

STEP-CLEANING EXPERIMENT ON THE GENESIS WAFERS. S. Huang¹, M. Humayun¹ and S. King¹, B. Goddard¹, ¹National High Magnetic Field Laboratory, Florida State University, 1800 E. Paul Dirac Dr., Tallahassee, FL, 32310 (huang@magnet.fsu.edu).

Introduction: The NASA GENESIS spacecraft mission collected the solar wind by exposing ultraclean wafers outside of the Earth's magnetosphere to the ion flux from the Sun. However, on return to the Earth the sample canister crash-landed into the Utah desert at a speed of about 200 km/h. This accident opened the sample canister. Consequently, it destroyed the ultraclean environment inside the sample canister, and the wafers were seriously contaminated with the Utah desert mud. Since the solar wind particles were implanted in the wafers at a depth of about 100-200 nm, careful cleaning of the wafer surface may remove the surface contamination and allow some of the chemical information of the implanted solar wind tracers to be restored. In this study, we examined the surfaces of GENESIS flight test samples using an ElectroScan E-3 Environmental Scanning Electron Microscope (ESEM) to identify the chemical compositions of particles >5 μm on the wafer surface. We then applied step-cleaning procedures on two wafers, 60045 and 60044, and analyzed the wafer surface with LA-ICP-MS before and after each cleaning steps to investigate the cleaning effects.

Experiments: We applied different step-cleaning procedures on flying Wafer 60045 and 60044. Below are the detailed steps.

Cleaning steps on Wafer 60045: **1. Acetone bath:** Wafer 60045 was put in a beaker with acetone, and the beaker was in ultrasonic bath for 5 minutes; **2. NH₄OH bath:** Then Wafer 60045 was put in a beaker with 10% NH₄OH, and the beaker was in ultrasonic bath for 5 minutes; **3. HNO₃ wash:** Then the surface of Wafer 60045 was attacked by 50% HNO₃, and it was immediately washed by 5% HNO₃. The wafer surface was analyzed by LA-ICP-MS before and after each cleaning steps, and results are summarized in Fig. 1.

Cleaning steps on Wafer 60044: **1. Acetone bath:** Wafer 60044 was put in a beaker with acetone, and the beaker was in ultrasonic bath for 5 minutes; **2. First HNO₃ wash:** Then surface of Wafer 60044 was rinsed with 2ml running 50% HNO₃, and immediately followed by 5% HNO₃ rinse; **3. NH₄OH wash:** Then the surface of Wafer 60044 was rinsed by 2 ml running 50% NH₄OH, and immediately followed by de-ionized water rinse. Then Wafer 60044 was put in a beaker with de-ionized water, and the beaker was in ultrasonic bath for 5 minutes; **4. Second HNO₃ wash:** Then surface of Wafer 60044

was again rinsed with 2ml running 50% HNO₃, and immediately followed by 5% HNO₃ rinse. The wafer surface was analyzed by LA-ICP-MS before and after each cleaning steps, and results are summarized in Fig. 2.

NH₄OH effect on wafer surface: In order to investigate the effect of NH₄OH on the Si wafers, two small pieces of clean control wafer were put in two beakers with 10% NH₄OH. One (**Sample A**) stayed overnight, and the other beaker (**Sample B**) was in ultrasonic bath for 5 minutes. Surface of **Sample B** was then analyzed by LA-ICP-MS.

Analyses: 1. ESEM: Electron microscopy was conducted using an ElectroScan E-3 ESEM at National High Magnetic Field Lab. A focused 20 keV, 120 nA beam was used for imaging. Semi-quantitative chemical analysis was conducted directly on flying wafers without any coating using a Princeton Gamma-Tech energy dispersive X-ray (EDX) and imaging analysis system. **2. LA-ICP-MS:** LA-ICP-MS analysis was conducted on the Finnigan ElementTM ICP-MS with a New WaveTM Nd-YAG 213 nm laser system at National High Magnetic Field Lab. The laser ablation setting is: 100 μm spot size, repetition rate 5Hz, 45% output and a scan rate of 25 $\mu\text{m}/\text{s}$. Elemental analyses were conducted under medium resolution using ²⁴Mg, ²⁷Al, ²⁹Si, ⁴⁴Ca, ⁴⁸Ti, ⁵²Cr, ⁵⁵Mn, ⁵⁶Fe, ⁵⁹Co and ⁵⁸Ni.

Results: 1. ESEM imaging and EDX semi-quantitative chemical analysis: ESEM imaging on the flight wafers shows a high density of particles, 10 μm in diameter and smaller. Larger particles were embedded into the wafers. The Princeton Gamma-Tech EDX analysis system allows semi-quantitative chemical analysis conducted on big (>5 μm in diameter) particles. Majority of the particles analyzed on the flight wafers were fragments of Si and Ge wafers from the collector assemblies. A small number of Zn-Ga spinel crystals (white paint inside the sample canister) were observed. Significantly, we did not identify a single particle, large enough to be analyzed by EDX, which originated from the Utah desert mud. Despite these observations, LA-ICP-MS scanning of the wafer surfaces showed large signals of Mg, Al and Fe, while Ca, Ti, Cr, Mn and Co were mainly below detection limits. **2. Wafer 60045 Step-Cleaning: a.** Acetone bath decreased surface Al, Mg and Fe (not shown in Fig. 1) abundances; **b.** NH₄OH bath decreases the surface Al abundance, but increased surface Mg (and Fe) abundances; **c.** HNO₃

