

**TOF-SIMS ION IMAGING FOR EVALUATION OF EFFECTIVENESS OF GENESIS SAMPLE CLEANING.** Y. S. Goreva<sup>1</sup> and D. S. Burnett<sup>2</sup>, <sup>1</sup>Dept. of Mineral Sciences, Smithsonian Institution, Washington, DC, [gorevay@si.edu](mailto:gorevay@si.edu), <sup>2</sup>California Institute of Technology

**Introduction:** Surface contamination from the return capsule crash has emerged as the major obstacle to completing Genesis science objectives; consequently, it is essential to use sensitive, but routinely available, methods of surface analysis as a means of evaluation of effectiveness of Genesis sample cleaning. Feasibility of using TOF-SIMS to evaluate Si wafer surfaces has been demonstrated earlier [1, 2]. We are establishing routine TOF-SIMS analysis at the Smithsonian Institution in order to maximize the scientific return from the Genesis mission.

**TOF-SIMS imaging of Genesis silicon wafers.** Analyses were performed in situ at the Smithsonian Institution using a ToF-SIMS IV instrument (ION-TOF GmbH). Ion imaging is done with a 25 keV Bi<sub>3</sub><sup>+</sup> beam at a pulsed current of 0.3 pA, rastered over the area of 500x500 μm for 100 to 300 sec. A focused 3 keV Ar<sup>+</sup> beam, scanned over an area of 500 x 500 μm<sup>2</sup> at a current of 50-100 nA was used for surface sputtering when needed. Blank FZ Si were run along with flight samples.

All analyses were performed with the instrument optimized for high mass resolution (bunched mode: m/Δ m of at least ~5000 at m/z 30). All the data was saved as raw data files which makes it possible to perform retrospective analysis: producing ion images of the specific masses and regions of interest after the data acquisition is completed.

Besides inorganic contamination, all 5 major classes of organics were detected in the surface layer: aromatic hydrocarbons, aliphatic hydrocarbons, phthalates, siloranes and PTFE (Teflon). Some organic compounds are likely residual “brown stain” (a 10-50 Å, polymerized silicone produced during space exposure); others, especially phthalates and siloranes, are most likely products of outgassing of plastic containers.

Ion images in figures 1-5 are produced using a rainbow intensity scale: dark blue to bright red colors correspond to lowest-highest ion yield for each ion. In order to monitor the exact areas before and after cleaning, we keep track of the analysis coordinates along with 2 or 3 fiduciary points, making it easy to locate previously analyzed areas.

Here we present 2 extreme cases of “cleaningness” of flight samples.

**Sample 60286.** This sample underwent multiple cleaning steps and was TRXRF-clean at the time of first TOF-SIMS imaging. Figure 1 is an ion images of the region where previous conventional SIMS analyses were done. Note the very high Cs signal in the SIMS

pits and raster area (optical image) derived from the primary Cs source. Na, Ca, K and Mg particle contamination is evident.

Figure 2 is roughly the same area after additional dilute HCl-HNO<sub>3</sub> cleaning. Additional cleaning was not very efficient, and sample handling may have produced additional contamination. Figure 3 shows the same area after removal of organic films (such as Si(CH<sub>3</sub>)<sub>3</sub>) by a gentle Ar sputtering. Organic films cover the entire surface (Fig 2), but is sputtered away within central square area of Fig 3. As a consequence, Si and other inorganic signals go up in the same area. Particle contamination is not removed by sputtering.

**Sample 60471.** 60471 are a Li-implant flight sample. Compared to 60289 it is exceptionally clean to TOF-SIMS, with areas not showing any particle contamination (Figs 4 and 5). After sputtering off the organic film, Al and <sup>6</sup>Li backgrounds were detected on the surface of the Si wafer. Dark band at the bottom of the images is a boundary of pre-sputtered area.

