**DEVELOPMENT OF CHEMICAL AND MECHANICAL CLEANING PROCEDURES FOR GENESIS SOLAR WIND SAMPLES.**

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**Introduction:** The Genesis mission was the only mission returning pristine solar material to Earth since the Apollo program [1, 2]. Unfortunately, the return of the spacecraft on September 8, 2004 resulted in a crash landing shattering the solar wind collectors into smaller fragments and exposing them to desert soil and other debris. Thorough surface cleaning is required for almost all fragments to allow for subsequent analysis of solar wind material embedded within. However, each collector fragment calls for an individual cleaning approach, as contamination not only varies by collector material but also by sample itself. In some cases, common cleaning methods employed in the semiconductor industry can be applied, but more often cleaning has to be specifically tailored for an individual sample. One major objective is to develop standardized cleaning procedures for each collector material, which can be applied on a routine basis, and will yield sufficiently clean samples for most solar wind analyses. If necessary, those procedures can be extended to a more aggressive treatment to remove specific contaminants. Cleaning should be evaluated on non-flight control samples of the same material before being used on actual flight samples. However, a direct transfer of a cleaning method from control to flown sample is often not possible as flown samples not only show different types of contamination, but also have experienced radiation damage during solar wind collection and physical damage from the crash.

Before and after a cleaning step the sample is inspected optically with a microscope and chemically by total reflection X-ray fluorescence (TXRF) spectrometry to investigate the effectiveness of the cleaning and its impact on the sample surface. [3]. The suitability of TXRF has been demonstrated for several Genesis solar wind samples before and after various cleaning methods including ultrapure water jet, acid treatment, gas cluster ion beam, and $CO_2$ snow jet [4 – 8].

The data presented in this work focus on the cleaning of synthetic single-crystal sapphire ($Al_2O_3$) collector fragments. Sapphire is one of the hardest and most chemically resistant materials known. These properties indicate that application of harsh chemical and/or mechanical treatments should not introduce any contaminants or result in surface damage.

**Experimental:** Two Genesis flight samples (30580 and 60644) and three flight controls from the same batch (3SAP00889) were selected for cleaning. Figure 1 shows micrographs of each flight sample and one micrograph of a control. Flight sample 30580 was initially cleaned using an ultrapure water (UPW) jet (18.2MΩ) at 3000RPM for 5 min at 40°C [4]. After that a second cleaning consisting of 1 part $H_2O$, 1 part concentrated HCl and 1 part concentrated $HNO_3$ was applied. Flight sample 60644 and one of the control samples underwent a 0.05µm $Al_2O_3$ powder rub followed by the RCA-1 procedure [9]. RCA-1 uses a mixture of 5 parts water, 1 part 29% $NH_4OH$ and 1 part 30% $H_2O_2$ for 10 minutes at 75°C in an ultrasonic bath followed by an ultrapure water rinse and air dry. Two additional control samples were tested for the effectiveness of the UPW and $NH_4OH$ treatments.

All samples were analyzed by TXRF (PicoFox, Bruker AXS, Madison, WI) for surface contaminations after treatment and if possible before treatment. However, when a sample had to be available for solar wind analysis on short notice the initial analysis was skipped.

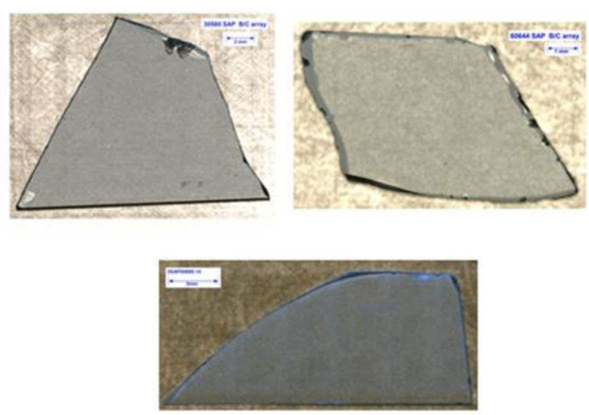

Figure 1: Micrographs of flight samples 30580 (top left, scale bar 2mm), flight sample 60644 (top right, scale bar 1mm) and control sample 3SAP00889,14 (bottom, scale bar 2mm). The material is synthetic single-crystal sapphire ($Al_2O_3$) for all samples.

