UPDATED STATUS OF THE GENESIS MO-PT FOILS FOR SOLAR WIND RADIONUCLIDE ANALYSIS.

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Introduction: Long-lived radionuclides, such as ^{10}Be, ^{26}Al, and ^{53}Mn, are produced on the surface of the Sun by nuclear interactions between solar energetic particles and the solar atmosphere and then entrained into the solar wind (SW). The expected fluxes of these nuclides in the SW at near-Earth orbit are <200 atom/cm^2/yr, but have large uncertainties. One of the objectives of the Genesis mission was to capture measurable quantities of these radionuclides in large foils deployed in the lid of the sample return capsule (SRC). The Genesis mission exposed ~8000 cm^2 of Mo-coated Pt foils to the Sun for 884 days. The collector foils consist of a Mo coating (~300 nm thick) on a Pt substrate (~48 µm thick) [1]. We originally planned to (1) remove loosely attached terrestrial dust without damaging the Mo coating; (2) identify and remove micrometeorite (MM) impacts, leaving <1 µg of residual MM contamination on the entire collector; (3) dissolve the Mo and separate all SW radionuclides from the Mo; and (4) measure the very low concentrations of SW radionuclides by accelerator mass spectrometry (AMS).

Challenges. Upon return to Earth, the sample return capsule (SRC) made an unexpected hard landing in the Utah desert, crushing the foils and contaminating them with Utah dirt and with pieces of spacecraft debris and collector materials [2]. The hard landing of the SRC in the Utah desert has presented us several difficult challenges: (1) stretching the Mo-Pt foils to near-flat condition; (2) mapping contamination by SEM; (3) removing large quantities of Utah dirt/salt (mainly NaCl and $CaCO_3$) as well as various types of spacecraft debris (paint, collector materials); and (4) verifying the cleanliness of the foils before dissolving the Mo coating. Only after these four steps are completed can we proceed with steps 2-4 of the original plan. Two unexpected complications are that (1) the crumpled foils are much stiffer than the non-flight foils and (2) the surface of the flight Mo-Pt foil is far more reactive than the non-flight foil due to oxidation of the Mo surface. The Mo surface is not as "inert" as we had anticipated, so we have to avoid aqueous solutions, making the chemical removal of contamination much more difficult.

Model calculations indicate that 1.5 % of Be, 2.5 % of Al and Mn are stopped in the top 3 nm of the Mo layer. The first requirement of the cleaning process is to ensure that >97-99% of the implanted SW radionuclide inventory is retained in the Mo layer, so no more than 2-3 nm of the Mo surface layer can be lost. Analysis of the Utah soil shows that it contains ~4x10^5 atoms of ^{10}Be/mg of dirt. Therefore, a second requirement of the cleaning process is that less than 100 ng of dirt/cm^2 remains. Below we discuss the progress on several of the challenges described above since our last report [2].

1. Stretching the foils: To image the contamination on the crumpled Mo-Pt foils by SEM before cleaning them, we had to straighten out the foils. We first cut the foils into pieces of up to 20 x 20 cm size (expanded size), then applied the "guitar tuning" method to each Mo-Pt foil in a clean chamber. This technique has allowed us to slowly stretch all of the crumpled Mo-Pt foils to their original size (or close to it) with minimal damage to the Mo surface.

2. Pre-Cleaning SEM Analysis: To scan the foils, we use a Tescan Vega SEM, equipped with a large chamber and stage that can hold a 20 x 20 cm piece of foil. Stretched foil pieces are secured on the SEM stage and are scanned in four subsections of 10 x 10 cm, obtaining backscattered electron (BSE) images with a resolution of ~4 µm. These BSE images were then used to identify surface contamination (Utah dirt/spacecraft materials), delamination of the Mo layer (revealing the underlying Pt), and locate MM impact craters.

3. SEM image analysis: To quantify the distribution of contamination on the foils and delamination (flaking) of the Mo layer, we developed a Python script that was used to automate the SEM image analysis.

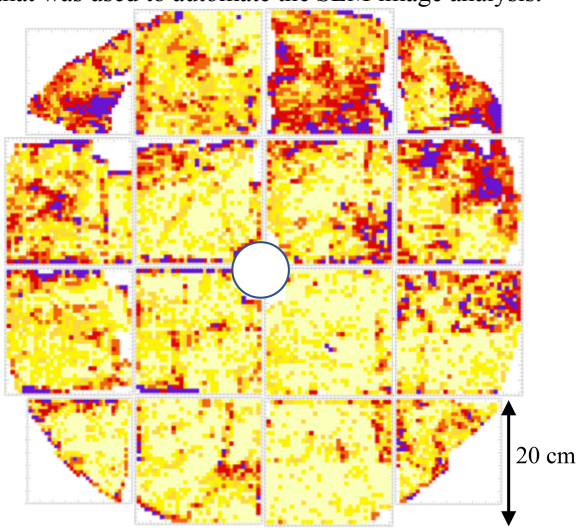

Fig. 1. Distribution map of low-Z contamination (dirt, paint or other spacecraft materials) on ~4500 cm^2 of circular foil, 50053, before cleaning. Contamination levels range from <1 µg/cm^2 (light yellow) to >100 µg/cm^2 (purple).