wash decreased surface Al, Mg and Fe abundances to blank level. **3. Wafer 60044 Step-Cleaning:** **a.** Similar to Wafer 60045, acetone bath decreased surface Al, Mg and Fe (not shown in Fig. 2) abundances. Specifically, in this situation the surface Mg and Fe abundances decreased to blank level; **b.** The two HNO₃ wash steps do not have any observable effect; **c.** NH₄OH wash decreases the surface Al abundance; **d.** In this case, the final surface Al abundance is still above blank level, indicating un-removed surface contamination. **4. NH₄OH effect:** The shining surface of **Sample A** was totally destroyed after staying in 10% NH₄OH for overnight; implying that NH₄OH reacts with Si. LA-ICP-MS result shows that the surface of **Sample B** does not show any enrichment of Mg or Fe.

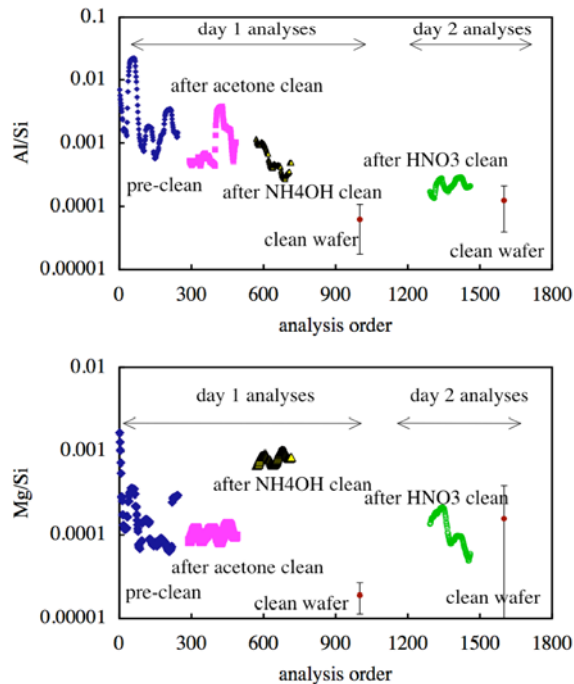


Fig. 1 Step washing effects on Wafer 60045. Pre-clean, after acetone clean and after NH₄OH clean, Wafer was analyzed on the same day, and the average \pm 2-sigma of 6 analyses on a clean control Si wafer is plotted at #1000 for comparison. After HNO₃ clean, wafer was analyzed on a second day, and the average \pm 2-sigma of 11 analyses on a clean control Si wafer is plotted at #1600 for comparison.

Discussion: **1.** Majority of the big ($>5 \mu\text{m}$ in diameter) particles embedded in the wafer surface are fragments of Si and Ge wafers. Utah desert dusts form small particles attached to (or embedded in) the wafer surface. Although the big particles can be

quantitatively removed easily, they may not introduce serious contamination. **2.** Acetone bath physically removes particles from the wafer surface; consequently, it decreases surface Al, Mg and Fe abundances. **3.** Since NH₄OH reacts with Si, it etches into the wafer beneath the imbedded particles. Consequently, it looses and removes the imbedded particles. **4.** Depending on the degree of the reaction between NH₄OH and the wafer, it removes some surface contamination (Wafer 60044) to all surface contamination (with the help of HNO₃, Wafer 60045). However, in the later situation, it is possible that it also removes the solar wind tracer layer. **5.** HNO₃ alone does not have any observable effect reducing contamination. In the case of Wafer 60045, it removes the atomic Mg and Fe layer caused by the bulk NH₄OH bath.

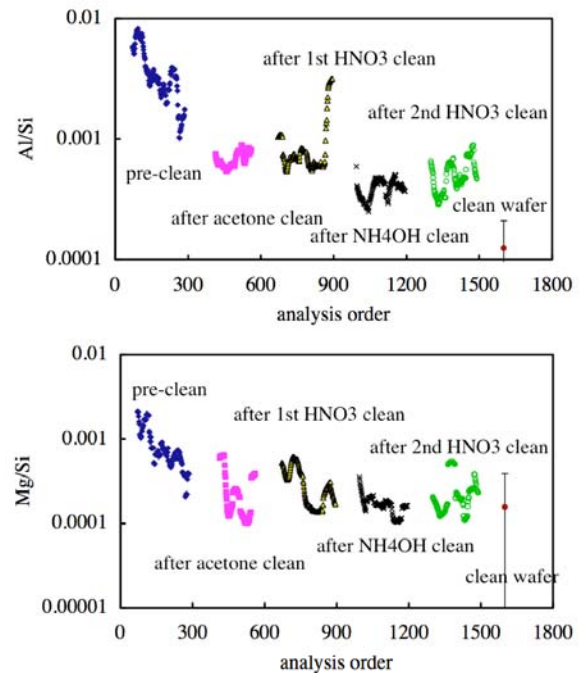


Fig. 2 Step washing effects on Wafer 60044. The average \pm 2-sigma of 11 analyses on a clean control Si wafer is plotted at #1600 for comparison.