Genesis Preliminary Examination – Ellipsometry Overview.

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Introduction: The Genesis spacecraft returned to Earth on September 8, 2004, experiencing a non-nominal reentry in which both the drogue and main parachutes failed to deploy causing the capsule to impact the surface of the UTTR desert at a speed of approximately 310 kph (193 mph). The impact caused severe damage to the capsule and a breach of the science canister in the field. The science canister was recovered and transported to the cleanroom at UTTR within approximately 8 hours of reentry. Although the ground water table did not rise to canister level before removal, damp soil and debris from the heat shield and other spacecraft components did enter the canister and contaminate some collector surfaces.

The objective of preliminary examination of the Genesis collectors is to provide the science community with the information necessary to request the most useful samples for their analysis. In order to achieve this aim preliminary examination includes

1) A complete inventory of collector materials available for scientific study. This includes: collector type, dimensions, notes on as-received condition, drawings, and photographs.
2) Traceability of collector fragments to their parent arrays, and where possible their flight location within that array.
3) Data on contamination levels, including particulate exposure and molecular contamination.
4) A detailed description of damage induced by physical abrasion, chemical reaction or other relevant processes.

Even though the collectors were not returned in the condition expected, the goals and most of the techniques planned for preliminary examination remain unchanged. Contamination of the collectors can be categorized as particulate, molecular, or chemical/reactive. One technique to address molecular contamination, spectroscopic ellipsometry, continues to be a valid and useful technique.

Ellipsometry Overview: Ellipsometry is a measurement technique using polarized light to characterize thin films, surfaces, and material microstructure (most commonly to measure thin film thickness and optical properties). The technique measures the polarization state change of light reflected from (or transmitted through) the surface of a material. Specifically, the values measured are expressed as psi (Ψ, relative amplitude change) and delta (Δ, relative phase change) relating the ratio of Fresnel reflection coefficients $\tilde{R}_p$ and $\tilde{R}_s$ for p- and s- polarized light [1].

Acquiring data at different angles provides the ability to assess the properties of material near the Brewster angle. When the angle of incidence is near the Brewster angle the measured Δ values are around 90° and this range of Δ values provides the most sensitive measurement of a material.

Limitations on the applicability of this technique are related to the wavelength of the incident light beam. The ability to characterize film thickness is best when the film and the probe wavelength are of the same general scale. In general, an infrared ellipsometer is best for thick films (100nm – 50μ) while the visible and near ultra-violet are best for thinner films (1Å – 1μ). Roughness at a surface or interface should be ≤10% of the probe wavelength. Larger features can cause non-specular scattering of the incident beam and depolarization of the specularly reflected beam. Film uniformity within the measured spot is also an important factor. If film thickness varies over 10% in the width of the beam spot the assumption of parallel interfaces may not be valid and modeled data cannot be expected to match experimental data. However, measurement of %-depolarization can help in the modeling of non-uniform samples.

General Procedure: Optical experiments rarely measure the direct parameter of interest (optical constant, film thickness, etc.); instead Ψ and Δ, which are a function of the desired parameter, are measured. The desired information must be extracted through a model-based analysis of the interaction of light and the material. In general, a model of the material and estimated surface characteristics is created based on knowledge of the physical characteristics of the material. This model is then fit to the measured data to determine the unknown physical parameter (such as thin film thickness). Best results occur from specular (mirror-like) surfaces. Inhomogeneities should be ≤10% the wavelength of light. When features (surface and interface roughness, grain sizes, etc.) are of similar dimension to the wavelength the light will scatter (diffuse) producing measurements that are difficult to interpret.

Standards: Since ellipsometry is an optical technique that requires developing a model of the material to analyze the measured data it is important to have an understanding of the optical constants of the substrate and deposition layers of the materials being measured. Although published optical constant sets are available for many materials, the optical constants of some materials are highly dependent on the deposition process. Metal films are notable because the grain structure and surface morphology greatly impact their apparent optical constants. For this reason we need to assess the optical properties of the Genesis flight-like materials before we can model and understand returned flight materials and any contamination or surface characteristics generated in flight or during impact.

Figure 1 shows the difference between published optical constants of gold metal [2, 3] and the measured optical constants associated with deposited gold film layer on sapphire for Genesis collectors. The Genesis flight-like standard was comprised of a 2000 angstroms layer of the highest-purity gold (6N; ESPI) that was e-beam deposited onto sapphire substrates [4]. Optical properties standards are being measured for all Genesis flight materials. A preliminary analysis of surface conditions for a returned Genesis collector using the gold standard shown in Figure 1 is in work [5].