The average amount of Mo flaking (before cleaning) is ~1% of the surface area. To calculate the amount of contamination, we assumed an average thickness of 10 μm and a density of 2 g/cm^3. The average contamination level is ~15 μg/cm^2 for the circular foil 50053, a factor of ~150 above our upper limit of 0.1 μg/cm^2. However, the contamination is not uniform, but varies by more than 3 orders of magnitude across the foil, indicating the contamination on the foil is highly localized (Fig. 1). The least contaminated foil sections contain only 2-3 μg of dirt per cm^2; removing ~10% of the (most dirty) subsections of these foils would further reduce the contamination level to ~0.6 μg/cm^2 *before cleaning*. This implies that a cleaning technique that removes ~90% of the Utah dirt would allow SW radionuclide analyses on significant parts of the Mo-Pt foil, which is encouraging.

4. Hydrogenation method: To test various cleaning methods we used Mo-coated stainless steel (Mo-SS) foils, flight spare control (non-flight) Mo-Pt foils that were kept at JSC during the Genesis mission, and small test pieces of flight Mo-Pt foils. Initial cleaning tests showed that the Mo layer was much more reactive than anticipated, showing significant amounts of Mo in the cleaning solutions analyzed by ICP-OES.

To alleviate the problem of the reactive Mo oxide surface we developed a hydrogenation method to convert the MoO_3 layer to a less oxidized form of Mo. Effective hydrogenation is achieved during 3-4 weeks in a Parr vessel with a H_2 pressure of 10-30 MPa, at 140˚C. FIB-TEM analysis of the H_2-treated foil shows that the H_2 treatment significantly reduces the O-concentration in the Mo layer. The effectiveness of the hydrogenaton method is also confirmed by cleaning tests which show much lower Mo losses of the hydrogenated test foils compared to the untreated foils.

5. AMS analysis. Recent upgrades of the AMS facility at PRIME Lab have lowered the detection limits of ^{10}Be and ^{26}Al measurements by a factor of 2-10, respectively. This higher sensitivity implies that we don't necessarily have to dissolve the entire foil to perform succesful SW radionuclide measurements, but can dissolve ~1000 cm^2 as long as the SW fluence is within a factor of ~2 of the expected values.

6. Cleaning method: The two requirements that more than 90-99% of the dirt is removed, while less than 2-3 nm of the Mo surface is lost, imply that a delicate balance needs to be found between effective dirt removal and minimum damage to the Mo layer. We have experimented with more than 70 reagents and several hundred different mixtures involving more than 4,000 different chemical test experiments. We use 0.5-1.0 cm^2 of control non-flight Mo-Pt and Mo-SS foil for each solvent test as well as flight Mo-Pt foil. To simulate the contaminated flight foils, we spray an aqueous salt solution (extracted from Utah dirt) on each non-flight foil, typically yielding 10-100 μg of salt/cm^2. Each test foil is soaked in a cleaning solution for ~60 min. and agitated in a megasonic bath (1200W, 1MHz) for 3-15 min. We then analyze the solution by ICP-OES to determine the amount of Mo loss and determine the efficiency of dirt removal by mass loss of the foils using a microbalance. We found the best cleaning results with mixtures of methanol and weak organic acids, such as glacial acetic acid or formic acid. Methanol alone dissolves the salt component of the Utah dirt, while the organic acids dissolve the carbonates. Application of this cleaning method on a ~90 cm^2 Mo-SS foil confirms the results obtained on small test pieces.

7. Identifying MM impacts. Before launch, we expected that (1) ~150 μg of MM would impact the entire foil surface during the 2-year exposure in space, and (2) a small but significant fraction of this MM material would survive as contamination in or around small impact craters on the foil. Since MM's contain high levels (10^4-10^6 atoms/μg) of ^{10}Be, ^{26}Al, and ^{53}Mn, we have to verify that <1 μg of MM material remains on the foils before dissolving the Mo layer. Although the SEM-BSE images of ~2,500 cm^2 of foil show hundreds of circular features, high-resolution SEM images of these features show that only 8 of them appear to be hypervelocity impacts (Fig. 2). Subsequent SEM-EDS analysis show no detectable amounts (<10 pg) of MM material (Fe,Mg-silicates or Fe,Ni-metal) on the foil in or around any of these craters, so MM contamination is negligible, which is the single most positive result of this project.

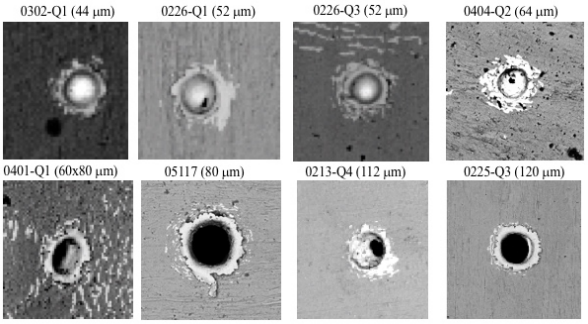

Fig. 2. BSE images of 8 circular (or near-circular) features of 44-120 μm diameter on the Mo-Pt foil that appear to represent hypervelocity impact craters.

Conclusions. We have succesfully developed and tested new techniques to clean the Mo-Pt foils, after stretching them and analyzing surface contamination by SEM. In the next few years we will apply these techniques to several large portions (1000-2000 cm^2) of the Mo-Pt foils to analyze SW radionuclides.

Acknowledgments. This work was supported by NASA's LARS program. **References:** [1] Jurewicz A. J. G. et al. (2003) *Space Sci. Rev*., 105, 535-560. [2] Nishiizumi K. et al. (2005) *LPS* XXXVI, Abstract #2266.