Characterization of Deposits on Glass Substrate as a Tool in Failure Analysis: The Orbiter Vehicle Columbia Case Study

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In connection with the accident investigation of the space shuttle Columbia, an analysis methodology utilizing well established microscopic and spectroscopic techniques was implemented for evaluating the environment to which the exterior fused silica glass was exposed. Through the implementation of optical microscopy, scanning electron microscopy, energy dispersive spectroscopy, transmission electron microscopy, and electron diffraction, details emerged regarding the manner in which a charred metallic deposited layer formed on top of the exposed glass. Due to nature of the substrate and the materials deposited, the methodology proved to allow for a more detailed analysis of the vehicle breakup. By contrast, similar analytical methodologies on metallic substrates have proven to be challenging due to strong potential for error resulting from substrate contamination. This information proved to be valuable to not only those involved in investigating the break up of Columbia, but also provides a potential guide for investigating future high altitude and high energy accidents.

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Background

- In connection with the accident investigation of the space shuttle *Columbia*, an analysis methodology was implemented for evaluating the environment to which the exterior fused silica cockpit glass was exposed.
- Using well-established microscopic and spectroscopic techniques, details emerged regarding the manner in which a charred metallic deposited layer formed on top of the exposed glass.
- Due to the nature of the substrate and the materials deposited, the methodology proved to allow for a detailed analysis of the vehicle breakup.

Figure 1: Top view of orbiter forward fuselage. Window frames and glass fragments were analyzed as part of the investigation (NASA)

- By contrast, similar analytical methodologies on films and coatings atop metallic substrates have proven problematic due to:
  - Error resulting from substrate contamination in the form of carbon diffusion at elevated temperatures [1,2].
  - Carbon diffusion from material deposited on metallic substrates depleting the coating of base materials and compromising the bond strength [1]
- Studies on thermal plasma spray coatings have used a similar methodology to characterize the morphology of the deposited metals [3,4]
- Spectroscopic techniques in the semiconductor industry requires calibrated sputter depth systems to ascertain layer information, which is only valid when layers are homogeneous and of uniform thickness [5].
- The following methodology of analyzing molten deposits on a orbiter vehicle *Columbia* glass substrate provides a potential guide for investigating future high altitude and high energy accidents.

Optical Microscopy (OM)

- Orbiter vehicle *Columbia* window fragments were removed from their corresponding frames for investigation. Samples were carefully detached while meticulously documenting fragment location and orientation with respect to the orbiter (Fig. 2).
- Photodocumentation of each piece was performed on interior and exterior surfaces to determine the presence of any macro-scale features as well as differences in char layer appearance based on location (Fig. 3).

Figure 2: Schematic of orbiter upper forward fuselage indicating window nomenclature (NASA)

Figure 3: Photograph of a window pane fragment. Exposed surfaces after fracture were covered with a "char layer" (NASA/KSC)

- Window sample cross sections were prepared using standard metallographic techniques and evaluated via OM.
- A layer of deposited material denoted as the "char layer" found on the window fragments proved to have significant information.
- The char layers examined were characterized based on mean thickness and deposit attributes (voids, inclusions, etc.)
- The relative reflectivity of the char layer variegated through the layer's cross-sectional profile, indicating that the composition and/or morphology of each constituent varied as well (Fig. 4).
- Several char layer thickness profiles differed. The char layer dimensions more closely resembled a thick coating, rather than a thin film, on a glass substrate.

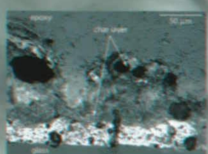

Figure 4: OM micrograph of a window pane cross section showing the features observed along the char layer, as well as the variation in thickness (NASA/KSC)

Scanning Electron Microscopy (SEM)

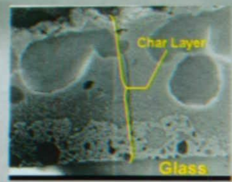

Figure 5. Back scatter electron image of Columbia window cross section. Image depicts typical char layer deposit on glass exterior surface. Magnification 500x (NASA/USC).

- Analysis continued at higher magnification in order to further characterize the morphology and composition of the deposited layer constituents (Fig. 5)
  - The polished cross section used for OM was coated with Pt for analysis in a Philips XL 40 Field Emission Gun (FEG) electron microscope.
- Significant findings of the SEM analysis revealed that:
  - The majority of porosity observed was confined to the upper-most regions, corresponding to final layers deposited.
  - Spherical globules were dispersed throughout the mid-section of the deposited layer.
  - Discrete regions of varying composition were correlated to OM observations of differences in reflectivity. Energy dispersive spectroscopy (EDS) revealed regions of aluminum, titanium, and silicon within the layer
  - Wispy features in the matrix which contained acicular formations were identified.
- Although SEM analysis provided semi-quantitative data, the above spectroscopic and microscopic techniques could not further resolve the nature of these structures.

