Step-Cleaning Experiment on the GENESIS Wafers. S. Huang[1], M. Humayun[2] and S. King[3], B. Goddard[4], D. Burnett[5], [1]National High Magnetic Field Laboratory, Florida State University, 1800 E. Paul Dirac Dr., Tallahassee, FL, 32310, huang@magnet.fsu.edu, [2]humayun@magnet.fsu.edu, [3]sking@magnet.fsu.edu, [4]goddard@magnet.fsu.edu, [5]Division of Geological and Planetary Sciences, California Institute of Technology, burnett@gps.caltech.edu.

Introduction: The NASA GENESIS spacecraft mission collected the solar wind tracers by exposing ultraclean wafers outside of the Earth's magnetsphere to the ion flux from the Sun. The solar wind tracer collection lasted for 884 days in the space. However, on return to the Earth, due to mechanical problems, 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 dusts. Since the solar wind tracers were implanted in the wafers at a depth of about 100 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 research, we examined the flying wafer surface using an Electrosacn E-3 Environmental Scanning Electron Microscope (ESEM) to identify the chemical compositions of the big particles on the wafer surface. Also we applied step-cleaning procedures on two flying 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. NH_4OH bath**: Then Wafer 60045 was put in a beaker with 10% NH_4OH, and the beaker was in ultrasonic bath for 5 minutes; **3. HNO_3 wash**: Then the surface of Wafer 60045 was attacked by 50% HNO_3, and it was immediately washed by 5% HNO_3. 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 HNO_3 wash**: Then surface of Wafer 60044 was rinsed with 2ml running 50% HNO_3, and immediately followed by 5% HNO_3 rinse; **3. NH_4OH wash**: Then the surface of Wafer 60044 was rinsed by 2 ml running 50% NH_4OH, 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 HNO_3 wash**: Then surface of Wafer 60044 was again rinsed with 2ml running 50% HNO_3, and immediately followed by 5% HNO_3 rinse. The wafer surface was analyzed by LA-ICP-MS before and after each cleaning steps, and results are summarized in Fig. 2.

NH_4OH effect on Wafer Surface: In order to investigate the effect of NH_4OH on the Si wafers, two small pieces of clean control wafer were put in two beakers with 10% NH_4OH. 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 Electrosacn 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 Element™ ICP-MS with a New Wave™ Nd-YAG 123 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 μm/s. Elemental analyses were conducted under medium resolution using ^{24}Mg, ^{27}Al, ^{29}Si and ^{56}Fe.

Results: 1. ESEM imaging and EDX semi-quantitative chemical analysis: ESEM imaging on the flying wafers shows a high density of particles, 10 μm in diameter and smaller. Larger particles are 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 flying wafers are 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 dusts. **2. Wafer 60045 Step-Cleaning: a.** Acetone bath decreased surface Al, Mg and Fe (not

shown in Fig. 1) abundances; **b.** NH_4OH bath decreases the surface Al abundance, but increases surface Mg (and Fe) abundances; **c.** HNO_3 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 lever; **b.** The two HNO_3 wash steps do not have any observable effect; **c.** NH_4OH 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. NH_4OH effect:** The shining surface of **Sample A** was totally destroyed after staying in 10% NH_4OH for overnight; implying that NH_4OH reacts with Si. LA-ICP-MS result shows that the surface of **Sample B** does not show any enrichment of Mg or Fe.

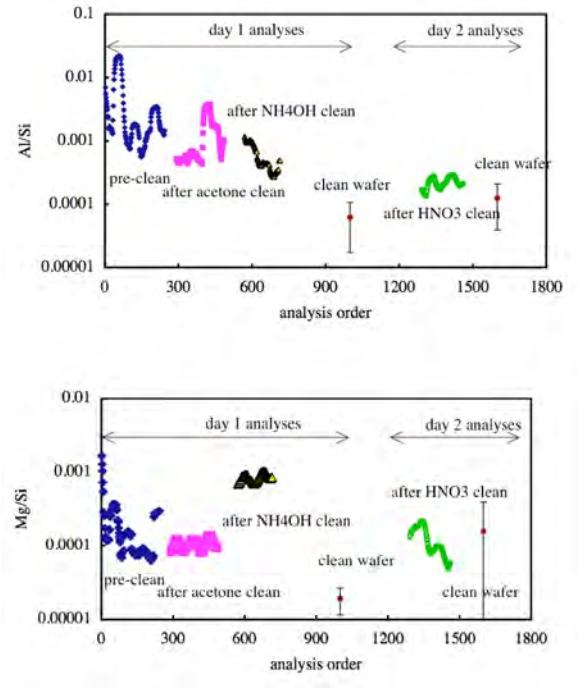

Fig. 1 Step washing effects on Wafer 60045. Pre-clean, after acetone clean and after NH_4OH clean were analyzed on the same day, and the average of 6 analyses, with +3 sigma, on a clean control Si wafer is plotted at 1000 s for comparison. After HNO_3 clean was analyzed on a second day, and the average of 11 analyses, with +3 sigma, on a clean control Si wafer is plotted at 1600 s for comparison.

Discussion: 1. Majority of the big (>5 μm in diameter) particles embedded on 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 NH_4OH reacts with Si, it etches into the wafer beneath the imbedded particles. Consequently, it looses and removes the imbedded particles. **4.** The surface enrichment of Mg and Fe on Wafer 60045 may be caused by the following steps. **a.** NH_4OH reacted with the particles on the surface and the back of the Wafer 60045, and formed a solution with $Fe^{2/3+}$ and Mg^{2+}. **b.** Atomic Si on the surface of Wafer 60045 replaces $Fe^{2/3+}$ and Mg^{2+} in the solution, and atomic Mg and Fe were deposited on the surface of Wafer 60045. **5.** Depending on the degree of the reaction between NH_4OH and the wafer, it removes some surface contamination (Wafer 60044) to all surface contamination (with the help of HNO_3, Wafer 60045). However, in the later situation, it is possible that it also removes the solar wind tracer layer. **6.** HNO_3 alone does not have any observable effect. In the case of Wafer 60045, it removes the atomic Mg and Fe layer caused by the bulk NH_4OH bath.

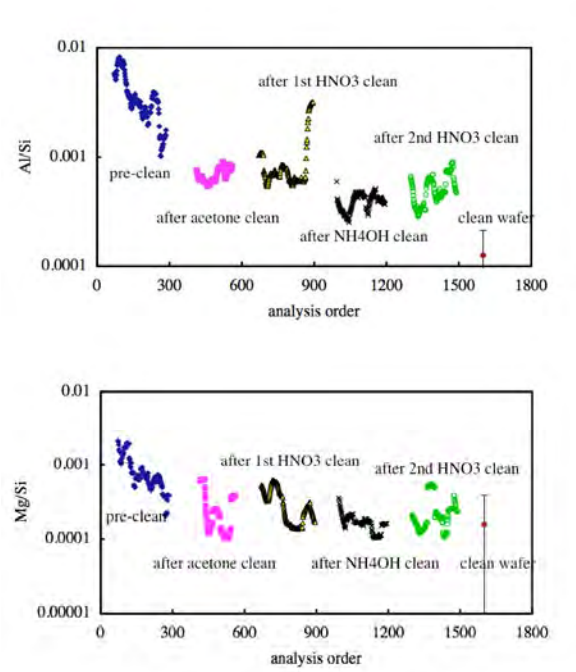

Fig. 2 Step washing effects on Wafer 60044. The average of 11 analyses, with +3 sigma, on a clean control Si wafer is plotted at 1600 s for comparison.