Depth Profiling of Genesis Diamond-on-Silicon Collectors: Direct Comparison Between Front side and Backside Approaches

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Motivation

Solar wind (SW) collectors of the Genesis mission contain SW matter implanted in a shallow region of ~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 analyzes 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***. This technique greatly reduces ion mixing artifacts thus improving overall accuracy and precision.

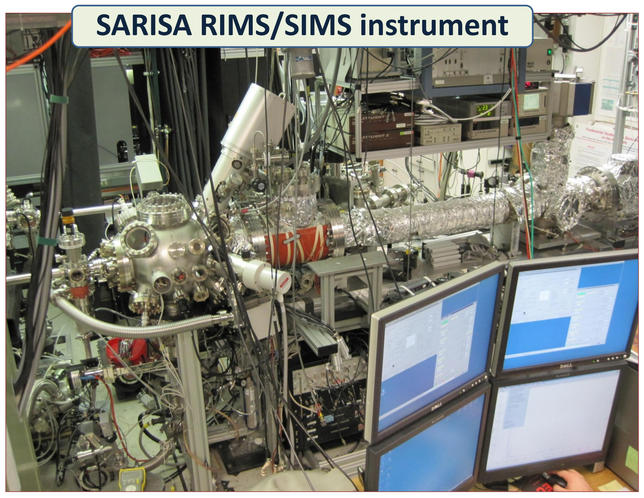
We tested this approach with RIMS on Genesis Si sample #60176 prepared for backside depth profiling using mechanical polishing equipment at the Argonne Electron Microscopy Center [LPSC-2008 #2396]. Although we then demonstrated a noticeable reduction of ion mixing artifacts, we did not obtain any quantitative 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 [Heber V. S. et al., LPSC-2010 #2234; LPSC-2011 #2642; LPSC-2012 #2921; LPSC-2013#2540 and #3028].

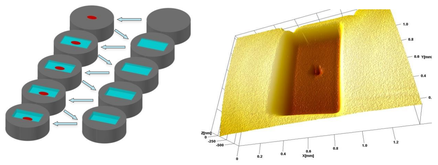
However, the mechanical thinning of Si samples remains hardly reproducible and causes serious difficulties in measurements of depth profile regions with max SW concentrations - because they are too close to the interface between the sample and the substrate it is glued on. At LPSC2013, we have presented a new approach to backside depth profiling, which is uniquely suitable for diamond-on-silicon (DOS) SW collectors [#2247 and #3030] 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 [LPSC-2013 #2247]. During 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 front side***.

SARISA RIMS/SIMS instrument





Experimental Procedure

Concentration versus depth profiles of SW implants in Genesis samples were obtained in the "classic" *Single Beam regime* using the same 10 keV Ar^+ ion beam with 60° incidence angle for both ion milling and for for time-of-flight mass spectrometry (TOF MS) analysis.

For ion milling, the ion beam was raster scanned over an area of 250×250 μm² in continuous (direct current, DC) mode. One ion milling (depth profiling) step corresponded to 1 s (one full frame) of the scanning. Then the center of the area was probed by the same beam in pulsed mode (5×10^4 pulses 400 ns each). Neutral atoms sputtered by the pulsed were converted into photo-ions by REMPI and then analyzed by TOF MS.

For front side depth profiling, no surface cleaning by ion irradiation was needed: the depth profiling analyses began immediately from the sample surface.

Prior to backside depth profiling, the same primary ion beam was raster scanned in DC mode over the same area for 100-200 s – to reduce the sample thickness in the analysis region.

Sample Preparation

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 and SIMS analyses.

Our results obtained with the Genesis DOS 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.