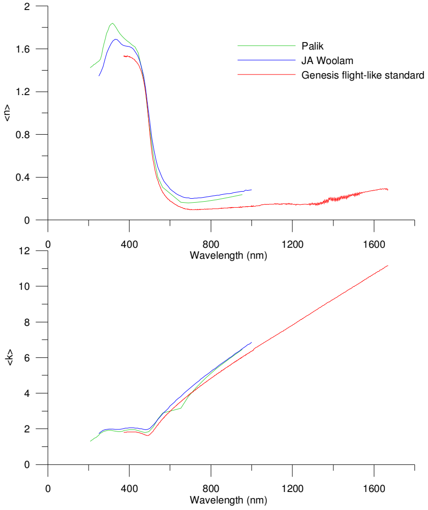

Figure 1: Optical constants of metal and film deposition

Size Study: The return of Genesis resulted in large numbers of small fragments (see [7] for details). Issues associated with small sample sizes include beam spot size, beam spot elongation at high angle of incidence, and edge effects. Therefore, a systematic study was undertaken to determine the smallest fragment for which spectroscopic ellipsometry measurements are valid. Understandable measurements are possible on a fragment as small as a 5mm × 4mm × 3mm triangle (Figure 3). It is also immediately obvious when edge effects occur as shown in the comparison between measurements near an edge but separated by 0.10cm. (see Figure 4).

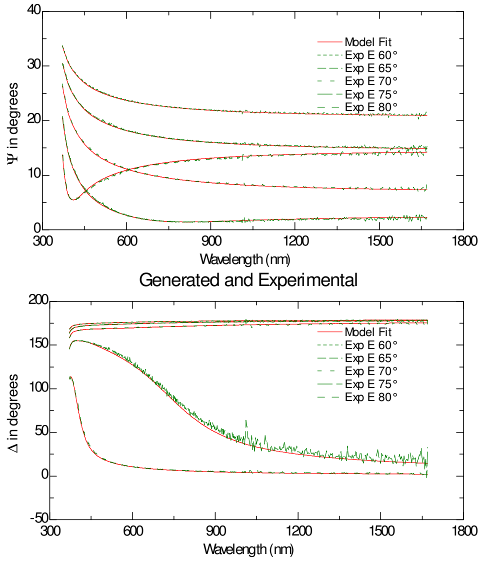

Figure 3: Psi and Delta for 5 x 4 x 3 mm flight-like Si fragment used in cutting experiments

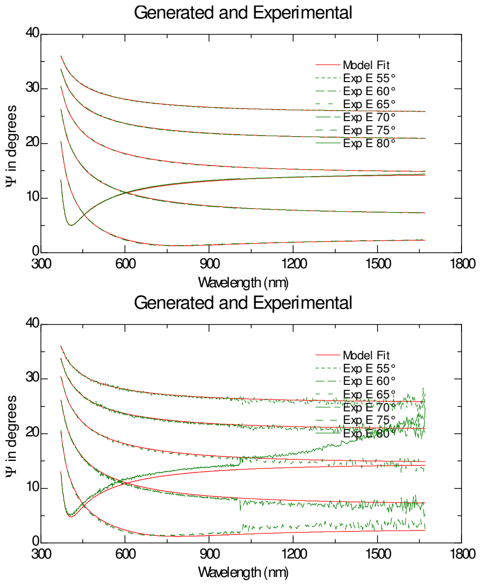

Figure 4: Psi measurements near the edge on a small Si flight-like fragment. The top graph is measurement is taken at position (-0.25, -1.1) and the bottom measurement was taken at position (-0.25, -1.2)

All measurements were taken using a variable angle spectroscopic ellipsometer shown in Figure 5.

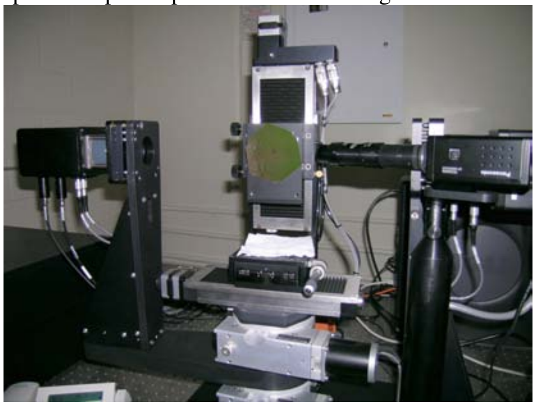

Figure 5: Variable Angle Spectroscopic Ellipsometer with full hexagon wafer mounted for analysis.

References:

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