Analysis of Molecular Contamination on Genesis Collectors through Spectroscopic Ellipsometry. K.M. McNamara[1], Eileen K. Stansbery[1], NASA, Johnson Space Center, Houston, TX 77058; karen.m.mcnamara@nasa.gov;[1]

Introduction: Before the spacecraft returned to Earth in September, the Genesis mission had a preliminary assessment plan in place for the purpose of providing information on the condition and availability of collector materials to the science community as a basis for allocation requests. One important component of that plan was the evaluation of collector surfaces for molecular contamination. Sources of molecular contamination might be the on-orbit outgassing of spacecraft and science canister components, the condensation of thruster by-products during spacecraft maneuvers, or the condensation of volatile species associated with reentry. Although the non-nominal return of the Genesis spacecraft introduced particulate contamination to the collectors, such as dust and heatshield carbon-carbon, it is unlikely to have caused any molecular deposition. The contingency team's quick action in returning the damaged payload the UTTR cleanroom by 6 PM the evening of recovery help to ensure that exposure to weather conditions and the environment were kept to a minimum.

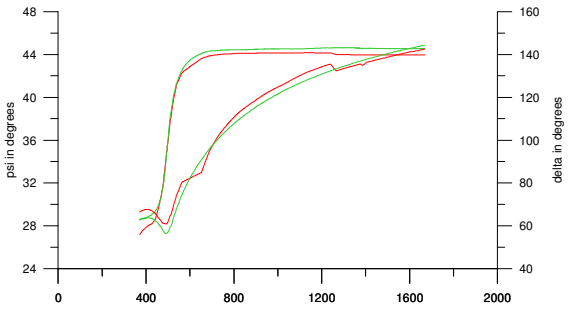
Evidence of Molecular Contamination: Inspection of the interior of the Genesis science canister in Utah, and subsequently at JSC, revealed a darkening on the aluminum canister shield and other canister components. Though there is no direct observation of film contamination on the collector surfaces, this might be difficult to observe with the naked eye and there is reason for concern regarding molecular contamination. To assess molecular contamination, collectors were examined using spectroscopic ellipsometry. The extension of the wavelength data to the near IR, becomes more critical as the identity of the molecular material may need to be determined.

Measuring contaminant film thickness: Spectroscopic ellipsometry measures the change in phase and ellipticity of reflected polarized light as a function of incident angle and wavelength to determine film thickness, index of refraction and extinction coefficients of contaminant layers on smooth surfaces. It is widely used in the semiconductor industry to measure the thickness of SiO_2 layers, polymer photoresist coatings, dielectrics, nitrides…etc. and is well suited to the Genesis collectors. The results of the analysis are an extremely sensitive thickness and estimated optical properties of contaminant layers. For ellipsometry to be accurate, however, it is important to have a good understanding of the substrate materials. Fortunately, we have samples of all of the Genesis collector materials from the same fabrication and processing batches as those used on flight. For comparison with the flight samples, we measured pristine materials that have been stored in the Genesis Laboratory at JSC since the time of payload assembly.

Selection of Samples: The first flight samples analyzed by spectroscopic ellipsometry were an intact silicon on sapphire (SiOS) from position X on the B array and a gold on sapphire (AuOS) half hexagon from position Y on the same array. The B array was selected because it is a bulk solar wind collector and exposed at all times during science collection. The two specific samples were chosen to represent positions on opposite sides of the array for spatial variation. In addition, silicon surfaces represent the largest total area of collectors and sapphire based collectors have a statistically higher survival rate.

Standards: The SiOS wafers on Genesis consist of an epitaxially grown layer of Si on single crystal sapphire. These are well understood and well characterized materials. Native and thermal oxides on silicon have also been studied extensively for many years [2], and the literature contains highly refined models for the optical constants of these materials. These models were tested on a flight qualified SiOS wafer from the Genesis archives, providing an excellent fit and baseline model for our system as: an Al_2O_3 substrate coated with 1850-1950 A of single crystal Si and a native oxide (SiO_2) of 17 A. This is typical of room temperature oxidation in air.[3]

Collectors such as aluminum on sapphire (AlOS) and AuOS, however were physically deposited and are not single crystal or in registry with the substrate. This makes them more difficult to model because of variation in optical properties with grain size and density due to differences in processing. Thus, for these materials, the flight spares become critical for interpreting measurements on the returned collectors.



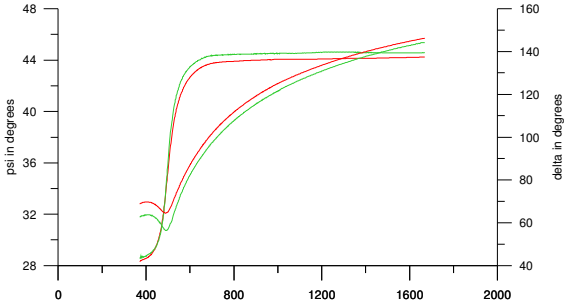


Figure 1. Fit of the JAW [5] and Palik [4] models to ellipsometry results (Δ) from flight qualified AuOS collector.