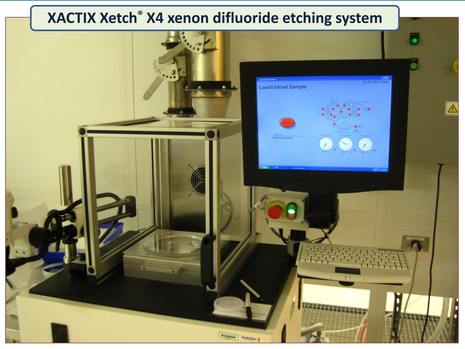
To solve this problem, we mounted the second Genesis DOS sample (#60387) on a semiconductor-grade GaAs wafer, which is not only smooth and clean but also makes very good compositional contrast with DLC, thus enabling simple mass spectrometric detection of the moment when analysis ion beam goes through the DLC film into the substrate.

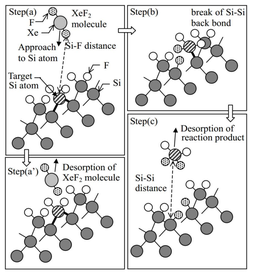
The flight sample #60622 for frontside depth profiling was mounted on Al SEM stub with silver epoxy.

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}.

XACTIX Xetch® X4 xenon difluoride etching system

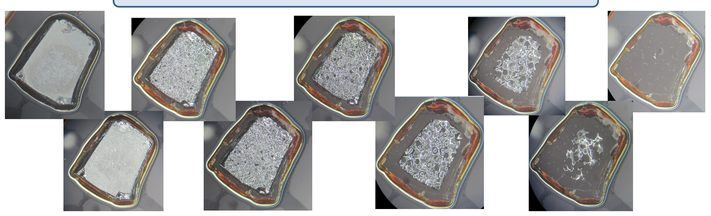




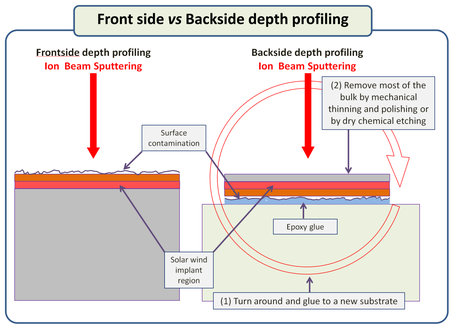
Dry etching of Si with XeF_2:

$$2XeF_2 + Si \rightarrow 2Xe + SiF_4$$

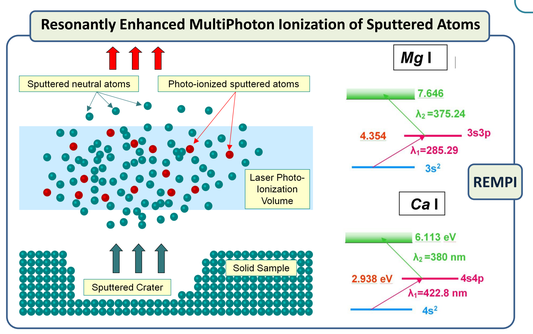
Dry etching of Genesis DOS # 61387 mounted on GaAs substrate



Front side *vs* Backside depth profiling

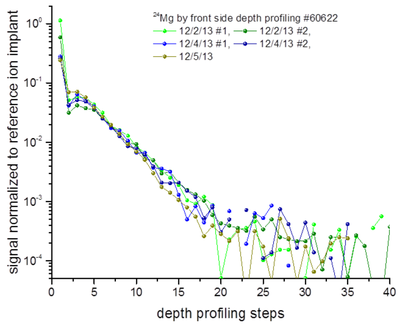


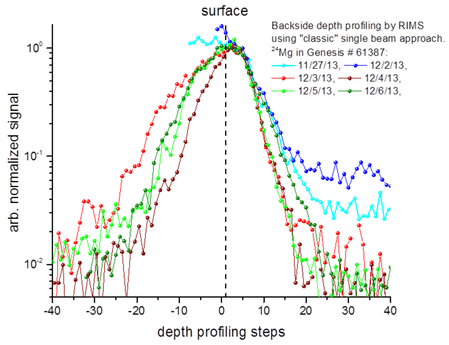
Resonantly Enhanced MultiPhoton Ionization of Sputtered Atoms

