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SURFACE ANALYSIS AND MATERIALS CHARACTERIZATION FOR THE STUDY OF THE CERAMIC TILES HRSI FROM THE SPACE SHUTTLE THERMAL PROTECTIVE SYSTEMS

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Abstract

The orbital thermal management systems fall into two categories, ablative or reusable. The first type was successfully applied on the Apollo missions, the second was used on the Space Shuttle Orbiters. The ceramic High-temperature Reusable Surface Insulation (HRSI) tile exemplifies one of several reusable Thermal Protective Systems (TPS) which are essential in shielding the cargo of a spacecraft during reentry to the Earth's atmosphere. Surface topography of the HRSI glossy coating on unflown and flown tiles was characterized using optical confocal microscopy instruments, and surface roughness analysis to extrapolate evidence of the impact of the atmospheric reentry on the surface and to complement optical secondary electron imaging produced by Scanning Electron Microscopy (SEM). The qualitative elemental analysis of the bulk material in the flown HRSI tile was characterized with Energy Dispersive X-ray Spectroscopy (EDS) and referenced with available technical sources from NASA's flight manuals. This multivariate investigation of TPS's HRSI provided a basis for better understanding of the space heritage artifact such as the Space Shuttle Orbiter Discovery that recently was acquired by the National Air and Space Museum; its TPS exemplifies the technological advancement of materials used in space exploration. The authors combine their diverse experience in surface metrology and artifacts' museum analysis and preservation contributing to the knowledge of material science embodied in space heritage artifacts.

Introduction

The term Space Shuttle is an abbreviation of a program known as the Space Transportation System (STS) which began its operation in 1981 and concluded with the last launch three decades later, in 2011. The fleet of five STS Orbiters comprised the first space vehicles in history that traveled multiple times to LEO (Lower Earth Orbit) and back to Earth. Multiple reentries into Earth's atmosphere posed engineering challenges to develop thermal protective systems (TPS) that would withstand physical impacts such as surface pressure, catalytic and radiative heat, shearing heat, vibration, turbulence and acoustic excitation, all simultaneously occurring during the reentry event. Ablative systems, which successfully protected space vehicles of the Apollo mission, could not be reused, they disintegrated upon reentry and the design of a monolithic shield was too heavy to apply on the large STS orbiters. The reusable TPS had to be light enough to cover the surface of a space vehicle that measured 37.2 meters x 23.8 meters. Because not all areas of the orbiters were exposed to the most extreme heat, reaching over 1,260° C on the nose cap and leading edges, several deferent TPS systems were applied to accommodate ranges of temperatures (figs. 1A,1B). Traveling at a speed of 27,360 Km/hour, friction with the atmosphere produces surface temperatures reaching 1,650° C [1].

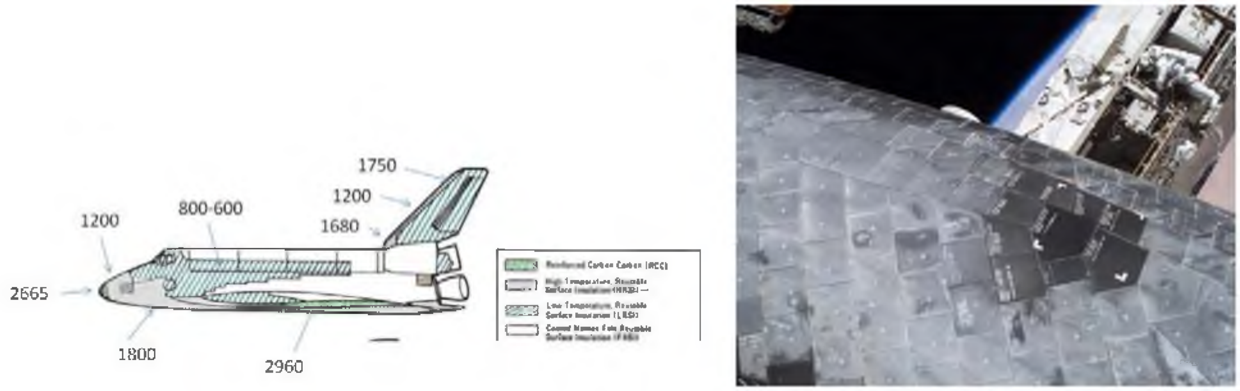


Fig. 1A Fig. 1B

Figure 1A. A diagram of STS Orbiter showing nominal maximum temperatures on the surface and location of deferent TPS systems). The HRSI tiles were applied in areas on the upper forward fuselage on the entire underside of the vehicle and near the orbital maneuvering system [2] Figure 1B. Black coated HRSI tiles attached to the STS orbiter. (Photo courtesy of NASA, public domain, open source).

