

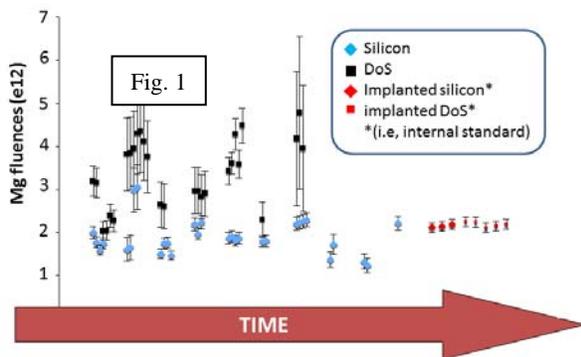
**Genesis DoS Wafers: What Every SIMS Analyst Needs to Know Before Measuring Solar Wind.** A.J. G. Jurowicz<sup>1</sup>, D. S. Burnett<sup>2</sup>, K. D. Rieck<sup>3</sup>, R. Hervig<sup>4</sup>, Y. Guan<sup>2</sup>, and P. Williams<sup>4</sup> <sup>1</sup>Arizona State University (CMS, PO 876004, Tempe AZ 85287; Amy.Jurowicz@asu.edu) for first author, <sup>2</sup>Caltech (GPS, m/c 100-10, Pasadena, CA), <sup>3</sup>LANL (SRS, ISR-2, m/s D-434 Los Alamos, NM), <sup>4</sup>ASU (SMC, Tempe AZ).

Genesis DoS wafers are amorphous, anhydrous, tetrahedrally-coordinated diamond-like carbon (DLC) films on silicon wafers [1, 2]. Using these wafers, excellent analytical results were achieved quickly after the Genesis return for noble gasses using laser ablation [3]. Yet, obtaining precise and accurate analyses by secondary ion mass spectroscopy (SIMS) has been problematic for a decade -- even when the data from each individual analysis looked superb [4]. Here we discuss the problems of SIMS analyses of Genesis DoS and how to mitigate them.

**Experimental:** Multiple SIMS data sets (2006-present) from various DoS wafers are given to show the issues are consistent between sessions and laboratories. Data were taken at either Caltech (Cameca IMF 7f) or ASU (Cameca IMF 6f). An O<sub>2</sub><sup>+</sup> primary beam was used. Other analytical parameters were varied.

**The Problem:** Nicknamed DoS (for Diamond on Silicon), these DLC-coated wafers were chosen for the Genesis mission for their low backscatter during solar wind (SW) collection, relatively low number of potential mass interferences affecting analysis, retentiveness of SW in the DLC film, especially volatiles, as well as physical and chemical durability. Because it is conductive and yield beautifully-shaped SIMS depth-profiles it is extremely tempting to use for SIMS analysis. However, quantification of the results is complicated.

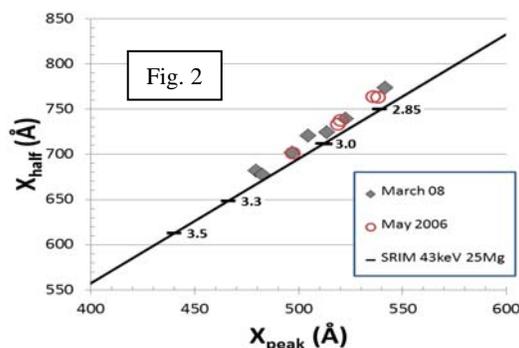
**Fluence Measurements.** Fig. 1 shows 3+ years of attempting to reliably reproduce SW Mg measurements in DoS. The breakthrough was made when a minor



