DEPTH PROFILING OF GENESIS DIAMOND-ON-SILICON COLLECTORS : DIRECT COMPARISON BETWEEN FRONTSIDE AND BACKSIDE APPROACHES. I. V. Veryovkin[1], A. V. Zinovev[1], C. E. Tripa[1], and D. S. Burnett[2], [1]Materials Science Division, Argonne National Laboratory, Argonne, IL, USA, verigo@anl.gov, [2]Division of Geological and Planetary Sciences, California Institute of Technology, Pasadena, CA, USA

Introduction: Solar wind (SW) collectors of the Genesis mission contain SW matter implanted in a shallow region of up to ~200 nm under their surface. To meet the mission goals, isotopic and elemental compositions of these samples must be precisely and quantitatively analyzed. Because of the heavy contamination of sample surfaces originating from the crash landing of the Genesis sample return capsule, these analyses are a serious challenge, especially, when SW elements with relatively low concentrations are of interest. This is why, for analytical techniques such as SIMS and RIMS that rely on sputter depth profiling, a search for solutions helping improve discrimination between SW signal and the contamination is ongoing. One such solution that emerged is *backside depth profiling* [1]. This technique greatly reduces ion mixing artifacts thus improving overall accuracy and precision. We tested this approach with RIMS on Genesis Si sample #60176 prepared for backside depth profiling using mechanical polishing equipment at the Argonne Electron Microscopy Center [2]. Although we then demonstrated a noticeable reduction of ion mixing artifacts, we did not obtain any quantitative results on SW Mg fluence because of contamination introduced by the polishing. Our SIMS colleagues at Caltech, UCLA and ASU have later demonstrated a very respectable progress in backside depth profiling of Genesis Si samples [3]. However, the mechanical thinning of Si samples hasserious difficulties in measurements of depth profile regions close to the interface between the sample and the substrate it is glued on. At the last year LPSC, we have presented a new approach to backside depth profiling, which is uniquely suitable for diamond-on-silicon (DOS) SW collectors [4, 5] and, most importantly, is highly reproducible. We obtained two Genesis flight samples to test this approach, # 61389 and #61387. The DOS sample # 61389 was the very first trial reported last year [4]. In 2013, we continued our work on optimization of the sample preparation procedures and sputter depth profiling protocols. In this presentation, we report results of the very first "*apple-to-apple*" comparison between two approaches to depth profiling of Genesis DOS samples: #61387 from the backside and #60622 from the frontside.

Experimental. Mechanical polishing does not work well with DOS collectors: their thin diamond-like (DLC) film tends to chip when the Si substrate is abraded away. On the other hand, DOS collectors have important advantages: (1) reduced diffusion of SW species, and (2) high hardness resulting in fewer surface scratches that could accumulate irremovable contamination. The other very important advantage of DOS collectors is that the DLC film is chemically inert, *enabling non-mechanical removal of the more reactive Si substrate by dry chemical etching with XeF_2 gas* via reaction $2XeF_2 + Si \rightarrow 2Xe + SiF_4$ [6]. Over the last year and a half, we have conducted a special series of experiments at the Argonne Center for Nanoscale Materials to identify sample mounting arrangements and dry etching regimes that reproducibly yield intact and perfectly flat DLC films ready for RIMS ([4] and this work) and SIMS [5] analyses. Our results obtained with the sample #60389 indicated that the surface of the invar metal substrate was too rough and non-flat, which resulted in difficulties with identification of the moment when analysis beam breaks through the sample surface into the SEM epoxy glue. Moreover, the substrate surface contained too many impurities that potentially could affect analyses of low abundance elements. To solve this problem, we mounted the second Genesis DOS sample (#60387) on a semiconductor-grade GaAs wafer, which is not only smooth and clean but also makes very good compositional contrast with DLC, thus enabling simple mass spectrometric detection of the moment when analysis ion beam goes through the DLC film into the substrate. The flight sample #60622 for frontside depth profiling was mounted on Al SEM stub with silver epoxy. Also, for each Genesis sample we have prepared a reference (standard) sample – using exactly the same procedure as for the flight samples. This approach enables measurements of the Genesis samples and the standards in the same fashion, which is necessary for minimizing analysis errors. For the standards, we used the same DOS material as in the flight samples, but implanted with 2 keV/amu $^{25}Mg/^{44}Ca/^{53}Cr$ ions at ion fluence of 5×10^{12} cm^{-2}.

After developing optimal sample preparation procedures, we conducted a series of RIMS experiments with the SARISA instrument at Argonne to identify optimal depth profiling approaches. Last year, we concluded that, for the backside, the single beam approach yielded better results [4] and higher analytical throughput than the time-consuming (but more accurate) gentle dual beam approach we developed for the frontside depth profiling of Si samples [7]. In these comparative experiments, we used Ar^+ ion beam with 10 keV energy and 60° incidence angle. The depth profiling measurements were done in exactly the same way from the

front and back sides: 1 sec raster scanning steps in a continuous current mode for milling 400×400 μm^2 square crater, each such step followed by RIMS analyses of the center of the crater (in pulsed current mode, with raster scan off). We have measured about a dozen spots on each sample. Typical depth profiles are shown in Fig.1. From profiles measured on the standards, one can clearly see for the frontside that ion mixing artifacts that are more pronounced for Ca than for Mg. On the other hand, while for the backside, the interface region between the sample and GaAs substrate was better and sharper than we measured for invar [4], it remains "blurred". For depth profiles SW Mg in Genesis samples, we observed very good agreement between both approaches. The advantage of backside approach was obvious from comparison of profiles of SW Ca: while surface contamination almost "buried" SW signal from the frontside, it was clearly distinguishable from the backside. While relatively high blank level of Ca impurity in the DOS collectors did not permit high precision measurements of SW Ca fluences, for other low abundance elements, our backside approach might be the only solution.