The need for reusable and light weight material led to the development of composites, such as Reinforced Carbon-Carbon (RCC) and High–temperature Reusable Surface Insulation (HRSI) ceramic tiles among many new materials. The history of materials' development and characteristics of RCC in the context of space heritage are discussed elsewhere [3]. This report focuses on HRSI, the surface analysis and materials characterization of the flown and unflown tiles which are not only testimony to technological advancement in material development but also provide information about the effects of atmospheric reentry on the materials. The early STS orbiters used 34,000 tiles which later were reduced to 26,000, replaced with flexible insulating blankets on areas which experienced moderate reentry temperatures.

Technological advancements of HRSI tiles

Relying on NASA fact sheets, which provide general information about the materials used in STS TPS, this overview was compiled to serve as a backdrop and reference for interpretation of our multivariate analysis of the HRSI tiles. The HRSI ceramic tiles were used on all STS

Orbiters protecting the areas where temperatures were below 1,260° C [4, 5]. HRSI's main component of the bulk material is high-purity silica in a form of amorphous fibers with ceramic bonding that ensures rigidity. The voids constitute 90% of the total volume and the material the remaining 10%, thus producing a light weight structure. The tiles are fabricated from water slurry with silica fibers frame-cast and bound with colloidal silica binder, sintered and then cut to specific dimensions. The tiles' thickness ranges from 25.4 millimeters to 127 millimeters, depending on where it is positioned on the orbiter according to the expected heat load; thicker in the forward area and thinner in the aft.

The external surfaces of the tiles that face the environmental extremes are coated with a mixture of powdered tetrasilicide and borsilicate glass, applied by spraying. Once coated, the tiles are heated in an oven to 1,260° C, turning the coating into a black, glossy layer. Because the tiles cannot withstand airframe load deformation, the stress isolation pads of Nomex felt were added to the side that attaches the tiles to the orbiter's surface. A thin layer of silicon adhesive RTV was applied to the reverse of the tile [4].

Samples' description

Two ceramic HRSI tiles were analyzed in this investigation, one being flown on mission STS 46 and the other unflown (pristine condition) (figs.2, 3). With the retirement of the Space Shuttle program, the unflown single tiles were distributed to museums across the country for educational purposes, however without supportive technical information. The serial number on the examined unflown tile, VO70-193005-205-JF9816, indicates that the tile was designed for the lower elevons on the wing of the Vehicle Orbiter (VO). Unwrapping several layers of a clear plastic that protects the friable material from mechanical damage permitted measurement and optical analysis of a limited area of 25 x 25 millimeters area of the surface, bulk and base (fig.2). Similarly, the surface coating, bulk and base were investigated on the flown tile. The flown tile however underwent additional testing after its removal from the orbiter, which involved exposure to high temperatures of a torch. Therefore characterization of the surface needs to take into consideration possible additional changes of the material that occurred during post-flight tests.

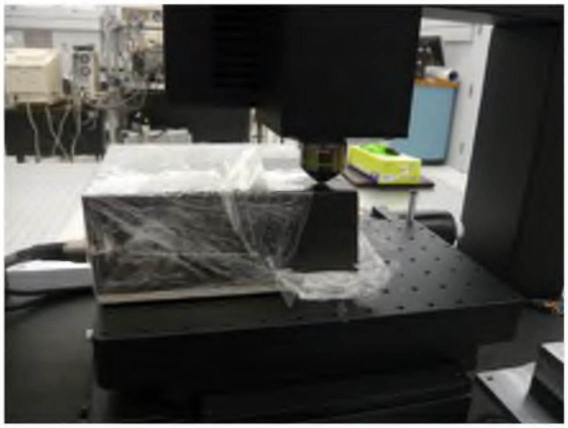


Fig.2

Fig.3

Figure 2. The unflown tile V070-193005-205-JF9816 analyzed with a disc-scanning confocal microscope. The unflown tile was examined using non-destructive surface metrology techniques. Figure 3. A flown tile from the Orbiter Atlantis (STS-46, 2009) after severe post-flight testing shows brittleness of the ceramic black coating. In addition to examination with a disc-scanning confocal microscope, samples of coating and bulk were examined using SEM-EDS analysis.

Testing protocol and instrumentation

The testing protocol applied in gathering data from unflown and flown tiles involved imaging and topography measurements of the surface, bulk material and base-side which was intended to be attached to the orbiter's surface. The elemental analysis was carried out on the flown sample. The pristine conditions of the unflown tile did not permit sampling of materials.