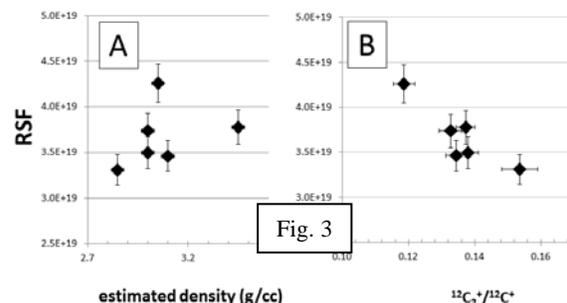
isotope (<sup>25</sup>Mg) was implanted directly into the DLC film below the SW layer and analyzed in the depth profile with the SW <sup>24</sup>Mg. This technique (1) brought SW Mg measurements from silicon and DoS wafers into agreement and (2) mitigated lateral variation in

the relative sensitivity factor (RSF) of Mg ions in the DLC-films [4]. In-house data from DoS with co-implanted H and <sup>25</sup>Mg did not clearly indicate an effect on ion yield so why this implantation technique worked was unconfirmed. In 2015, for Na, *backside* implantation yielded *spatially consistent* results in multiple pieces of DoS, but *the results were offset ~3x from Si*. This may indicate the SW damage has an effect on the RSF, although there are other hypotheses[5].

Calculations for different C-film densities using SRIM [6] were performed *in house* and compared with data from SIMS depth profiles of monoenergetic implants into DoS. Results plotted with parameters for depth profile shape (peak position= $X_{peak}$ ; depth of half peak intensity= $X_{half}$ ) showed that the DLC film density varied laterally (e.g., Fig. 2). However, variations in



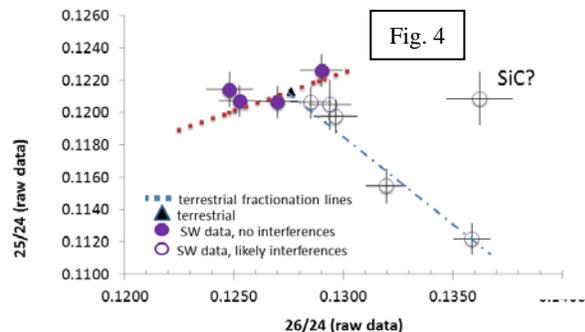
density alone should not change RSF. This is illustrated in Fig. 3, which plots RSF vs. both film density and C<sub>2</sub>/C, the ratio of measured matrix-ion intensities. RSF



vs. density is scattered; RSF vs. C<sub>2</sub>/C is linear. C<sub>2</sub>/C likely reflects film structure (sp<sup>3</sup> vs sp<sup>2</sup>), film electrical conductivity, and (by dilution of carbon bonds) Si content [7]. SIMS and EDS analysis suggests that Si is a ubiquitous, variable component of the DLC. Im-

portantly, one area gave an EDS spectrum having no Si. Si content can affect the value of the RSF. “No” Si means the  $O_2^+$  primary beam generates  $CO$ ,  $CO_2$  gases on impact; the presence of Si means the  $O_2^+$  primary beam generates  $SiO_x$ .  $SiO_x$  is not volatile, allowing O to build up in the sample matrix, thus increasing secondary ion yield [8].

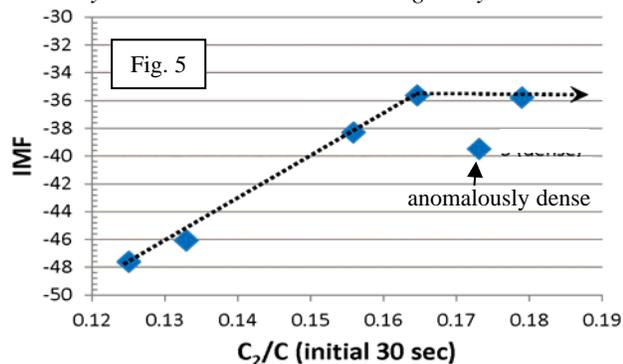
*Isotope measurements.* Inhomogeneity in the composition of the DLC film can also affect isotopic measurements. In Fig. 4, Mg isotope results include unexpected, variable interferences (cf., [7]). Instru-