**Results and Conclusion:** Figures 2 to 4 show the TXRF analysis results for the different cleaning procedures. Both flight samples have significantly more contamination than the controls. This is not surprising considering that those samples are shattered material from the original collectors, which were exposed to debris from the landing site. A closer inspection of the micrographs in figure 1 indicates that both samples have scratches on the surface. In case of sample 30580 (figure 2), a cleaning step involving concentrated HCl and $HNO_3$ was not able to remove Cr, Mn, and Fe. Moreover, it appears that Br was introduced as an artifact after the acid treatment.

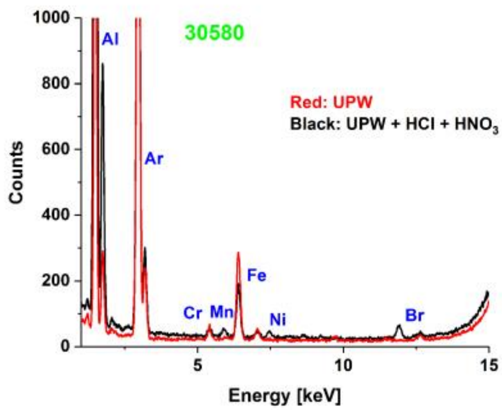


Figure 2: TXRF spectra of flight sample 30580 after ultrapure water (UPW) jet cleaning (red) and after additional treatment with concentrated hydrochloric and nitric acids.

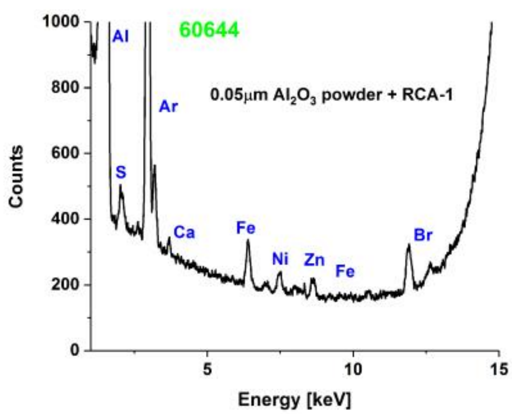


Figure 3: TXRF spectrum on flight sample 60644 after application of 0.05µm $Al_2O_3$ powder rub and subsequent RCA-1 cleaning.

Sample 60644 (figure 3) was analyzed after application of a 0.05µm $Al_2O_3$ powder rub and RCA-1 treatment. The data show not only a number of contaminants including Br, but the elevated background of the spectrum also indicates an increased surface roughness. An increased surface roughness and Br as contaminant was also found for the control sample, which underwent a similar treatment as the green spectrum in figure 4 attests. A 0.05µm $Al_2O_3$ powder rub should not damage the surface of the single-crystal synthetic sapphire thus the observed surface roughness is likely caused by remnants of the alumina powder still adhering to the sample surface. Additional cleaning tests to support this observation are underway. The spectra for the other control samples show far less contaminants. Both UPW and $NH_4OH$ did not introduce any elements or impact surface roughness (red and blue spectra in figure 4). In fact, it appears that $NH_4OH$ did remove some of the Fe that was present in the non-cleaned control sample (black spectrum in figure 4).

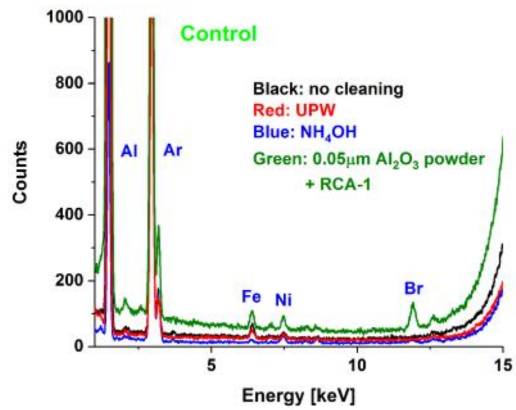


Figure 4: TXRF data for different cleaning procedures applied to flight control or spare samples (3SAP00889) made of synthetic single-crystal sapphire. Black: no cleaning; red: ultrapure water (UPW) jet cleaning; blue: ammonium hydroxide cleaning; green: 0.05µm $Al_2O_3$ powder rub with subsequent RCA-1 cleaning.

**References:**

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