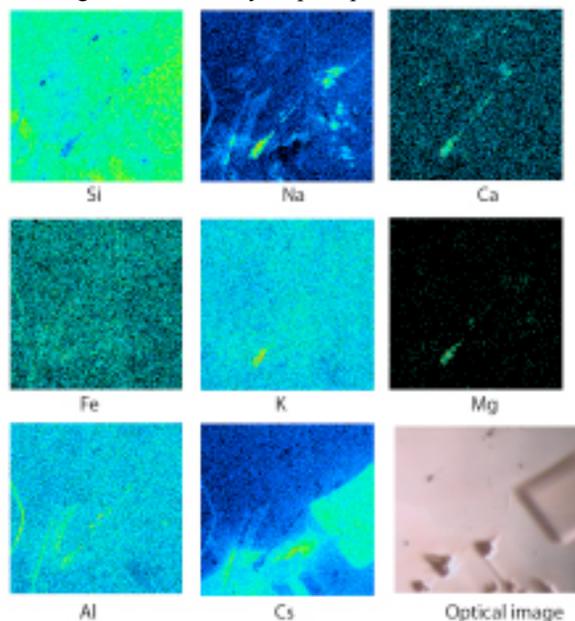


Figure 1. 500x500 μm ion image of 60286 flight sample taken in 2011 after surface sputtering (100 nA pulsed Ar beam for 3 sec). 100 sec analysis time.

**Further work:** Routine pre- and post-cleaning imaging of Genesis samples is underway. In addition to conductive samples, we are planning on using electron flood gun for charge compensation for analysis of sapphire-on-silicon wafers.

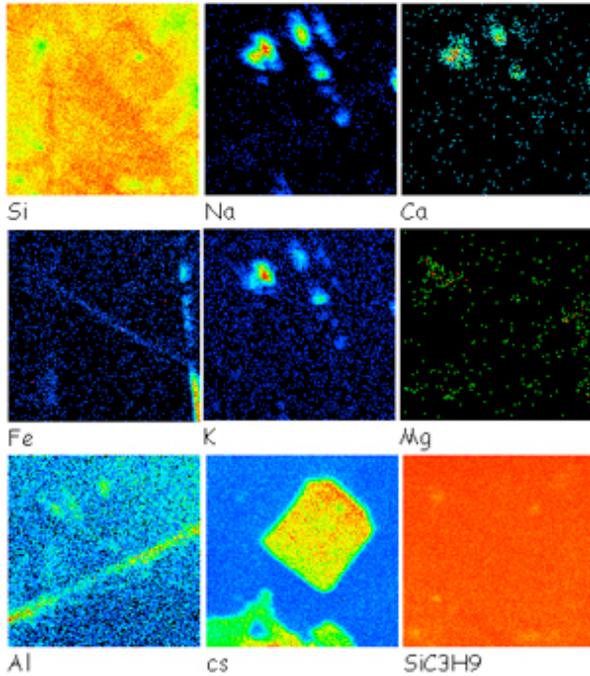


Figure 2. 500x500  $\mu\text{m}$  ion image of roughly the same area of the sample 60289 taken in 2012 after acid cleaning. 300 sec analysis time.