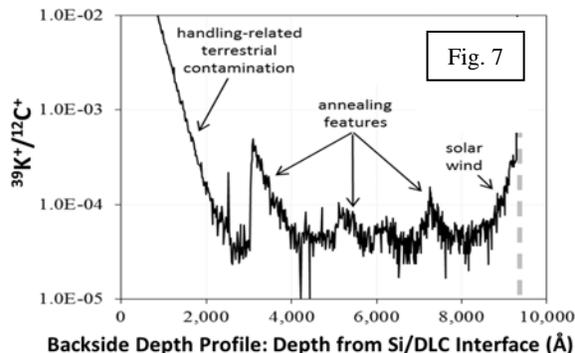
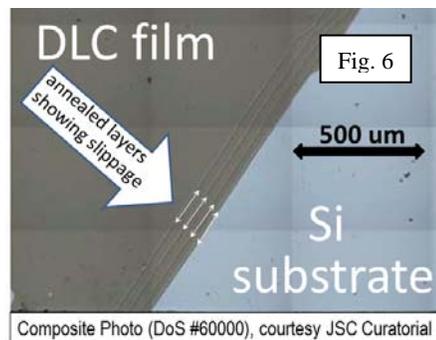
mental mass fractionation (IMF) also varied during this session (Fig. 5), the same data set as Fig. 3.

The initial  $C_2/C$  includes non-steady state (transient) sputtering, but  $^{12}C_2^+$  gives a simple function for IMF variations. Note: in the transient zone,  $^{12}C_2^+$  was effectively constant and  $^{12}C^+$  varied only ~10%.

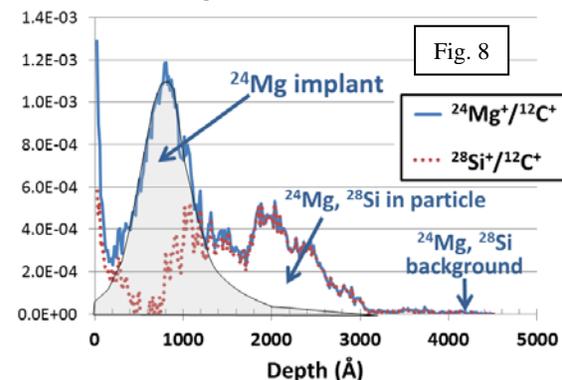
*Physical and Electrical Inhomogeneity.* The DLC



film is manufactured by building-up thin layers of the amorphous tetrahedral carbon (ta-C). Internal stress in each layer can be enormous, sometimes even crystallizing diamond [e.g., 9]. Accordingly, after each layer is deposited, the DoS wafer is annealed to reduce internal stress. This annealing may include a volume change as well as a change in the  $sp^3/sp^2$  ratio, and thus the electrical conductivity [2]. Fig. 6 shows these layers clearly at an uncoated edge of the Genesis  $^{13}C$  DoS Concentrator Target. Although annealed under vacuum, in some analyses, contamination has been observed at these steps (Fig. 7). In other analyses, changes in matrix ion intensity have been observed.



Embedded particulates (dust) have also occasionally been observed (Fig. 8).



**Summary:** DoS wafers are inhomogeneous in density, electrical conductivity and minor element composition. These factors induce matrix effects during SIMS analysis. Issues can be mitigated by (1) parametrizing data using molecular secondary ions, (2) novel standardization techniques, and (3) by SRIM modeling.

**References:** [1] Jurewicz A. J. G. et al. (2003) *Space Sci Rev*, 105, 535–560. [2] Sullivan J. P. et al. (1998) *Mat. Res. Symp. Proc.*, 498, 97-102. [3] Heber V. S. et al. (2009) *Geochim Cosmochim Acta* 73, 7414–7432. [4] Jurewicz A. J. G. et al. (2008) *LPS XXXIX*, Abstract #2272. [5] Rieck K. D. (2015) dissertation. <https://repository.asu.edu/items/36456>. [6] [www.srim.org](http://www.srim.org). [7] Jurewicz A. J. G. et al. (2016) *LPS IXVII*, Abstract #2350. [8] Williams P. (1979) *Surf. Sci.*, 90, 588-634. [9] Logothetidis S. et al. (1998) *Diamond & Rel. Mat.*, 7, 449-453.