Transmission Electron Microscopy (TEM)

- To ascertain the compositional characteristics of the char layer, relative to possible environments encountered during vehicle break-up, TEM analysis was carried out with specialized preparation by the focused ion beam (FIB) "lift-out" method.
  - TEM sample was prepared using an FEI 200 TEM FIB with a 30kV gallium liquid metal ion source.
  - Chemical and crystallographic analysis of the specimen was performed with an FEI Tecnai F30 TEM containing a 300 kV field emission source.
  - TEM was equipped with scanning transmission electron microscopy (STEM), high angle annular dark field spectroscopy (HAADF), and EDS.

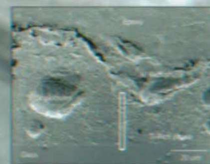

Figure 6. Ion image of char layer cross section before FIB in-situ preparation. Boxed region is outlined for Pt deposition. (NASA/KSC, UCF).

Figure 7. Ion image of char layer cross section after craters milling and prior to specimen lift-out. (NASA/KSC, UCF).

- An electron-transparent representative sample was prepared using the FIB "lift-out" procedure whereby the ion beam creates trenches around the region of interest (Fig. 7). An in-situ needle was spot welded to the sample using Pt; the sample was then liberated from its surrounding material by ion milling.
- Following sample release, a ditch slightly larger than the TEM sample was created on the inner diameter of ¾ TEM copper grid. The in-situ needle was lowered and carefully inserted into the ditch, the sample was spot welded to the Cu grid, and the needle tip was removed by the ion beam.
- FIB thinning was performed until the final thickness of the sample was less than 1000 Å. At this juncture, TEM investigation was able to commence.
- Crystals within the char layer were identified based upon the following morphological features:
  - Phases near the glass substrate appeared nodular with varying length scales (micro- to nano-scale) and had a more densely packed appearance than the grains identified in other regions (Fig. 8).
  - Phases identified in regions further away from the substrate interface were markedly more faceted and elongated in structure.
- Additionally, each crystal was characterized through chemical analysis via EDS and crystal structure via selected area electron diffraction (SAED).

Figure 8. Bright field TEM image of Columbia window near the interface of the glass and char layer (left). Crystals were characterized based upon morphology, chemistry, and crystal structure (right) (NASA/KSC, UCF).

- The observations made from TEM analysis provided insight into the behavior of the molten deposits on a glass substrate in a plasma environment.
- In the field of failure analysis such information can be critical in ascertaining the sequence of events which occurred.

Powder X-Ray Diffraction (XRD)

- Powder XRD data was generated, used to baseline trends, and verify compounds identified via the TEM.
  - The samples were evaluated utilizing a polarized light microscope and a Phillips XED powder X-Ray diffractometer.
- XRD samples were removed from a glass fragment originating from the same window as the TEM sample.
- A clear phase was identified as conchoidally fractured fused silica amorphous glass and comprised an estimated 40% of the sample weight. This phase was removed prior to subsequent testing to ensure analysis was restricted only to the char layer.

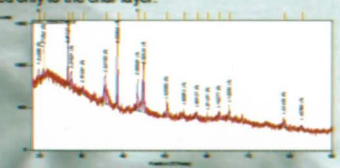

Figure 9. Typical XRD spectral data for char layer. Significant peak positions are noted by d-spacing calculation of 2-theta position. (NASA/KSC).

Conclusions & Future Work

- A methodology of analyzing deposited material on space shuttle *Columbia* windows was employed to further understand the sequence of events which occurred during the loss of the vehicle. This information proved valuable, not only to those involved in the *Columbia* accident investigation, but also to subsequent follow-on work. Analysis consisted of OM, SEM, EDS, FIB, TEM, and XRD techniques utilized to characterize deposited materials. The FIB lift-out method proved to be beneficial in attaining an information-rich sample representative of the char layer.
- Likewise, the methodology provides a potential guide for future high altitude, high velocity, high temperature re-entry accident investigation procedures (the *Columbia* was traveling at a velocity in excess of Mach 18 at an altitude of over 200,000 ft and experiencing temperatures in excess of 3200°F [6]).
- The myriad observations made on a thick layer of char atop an innocuous fragment of glass which survived the *Columbia* tragedy only serves to highlight the wealth of information that lies within her debris. The nature of the incident and subsequent breakup allow both engineers and scientists to glean much needed information, to learn how to not only make our existing space flight efforts safer, but provide a greater breadth of core knowledge for the next generation of vehicles.

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