For Mg, the average SW fluences determined by both approaches showed a very good agreement: $(1.46\pm0.11)\times10^{12}$ cm^{-2} for the backside and $(1.48\pm0.05)\times10^{12}$ cm^{-2} for the frontside, under the assumption that our standards had been implanted with 5×10^{12} cm^{-2}. But for the frontside a few spots had to be discarded because surface contamination was too severe there, which was not the case for the backside.

Conclusions: We have developed a new approach to backside depth profiling of Genesis DOS collectors and validated it by comparison with the frontside approach, applied under exactly the same experimental conditions. For quantitative RIMS analyses, the new approach has advantages over the conventional frontside approach because it has noticeably better reproducibility and about the same experimental throughput with single ion beam.

Acknowledgements: This work is supported by NASA through grant NNH09AM48I, and by UChicago Argonne, LLC, under contract No. DE-AC02-06CH11357. Use of the Center for Nanoscale Materials was supported by the U. S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.

References: [1] Palmstrøm C. J. et al. (1990) *J. Appl. Phys.* 67, 334. [2] Veryovkin I. V. et al. (2008), *39th LPSC* #2396. [3] Heber V. S. et al., (2010), *41st LPSC* #2234; (2011), *42nd LPSC* #2642; (2012), *43rd LPSC* #2921; (2013), *44th LPSC* #2540 and #3028. [4] Veryovkin I. V. et al. (2013), *44nd LPSC* #2247. [5] Rieck K. D. et al. (2013), *44nd LPSC* #3030 [6] Winters H. F. and Coburn J. W. (1979) *Appl. Phys. Lett.* 34, 70. [7] Veryovkin I. V. et al. (2011), *42nd LPSC* #2308; and (2012), *43rd LPSC* #2296.

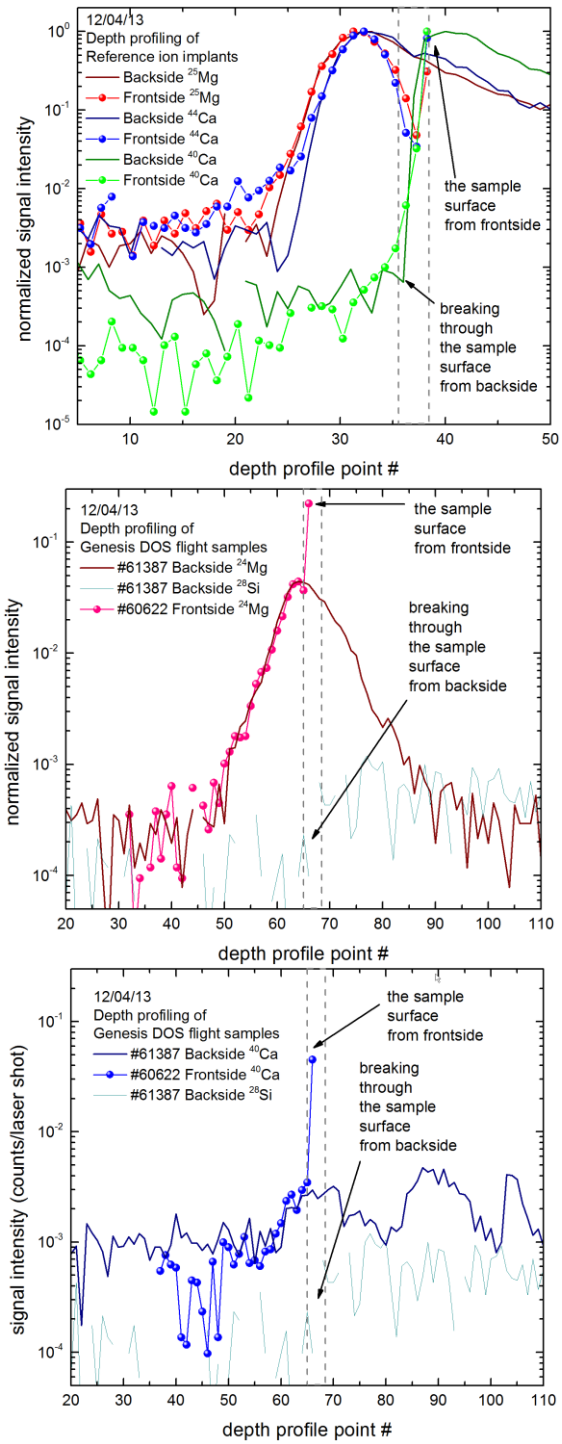

Figure 1. Direct comparison of frontside and backside depth profiling for standards (top plot) and two Genesis samples (middle plot for ^{24}Mg, and bottom plot for ^{40}Ca).