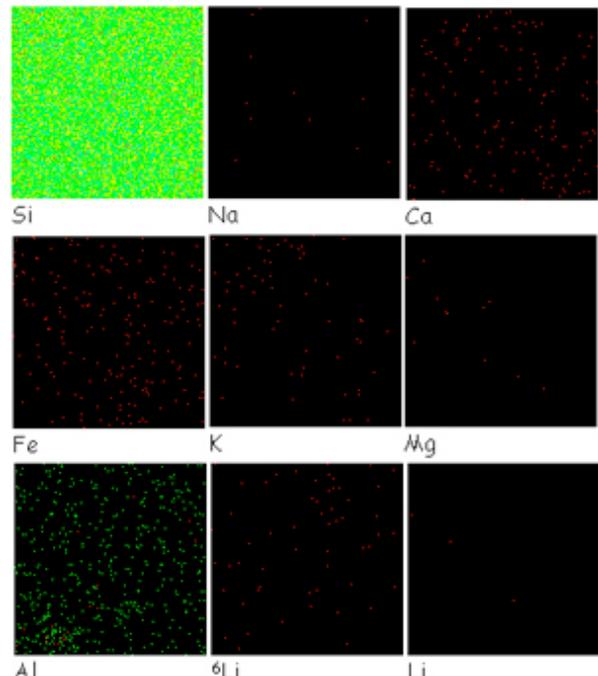


Figure 4. 500x500  $\mu\text{m}$  ion image of Li-implant sample 60471. 300 sec analysis time.

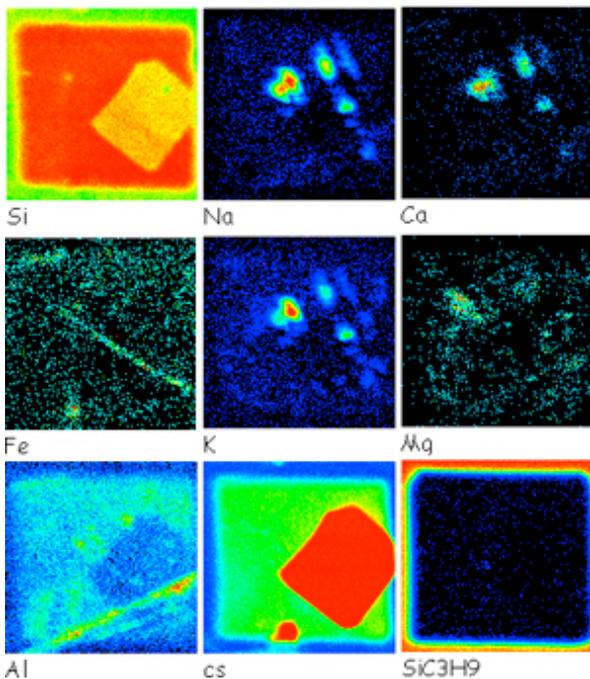


Figure 3. Same area as in figure 2 after surface sputtering (50 nA pulsed Ar beam for 5 sec). 300 sec analysis time.

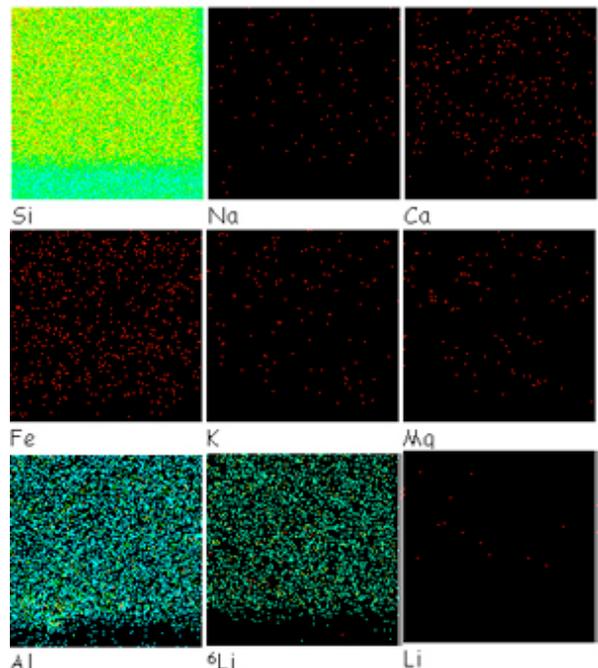


Figure 5. Same area as in figure 2 after surface sputtering (50 nA pulsed Ar beam for 5 sec). The band at the bottom of the image is the boundary of the sputtered area. 300 sec analysis time.

**References:** [1] Kuhlman et al., 2010, *LPSC 41*, #1822  
 [2] Lyon et al. (2011) *LPSC 42*, #2528