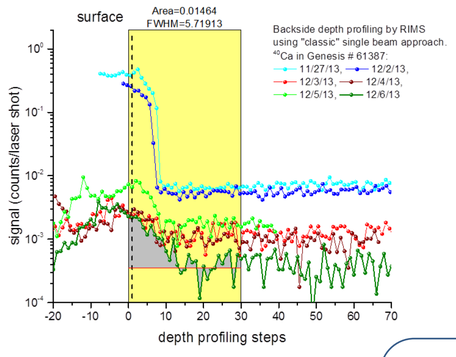


Backside	SW Fluence ^{24}Mg	SW Fluence ^{25}Mg	SW Fluence ^{26}Mg	SW Fluence all Mg	SW Fluence ^{40}Ca
Expt. Date					
Nov. 27 2013	1.21E+12	1.62E+11	1.73E+11	1.54E+12	
Dec. 2 2013	1.12E+12	1.44E+11	1.63E+11	1.43E+12	
Dec. 3 2013	7.01E+11	8.57E+10	1.08E+11	8.95E+11	9.31E+10
Dec. 4 2013	1.37E+12	1.70E+11	1.74E+11	1.71E+12	8.58E+10
Dec. 5 2013	1.55E+12	1.92E+11	2.07E+11	1.94E+12	1.60E+11
Dec. 6 2013	1.29E+12	1.55E+11	1.60E+11	1.60E+12	1.16E+11

Front side	SW Fluence ^{24}Mg	SW Fluence ^{25}Mg	SW Fluence ^{26}Mg	SW Fluence all Mg	SW Fluence ^{40}Ca
Expt. Date					
Dec. 2 2013	1.58E+12	1.73E+11	1.93E+11	1.94E+12	4.51E+11
Dec. 2 2013	1.73E+12	2.26E+11	2.26E+11	2.19E+12	2.54E+11
Dec. 4 2013	1.43E+12	1.65E+11	1.77E+11	1.77E+12	1.79E+11
Dec. 4 2013	1.33E+12	1.58E+11	1.80E+11	1.67E+12	1.02E+11
Dec. 5 2013	1.47E+12	1.73E+11	1.91E+11	1.83E+12	8.90E+10



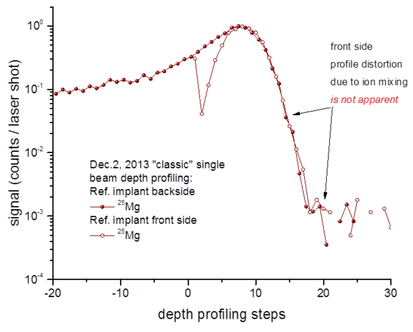


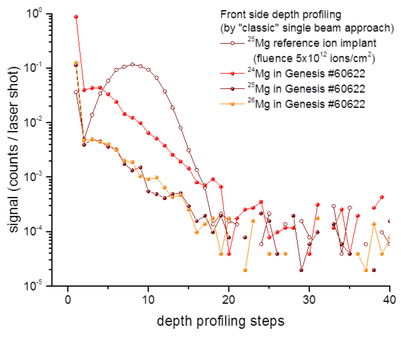


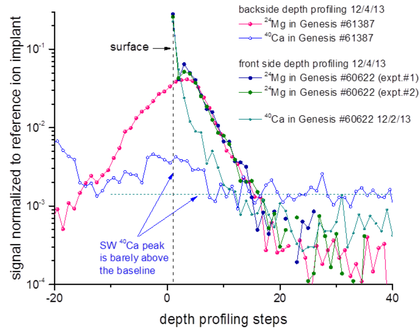
Average SW fluences:

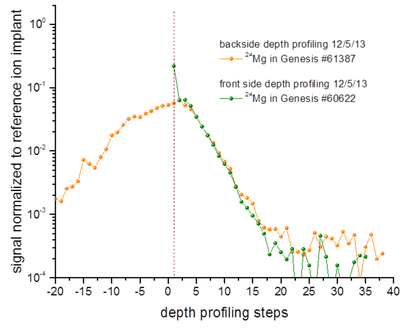
Mg (backside):
$(1.65\pm0.19)\times10^{12}$ cm^{-2}
^{40}Ca (backside):
$(1.14\pm0.33)\times10^{11}$ cm^{-2}

