

SIMS-AMS METHOD FOR MEASURING SOLAR WIND SILICON IN DLC GENESIS COLLECTORS.

C. Cetina¹, K.S. Grabowski², D.L. Knies², and L.T. Demoranville³, ¹Nova Research, Alexandria, VA 22308, email: catalina.cetina@nrl.navy.mil, ²Naval Research Laboratory, Washington DC 20375, ³University of Maryland, College Park, MD20742

Introduction: SIMS analysis is widely used for Genesis sample measurements at various institutions, but only two laboratories [1,2] offer accelerator-based SIMS. The use of a tandem accelerator allows for breakup of molecules during the acceleration process, thus assuring removal of molecular interferences without significant loss in sensitivity. Additionally, both mass spectrometers [1,2] are capable of simultaneous analysis of different ion species. The MegaSIMS [1] instrument has already successfully provided high-quality oxygen isotope ratio results. We are illustrating the use of the Naval Research Laboratory (NRL) facility [2] to determine the amount of solar wind (SW) silicon retained in the diamond-like carbon (DLC) collectors. We are also encouraging the Genesis science community to consider the method presented below as a possible alternate solution in various other cases of interest, e.g. higher-mass elements where molecular interferences are a more serious issue, making the SIMS-AMS technique better suited.

The DLC for the Genesis collectors is a thick (1.5-3 μm) layer on a Si substrate and was produced at Sandia National Laboratories by pulsed laser deposition from high density graphite (pellets of hot-pressed carbon powder). It was intended for analysis of nitrogen and noble gas isotopes, but can be also considered for SW Si analysis, which can not be done in Si or Si-containing substrates. The expected mean implantation depth of SW Si in DLC is close to 100 nm with a total expected fluence of $1.9 \cdot 10^{12}$ atoms/cm² from the 2-year flight period. Previous XPS and SIMS data [3] show ubiquitous Si on DLC but only as a surface layer, up to about 30 nm, making it a reasonable candidate for Si analysis. More terrestrial contamination can appear as Si-rich areas in deeper layers, due to annealing steps during fabrication of the thick DLC layer from successive thin layers. Other options for Si analysis would be Ge substrates, mostly destroyed in the landing, or sapphire, which would need Au coating, which in turn might introduce additional contamination.

Experimental Technique: The SIMS-AMS facility at NRL is using a modified Cameca 6F to provide a Cs primary beam. Desired ions, in this case ²⁸Si and ¹³C, are selected through a mass-filtering recombination magnet and injected simultaneously in a 3-MV tandem Pelletron accelerator. After acceleration, only ions in a given charge state are sent to the spectrograph magnet for parallel detection. The ¹³C matrix beam

was monitored in an ETP14150 electron multiplier mounted in a shielding box. For the low-concentration ²⁸Si we had a large-area position-sensitive microchannel plate (MCP) detector, used in transmission mode, and followed by a silicon-implanted energy detector. The combination of two detectors allows for removing possible mass-to-charge ambiguities. The power of background reduction using position/mass and energy coincidence filtering, as well as the ability of the SIMS-AMS system to break apart injected molecules and analyze resulting atomic fragments were described in more detail Ref. [4] and references therein.

Initial Implanted Standard Analysis: Initial results were obtained from a standard consisting of $4 \cdot 10^{13}$ atoms/cm² of 84 keV ²⁸Si implanted in a 1 μm -thick DLC layer on a Si substrate and a blank consisting of unimplanted DLC. The ion-implanted standard and control DLC material were cleaned with a modified RCA process using semiconductor-grade chemicals. The selection mask in the injection magnet had two approximately 1-u wide openings, the terminal voltage was set at 2.4 MV, and charge state was 3+ was selected, yielding 9.6 MeV ions. We verified that there is no background at mass 28 u in the spectrograph by showing that the energy spectrum contains only the 9.6 MeV peak. A depth profile for ²⁸Si is shown in Fig. 1 together with the time dependence of the ¹³C matrix beam.