AuOS is an excellent example. Figure 1 shows the best fit of two literature models to a clean Genesis flight qualified AuOS collector. It is readily observed that neither fit is acceptable.

The flight spare becomes critical for measuring the actual optical properties of the material so it can be compared to those collectors returned from space. In this case, our gold coating has optical properties well bounded by the two literature models, but sufficiently different to make the fit unacceptable. The actual fit using the measured optical coefficients is shown in Figure 2.

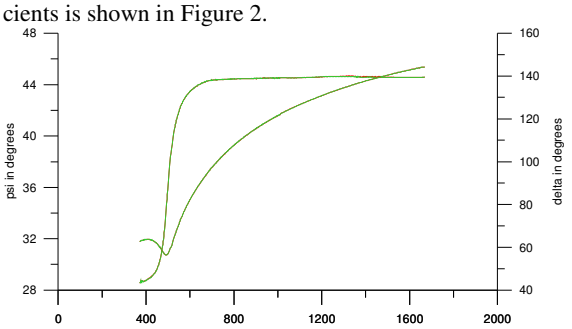


Figure 2. Fit using experimentally measured optical parameter, Δ. The fit corresponds to a clean gold surface with an average roughness of 4 A.

Results: Several areas were measured on each of the flight wafers, and the models discussed above were applied to assess the presence of molecular contamination on the collectors. The results were as follows:

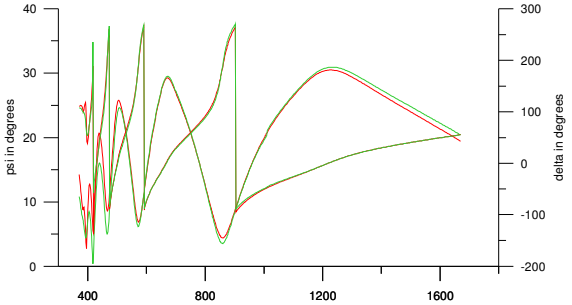


Figure 3. Experimental vs.model fit for the optic parameter Ψ for flight SiOS.

The flight SiOS wafer is best fit to a model consisting of a sapphire substrate covered with an epitaxial layer of Si that is 1814 +/- 15 A thick, and an SiO_2 layer with a thickness of 35 +/- 4 A.

The flight AuOS collector is best modeled as a pristine gold surface with optical properties matching the flight reference sample and 8 A of surface roughness.

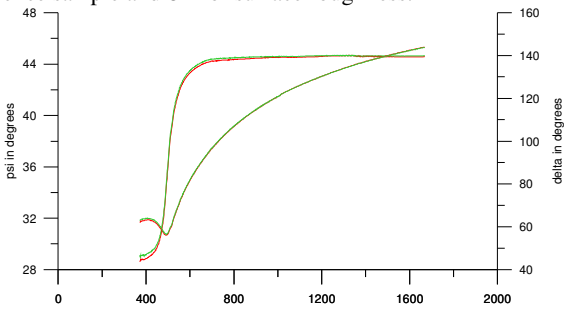


Figure 4. Experimental vs.model fit for the optic parameter Δ for flight AuOS.

Discussion: The flight SiOS shows an increase in oxide thickness by a factor of 2. This is consistent with the fact that flight collectors on the B array remained at elevated temperatures (as high as 200°C) for more than two years of science collection. Of course, no new oxygen is available, however oxygen will diffuse under these conditions forming a thicker layer of oxide with a sub-stoiciometric oxygen content. In fact, if one looks carefully at the fit to the oxide data (Figure 4), there is a small deviation in the model and the experiment. This is a result of the optical anisotropy dielectric constant of the suboxide.[6,7] The increase in index of refraction can also be related to the densification and increase in stress within the suboxide at relatively low growth temperatures or as a result of irradiation.[6]

The AuOS sample is an excellent match to the control sample. The only difference observed is a slight roughening of the surface, consistent with solar wind bombardment for 2+ years.

An attempt was made to fit the above data to a series of alternative models, including layers of hydrocarbon and silicone contaminants. No acceptable fits could be found.

Conclusions: Preliminary studies indicate that contamination as a result of out-gassing or other sources during flight did not occur to any significant extent during the Genesis Mission.

References: [1] E. K. Stansbery, LPSCXXXVI, Abstract, this volume; [2] B. E. Deal, A.S. Grove; *J. Appl. Phys*., **36(12)**, 3770 (1965); [3] I.W. Boyd, J.I.B. Wilson, *Appl. Phys. Lett*, **50(6)**, 321 (1987); [4] Palik; [5] J.A. Woolum Co., OptCcal constants Library (2004) [6] D.E. Aspnes, J.B. Theeten, *Phys. Rev. Lett*, **43(14),** 1046 (1979); [7]Y. Levy, M. Jurich, J.D. Swalen, *J. Appl. Phys*., **57(7)**, 2601 (1985).