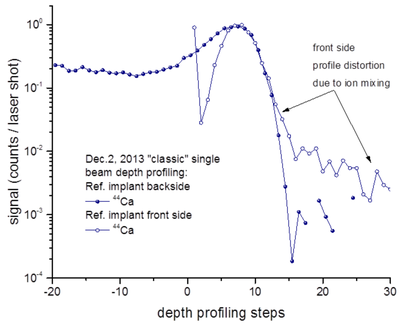
Mg (front side):
$(1.88\pm0.20)\times10^{12}$ cm^{-2}
^{40}Ca (front side):
$(1.56\pm0.76)\times10^{11}$ cm^{-2}

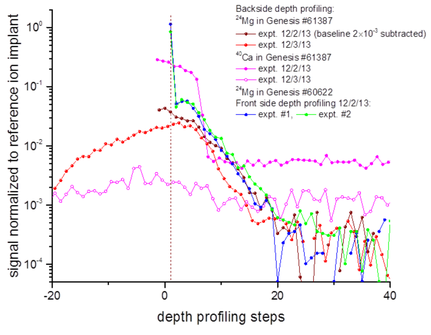


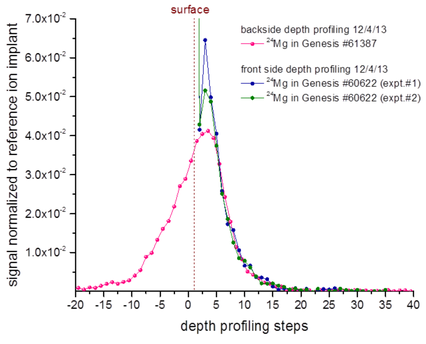


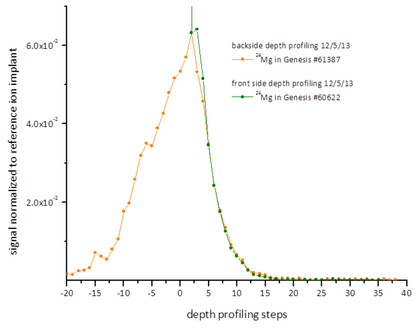












Summary and Conclusions

We have developed a new efficient approach to backside depth profiling of Genesis DOS collectors and validated it by comparison with the front side approach applied under exactly the same experimental conditions.

Results:

(1) SW fluences for Mg overall look consistent with our previous measurements. Precision is near the target value from the Genesis mission objectives: 11.8% for backside and 10.4% for front side. However, accuracy is still questionable because the discrepancy between average fluences obtained by two approaches is 13%.

(2) Accurate measurement of SW Ca with the front side approach is hopeless, while with the backside approach it looks more promising and feasible.

(3) Comparison of reference ion implants measured by both methods reveals that (a) ion mixing artifacts with front side approach are not apparent for Mg and very noticeable for Ca, (b) near surface regions of profiles measured by backside approach are distorted/smeared. Both kinds of artifacts suggest that high depth resolution is needed to accurately measure not only Genesis samples but also standards.

(4) It is a challenge to properly take surface contamination into account with the front side approach. This is why SW fluences determined by the front side approach can appear higher than those obtained by the backside approach. This explains the observed discrepancy in SW fluences.

Something to keep in mind using this approach:

(1) It is applicable to RIMS, SIMS and laser ablation MS

(2) DOS collectors might have high blank concentrations for some SW elements. More work is needed to determine what are the best elements to measure with this approach.

(3) Careful monitoring and matching of thicknesses of DLC films on Genesis sample and reference ion implant is required to perform accurate quantitative analysis.

(4) Temperature stability of the dry etched DOS samples prepared for backside depth profiling should be assured, because thermal expansions and contractions can lead to cracking of the DLC film.

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