Accurate analysis of shallow solar wind ion implants by SIMS backside depth profiling

V.S. Heber¹, K.D. McKeegan¹, S. Smith², A.J.G. Jurewicz³, C. Olinger⁴, D.S. Burnett⁵, Y. Guan⁵

¹ Dept. Earth, Planetary, and Space Sciences, UCLA, Los Angeles, CA, USA, heber@ess.ucla.edu; ² Evans Analytical Group, Sunnyvale, CA, USA; ³ Arizona State University, Tempe, AZ, USA; ⁴ LANL, Applied Modern Science Group, Los Alamos, USA; ⁵ California Institute of Technology, Pasadena, CA, USA.

We describe a method for quantitatively determining the fluences of shallowly-implanted solar wind (SW) ions returned to Earth by the Genesis Discovery mission [1,2]. Through backside depth-profiling, we can recover nearly complete depth profiles of implanted SW for several non-volatile elements including Mg, Al, Ca, Cr, and to a lesser extent, Na, in Genesis targets that collected bulk SW and solar wind from specific velocity regimes. We also determine fluences of the volatile elements C, N, and O in non-concentrated targets that collected bulk solar wind. Fluences as low as 2×10¹⁰ atoms/cm² can be determined with precision and accuracy typically in the few percent range. Specific approaches to sample preparation, sputtering artifacts during in-depth analysis by secondary ion mass spectrometry, and quantification are discussed [3].

**Introduction**

We documented our approach for quantitatively analyzing fluences of shallowly-implanted SW ions. Main analytical challenges overcome by backside depth-profiling are mixing of residual surface contaminants into the SW signal and transient sputtering. With low impact energy sputtering we recover nearly complete depth profiles of non-volatile elements, and ~80% complete profiles for C,N,O. Careful modeling allows to obtain precise integrated depth profiles for all 8 elements investigated here.

**Sample analysis**

Each element was measured separately (except Mg, Na) to maximize data coverage over SW peak and down the front-side of the profile. Standard-sample bracketing applied (standards: artificial implants, derivation of S and RSF [5]; this procedure is crucial for SW procedure analysis: relative fluences may differ by only some to ~10% [6,7].

An increased mobility of Na in Si can be excluded based on our data.

**Data reduction in backside depth profiles**

C,N,O break-through into the SiO₂ layer and epoxy occurred near the peak in all profiles. Mg, Ca, Cr, Al resulted in nearly complete profiles, Na was in-between.

For accurate fluences we must account for incomplete recovery of the entire depth profile by adjustment of SRIM curve [2,8]. No Na contamination due to Na.

**Summary**

We present the fits used to extrapolate from the last “good” measured point to the real target surface for D₂ (distance) and F₂, e, f depict profiles with no or few data at the front-side. Here complete characterization of Mg (of the same SW regime) was used to obtain the fitting parameters.


**Backside depth profiling**

Edge-on view of a backside sample. The gray-scale gradient in the Si target mimics SW concentration distribution with the highest concentration close to the front-side.

Area and sputter pit are without marks up to 3000s (a,b); >3200s a hole opened (c,d) and created a pathway to the near-surface, unrecovered portion of the Si target: sufficient space between contamination of C,N,O.

800nm is the optimum thickness of a backside sample: sufficient space between contamination of rear surface and high energy tail of SW (600 nm from front-side) for accurate background-level characterization at e.g. 200nm sputtering time.

Documentation of the original sample surface to avoid “breakthrough” by hidden scratches.

**SIMS analytical conditions**

<table>
<thead>
<tr>
<th>Element</th>
<th>Cameca</th>
<th>7F-Geo (Caltech)</th>
<th>8TR (UCLA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C, N, O</td>
<td>Cs⁺</td>
<td>5 keV</td>
<td>7 keV</td>
</tr>
<tr>
<td>Na, Mg, Al, Ca, Cr</td>
<td>Cs⁺</td>
<td>5 keV</td>
<td>7 keV</td>
</tr>
<tr>
<td>Primary beam stuck</td>
<td>100 x 100 µm</td>
<td>100 x 100 µm</td>
<td></td>
</tr>
<tr>
<td>Impact energy</td>
<td>5 keV</td>
<td>7 keV</td>
<td></td>
</tr>
<tr>
<td>Raster aperture</td>
<td>12 µm</td>
<td>40 µm</td>
<td></td>
</tr>
<tr>
<td>E-gating</td>
<td>50-70%</td>
<td>none</td>
<td></td>
</tr>
<tr>
<td>O₂ flood</td>
<td>no</td>
<td>1.1×10⁻¹⁰ Torr</td>
<td></td>
</tr>
<tr>
<td>Sputter rates (S)</td>
<td>0.1 – 0.24 nm/s</td>
<td></td>
<td></td>
</tr>
<tr>
<td>M/AM</td>
<td>Adjusted to the respective element</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Low impact energy sputtering results in high depth resolution.

Flat-bottomed crater for uniform break-through at the end of analysis.

84Ca⁺ depth profile measured in fast SW vs. number of cycles (δD=0.5nm per cycle). Different gray-scales in the photographs (taken during analysis) reflect changes in reflectivity as the beam penetrated through the fringes in Si into the epoxy and were used to assess the flatness of the crater bottom. The break-through through the surface into epoxy is characterized by an immediate and strong increase of the count rate (red) due to the contaminating ions.

Figs. 1 and 2 present examples of measured backside depth profiles in comparison with the respective SRIM model curves (blue, Fig. 1) and in Fig. 2 with the respective fits used to extrapolate from the last “good” measured point to the real target surface for D₂ (distance) and F₂, e, f depict profiles with no or few data at the front-side. Here complete characterization of Mg (of the same SW regime) was used to obtain the fitting parameters.

**References:**