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# Microscopic and metallurgical aspects of the Space Shuttle Columbia accident investigation and reconstruction

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The Space Shuttle Columbia was descending for a landing at the Kennedy Space Center (KSC) on February 1, 2003. Approximately 20 minutes prior to touchdown, the Columbia began disintegrating over the western United States; the majority of debris eventually impacted in eastern Texas and western Louisiana. A monumental effort eventually recovered approximately 84,000 pieces of debris, approximately 38% of the Orbiter's original dry weight. The debris was transported to KSC, where the items were catalogued and evaluated. Critical areas of interest, such as the left and right leading edge surfaces and the underside of the ship, were placed upon a grid to aid in the reconstruction. Items of interest included metallic structures, reinforced carbon-carbon composites, and ceramic heat insulation tiles.

Many of the leading edge elements had re-solidified metallic deposits spattered on them. These deposits became known as slag and were one of the main focuses of the investigation. In order to help determine the sequence of events inside the left wing during the accident, the slag's composition, layering order, and directionality of deposition were studied.

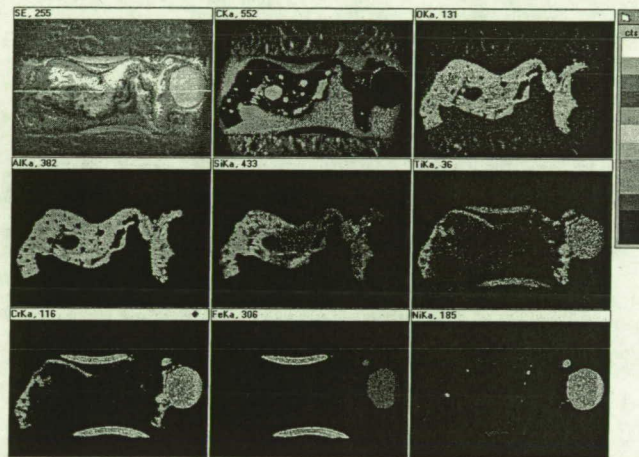
A myriad of analytical tests were performed in an attempt to ascertain the compositional and depositional characteristics of selected slag deposits, including the ordering of deposited layers within each individual slag deposit harvested. Initially, Scanning Electron Microscopy and Energy Dispersive X-Ray Spectroscopy (SEM/EDX) were performed to quickly characterize the overall composition of individual slag deposits. SEM utilizes a narrowly-focused high-energy electron beam impinging upon a specimen. The incident beam excites and liberates lower energy secondary electrons, which are detected and analyzed, providing a visual representation of the sample's surface topography. EDX also relies on an incident electron beam, except an EDX unit measures X-ray energies generated by the impinging beam. Each element generates a unique X-ray signature; the EDX detector measures these discreet energies. EDX actually penetrates approximately 2 microns into the bulk of the sample. However, random examination of various portions of slag, coupled with the semi-quantitative nature of the SEM/EDX analysis, did not yield convincingly pertinent data.

Therefore, X-ray dot mapping was conducted, which provided more understandable data, both in terms of slag layering and composition. An X-ray dot map is generated by performing numerous EDX scans for individual elements, then compiling the scans in a visual representation. Eventually, specimens consisting of not only the slag, but of the adjacent RCC substrate as well were cross-sectioned. X-Ray dot mapping of the materialographically-mounted and -polished cross-sections provided a visual representation of both the layering sequence and compositional characteristics of the slag, Figure 1.

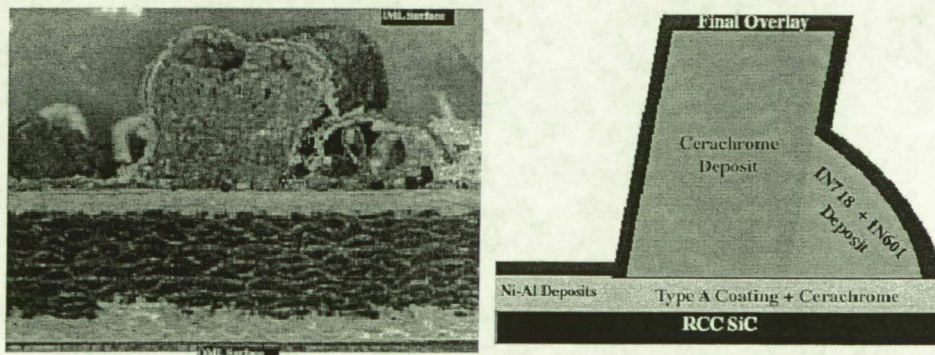
Contemporaneously, Electron Spectroscopy for Chemical Analysis/X-Ray Photoelectron Spectroscopy (ESCA/XPS) and powdered X-Ray Diffraction (XRD) were performed to further characterize the deposits and to attempt to identify what, if any, compounds were present. The ESCA/XPS analysis allowed the analyst to "sputter" into the sample with an electron gun, aiding in the identification of the layering sequence. XPS uses photons, rather

than electrons, which impinge upon the surface of the sample. XPS measures the electrons emitted from within the first 5 nm of the sample's surface. The XRD measures the scatter angles of incident X-rays; the angle and intensity of scatter depend upon the crystalline structure of the pulverized sample. XRD is considered a qualitative rather than quantitative technique. ESCA/XPS revealed that the final layer to deposit was predominantly carbonaceous. XRD was successful in identifying specific compounds, such as  $\text{Al}_2\text{O}_3$ , Al and/or  $\text{Al}_{3.21}\text{SiO}_{4.7}$ , mullite ( $3\text{Al}_2\text{O}_3\text{-SiO}_2$ ), and nickel-aluminides.

Eventually, Electron MicroProbe Analysis (EMPA) was conducted on the materialgraphically-prepared cross-sections of selected slag deposits. Microprobe combines SEM and Wavelength Dispersive X-Ray Spectroscopy (WDS), and, like EDX, uses a narrowly-focused high-energy electron beam impinging upon a specimen to elicit, in the case of EPMA, characteristic X-rays with specific wavelengths. This quantitative, analytical tool proved the most useful in determining depositional layering and composition of the slag deposits, Figure 2. This information was utilized in verifying the location of the breach in the left leading edge of the wing of the Columbia.



**Figure 1.** X-Ray Dot Map of a slag deposit harvested from LH RCC Panel 8. The top left pane represents the cross-section; each sequential pane represents the relative amount present of a specific element, respectively C, O, Al, Si, Ti, Cr, Fe, and Ni. The vertical legend to the right of the dot map indicates the relative intensity of counts for each element; black indicates fewer counts, white denotes a higher number of counts. [1]



**Figure 2.** Materialgraphically-prepared cross-section and schematic representing results obtained via Microprobe Analysis of a slag deposit from LH Panel 8 [2].

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