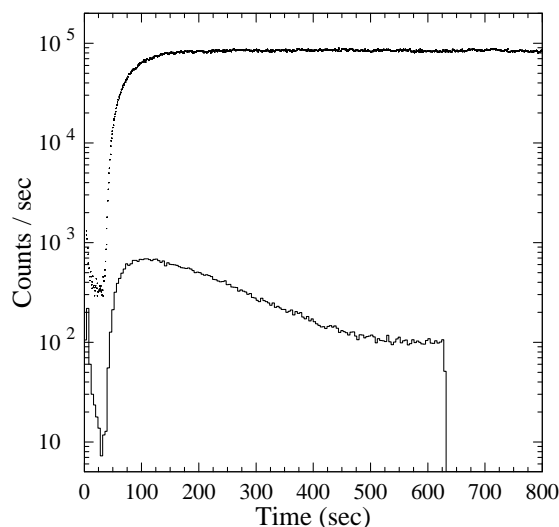


Fig. 1. Measured depth profile of 84 keV ²⁸Si implanted in DLC with $4 \cdot 10^{13}$ atoms/cm² (solid) and time dependence of the matrix ¹³C beam (dotted). Charge state 3+ (9.6 MeV) was analyzed.

The data exemplifies one of the challenges of measuring the Genesis collector materials. The implanted silicon signal begins to appear before the Cs sputtering ion reaches an equilibrium concentration in the DLC, as indicated by a steady yield of ^{13}C from the matrix. Hence, quantification of the Si concentration is more difficult to determine.

Further, while the implanted Si depth profile is expected to be nearly Gaussian, as shown in Fig. 2 from a SRIM 2003 calculation, the measured profile contains a rather long tail into the DLC matrix. While such a tail is expected from recoil mixing of the implanted Si by the Cs sputter beam, the extent observed here is greater than anticipated. Crater wall effects were controlled by use of the Field Aperture to prevent secondary ion collection from outside of the crater bottom, so they are not likely the cause. An effort will be needed to investigate its cause and reduce its duration.

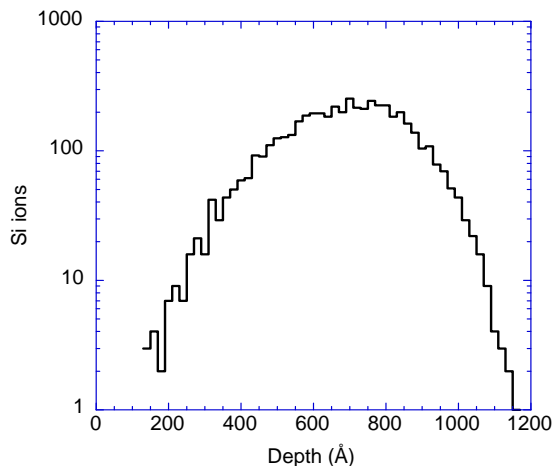


Fig. 2. SRIM 2003 calculation of Si depth profile in DLC from 84 keV ^{28}Si ions, using 5000 ions and a density of 3.0 for DLC.

Another challenge is the observed plateau in Si concentration at the end of the Si depth profile. This too is larger than expected and needs to be reduced to measure solar wind concentrations expected to be at levels 20 times lower than the implanted standard used here. Possible sources include contamination of the sample itself during fabrication or handling, or of the extraction electrode in the SIMS from sputtering prior Si samples, which transfers to the sample during measurement.

Conclusions. We will report preliminary measurements at the LPS 2009 meeting, and the results of our efforts to improve the depth resolution and reduce the background level of Si in the DLC.

Acknowledgements. This work was supported by the NASA Discovery Program as part of the Genesis mission. Special thanks to the MegaSIMS group for

sharing their experience, to Dr. Amy Jurewicz for providing test DLC material, and especially to Dr. Don Burnett for continuous encouragement and useful discussions and information.

References: [1] P.H. Mao et.al. (2008) *Appl. Surf. Sci.*,255, 1461. [2] D.L. Knies et.al. (2006) *Appl. Surf. Sci.*,252,7297. [3] D.S. Burnett (2008) *Private communications*. [4] C. Cetina et.al. (2008) *Appl. Surf. Sci.*,255, 1479.