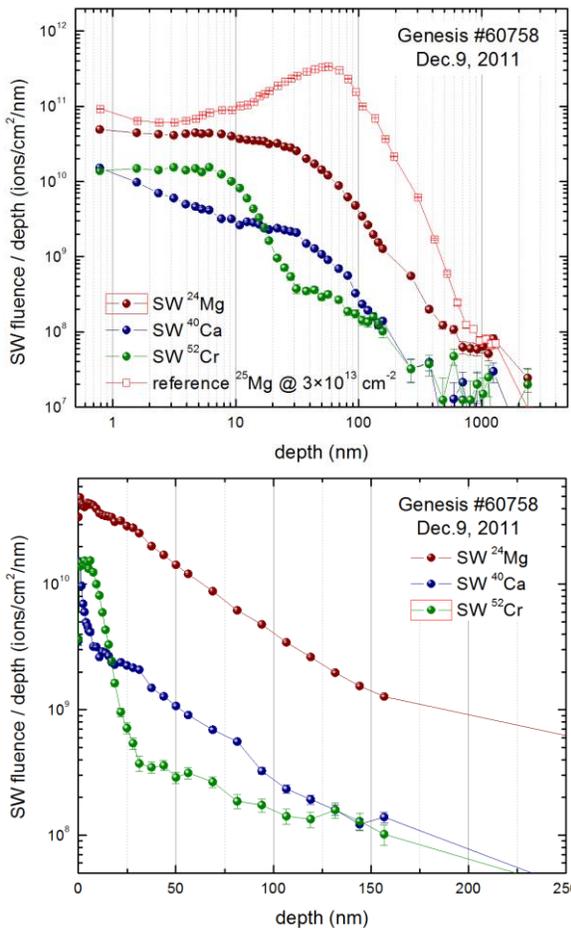


Figure 1. Depth profiles of SW ^{24}Mg , ^{40}Ca and ^{52}Cr , calibrated (1) in intensity against the corresponding 2 keV/amu reference ion implants ^{25}Mg , ^{44}Ca and ^{53}Cr (^{25}Mg shown for comparison) and (2) in depth using the white light interferometry. For Mg, this measurement showed *almost no artifacts from surface film contamination and particulates*. For all elements, it is possible to clearly distinguish the Solar Wind from the contamination. The SW profiles reconstructed from such experiments can be directly compared to Monte-Carlo (TRIM-based) simulations of the solar wind implantation.

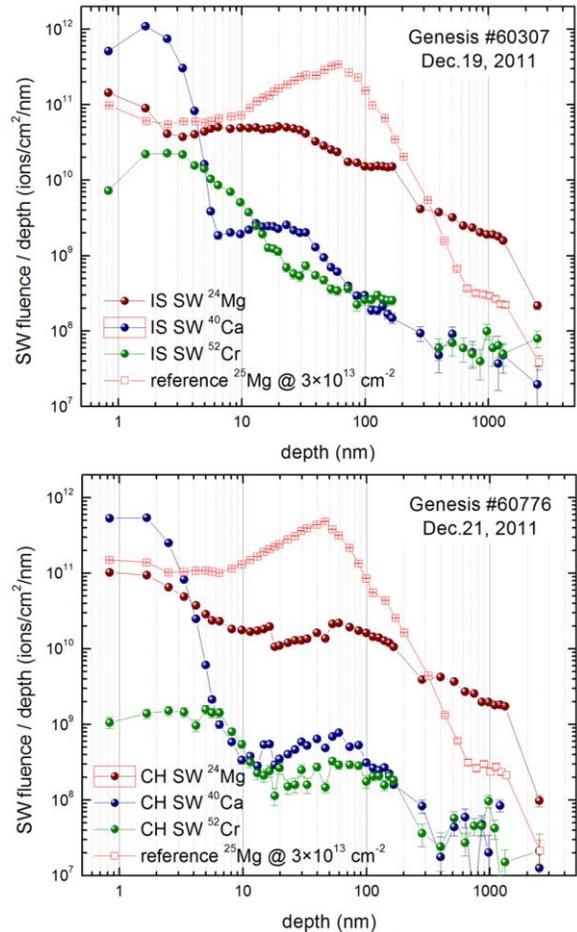


Figure 2. The *very first* experimentally measured sputter depth profiles of slow (interstream, IS, top) and fast (coronal holes, CH, bottom) SW for ^{24}Mg , ^{40}Ca and ^{52}Cr , calibrated in the same way as those in Fig.1. For the fast SW, we can see that the implant concentrations peak at depth of ~ 60 nm. In contrast, for slow SW, we detected that the implant concentrations peaked at ~ 20 nm below the collector surface (similar to the bulk SW in Fig.1). Our attempts to determine SW fluences for CH and IS regimes resulted in artificially high fluences of Mg for both (see Table 1). Apparently, the tails of profiles at high depths (typical for particulate artifacts) are responsible for this. *But data on Ca have NO artifacts!*

Table 1

Sample #	Date /2011	SW elemental fluences (ions/cm ²)				
		^{24}Mg	^{25}Mg	^{26}Mg	^{40}Ca	^{52}Cr
60758	11/28	3.5×10^{12}	4.1×10^{11}	4.9×10^{11}	3.6×10^{11}	5.1×10^{10}
60758	11/30	1.5×10^{12}	2.0×10^{11}	2.2×10^{11}	8.5×10^{10}	5.0×10^{10}
60758	12/02	2.0×10^{12}	2.4×10^{11}	2.5×10^{11}	8.9×10^{10}	3.2×10^{10}
60758	12/06	3.0×10^{12}	4.1×10^{11}	4.7×10^{11}	1.7×10^{11}	4.0×10^{10}
60758	12/09	2.0×10^{12}	2.5×10^{11}	2.9×10^{11}	1.3×10^{11}	1.3×10^{10}
60758 average	5 spots	2.8×10^{12}	3.0×10^{11}	3.4×10^{11}	1.6×10^{11}	3.7×10^{10}
60428 (GCIB)	12/13	1.2×10^{12}	1.4×10^{11}	1.7×10^{11}	8.5×10^{10}	2.1×10^{10}
60428 (pristine)	12/15	1.7×10^{12}	2.0×10^{11}	2.3×10^{11}	4.1×10^{11}	6.1×10^{10}
60307 (IS/slow)	12/19	5.0×10^{12}	5.7×10^{11}	6.0×10^{11}	9.8×10^{10}	4.3×10^{10}
60776 (CH /fast)	12/21	3.2×10^{12}	2.6×10^{11}	3.5×10^{11}	7.0×10^{10}	3.4×10^{10}

Acknowledgements: This work is supported by is supported by NASA through grants NNH08AH761 and NNH09AM481, and by UChicago Argonne, LLC, under contract No. DE-AC02-06CH11357