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Cellulose Acetate Replica Cleaning Study of Genesis Non-Flight Sample 3CZ00327

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Introduction: The Genesis mission collected solar wind and brought it back to Earth in order to provide precise knowledge of solar isotopic and elemental compositions. The ions in the solar wind were stopped in the collectors at depths on the order of 10 to a few hundred nanometers. This shallow implantation layer is critical for scientific analysis of the composition of the solar wind and must be preserved throughout sample handling, cleaning, processing, distribution, preparation and analysis.

We are working interactively with the community of scientists analyzing Genesis samples, using our unique laboratory facilities -- and, where needed, our unique cleaning techniques -- to significantly enhance the science return from the Genesis mission. This work is motivated by the need to understand the submicron contamination on the collectors in the Genesis payload as recovered from the crash site in the Utah desert, and -- perhaps more importantly -- how to remove it. That is, we are evaluating the effectiveness of the wet-chemical “cleaning” steps used by various investigators, to enable them to design improved methods of stripping terrestrial contamination from surfaces while still leaving the solar-wind signal intact.

Replica cleaning of SOS sample 60966: Genesis flight sample 60966 is a piece of silicon on sapphire (SOS), which the Genesis team wants to analyze using laboratory total reflection X-ray fluorescence (TRXRF). This technique is extremely sensitive to surface contamination. However, since the substrate is an insulator, SEM analysis proved to be extremely difficult. Therefore, Prof. Burnett requested cleaning of the sample using the cellulose acetate extraction replica technique shown in Figure 1 [1]. The SOS did not wet and adhere to the replica film the same way that a sample of pure silicon does. Thus, four replicas were made, with the last leaving the sample looking visibly clean. Sample 60966 was then returned to Prof. Burnett for xylene cleaning and passed on to Prof. Schmeling for analysis using TRXRF.

This analysis demonstrated that the surface of sample 60966 was roughened more than it was during a previous analysis. Prof. Burnett requested that we examine the extraction replicas. We coated the replicas with 60 nm of carbon using a sputter coater and analyzed them under the SEM before moving on to removal of the replica film. Replicas 1 and 3 appeared to be upside down. It is very easy to mix up top and bottom without a distinct duplicate of the sample pressed into the acetate film. Replica 2 appeared to have very few particles in it, one with a sodium and aluminum signal and one with only silicon. Replica 4 appeared to be much more robust with many particles in it as expected based upon the wetting of the sample. Of 25 particles analyzed in the film, 4 contained only iron, 11 were predominately carbon, 4 were AlO, 3 contained Mg with Al, Si and O, and the remaining particles were either Si or SiO.

Investigation of possible residues left by replica cleaning: Due to our results on sample 60966, we investigated the possible surface contamination left by cellulose acetate replica cleaning using a pair of control samples cut from non-flight silicon sample 3CZ00327. Subsample 3CZ00327,81 was UPW cleaned at JSC using normal protocols [2] after which a single cellulose acetate replica cleaning was performed. The sample was then sent to Prof. Burnett for a hot xylene and acetone treatment and returned to JSC optical imaging before and after a final UPW cleaning. The cellulose acetate film appears to have left some optically visible residue on the sample surface, which was removed by the subsequent hot xylene and acetone treatment. The sample was then sent to Prof. Schmeling for TRXRF analysis. Subsample 3CZ00327,59 was UPW cleaned, but not treated in any other way. The samples were analyzed using TRXRF (Figure 2) and because the background of the spectra look similar, Prof. Schmeling determined that no organic residue remained from the replica cleaning, although sample 3CZ00327,81 showed signs of iron contamination and 3CZ00327,59 showed signs of nickel contamination.

Figure 1a). A thin film of cellulose acetate is wetted with acetone and applied to the sample. Once dry, the film hardens and is easily removed from the substrate. 4b) the film is easily removed from the substrate, taking most of the particles with it. 4c) shows the freestanding cellulose film evaporated with 60 nm of carbon. The acetate film can then be evaporated using acetone vapor to dissolve the cellulose acetate film, leaving behind a carbon film suspended on a copper TEM grid containing the particles removed from the Genesis sample.

Figure 2. TRXRF analyses of Genesis non-flight samples 3CZ00327,81 and 3CZ00327,59 following cellulose acetate replica cleaning of 3CZ00327,81. Sample 3CZ00327,59 was only UPW cleaned. Spectra courtesy of Prof. Schmeling.