The surface topography of the unflown and flown tiles was examined with an Olympus DSX 100n high resolution optical digital microscope with extended focus imaging (EFI) capability. Macro-features of the flown tile coating and bulk material were imaged with a SEM Hitachi S4700. The surface topography of the black coating and bulk material of both tiles, unflown and flown, were measured with a NanoFocus µSurf Nipkow disc-scanning confocal microscope. The morphological features of the flown tile coating and bulk material were imaged with the back scattered and secondary electron SEM, Hitachi SU6600 coupled with EDS for qualitative elemental analysis. The spectral data of present chemical elements was mapped using Bruker's processing software. Although SEM-EDS are considered non-destructive techniques, sampling of the original material is required. This limitation does not apply to the surface topography measurements which were performed in a non-contact mode and do not alter the sample being measured. The ability to measure objects without the necessity of sampling is a particularly desirable feature in the examination of cultural heritage material.

Results, surface topography and materials characterization.

The intended purpose of the coating on the STS tiles was to minimize the effect of solar heat absorptance [6] which made the analysis of the samples with light-based instrumentation particularly challenging. The surface topography of the surfaces on the unflown sample was compared to that of the flown tile. Although the surface features of the coating on the flown tile indicated changes, as expected (fig.4,5) they were not significant. Furthermore, this applies to changes of the surface only; the body of the coating below the surface showed unchanged macro-features (fig.6).

The quantifiable changes of the surface were captured using a Nipkow disc-scanning confocal microscope and produced three dimensional topographic maps of both surfaces (fig.7,8). The aeral surface roughness (*Sa*) of each measurement was compared using digital Gaussian filters with a long cutoff of 0.25 mm to remove surface tilt and long wavelength components that are part of the form [7]. Each surface showed a *Sa* of between 2 µm and 2.5 µm. While the *Sa* does not change significantly enough to make any definitive conclusions on the overall roughness, there are structural changes that show the emergence of cracks in the top surface coating of the flown tile. The evidence and extent of alteration is visible when comparing the maps to each other. As mentioned earlier, it should be noted that the flown tile has also undergone significant heat testing post-flight. Therefore a direct comparison cannot be made at this time. If suitable flown tiles can be acquired in the future, that are in good condition with no post-flight testing, then a more direct comparison of the surface topography can be made.


Fig.4

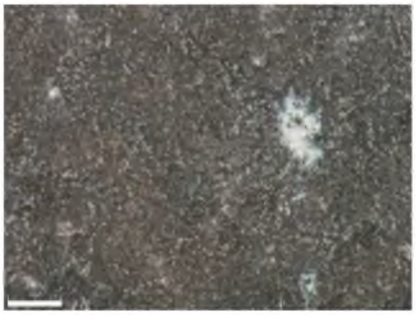
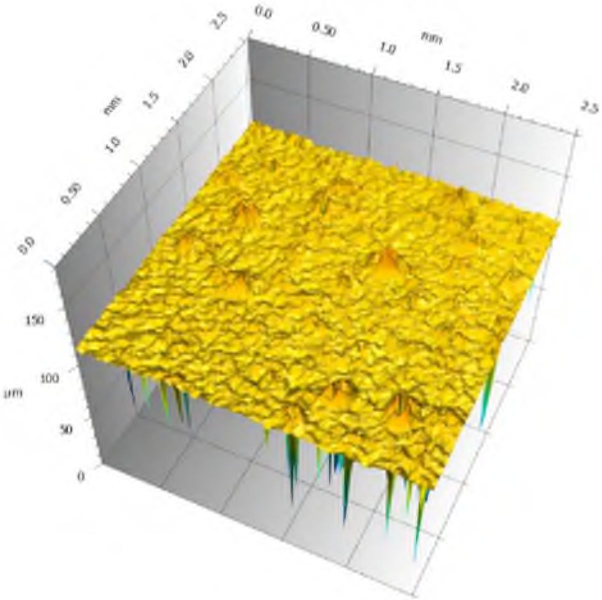


Fig.5


Fig.6

Figure 4. Micrograph showing features of the surface topography of black coating on the unflown tile, captured with an Olympus DX100 digital microscope, in the extended focus imaging mode, at 45° angle; scale: 200µm.
Figure 5. Micrograph showing features of the surface topography of black coating on the flown Atlantis tile, captured with an Olympus DX100 digital microscope, in the extended focus imaging mode, at 45° angle; scale: 200µm.
Figure 6. Cross-sectional view of the black coating from the same flown Atlantis tile showed

unchanged features of the coating. Image taken with an Olympus DX100 digital microscope, in the extended focus imaging mode; scale: 200µm.



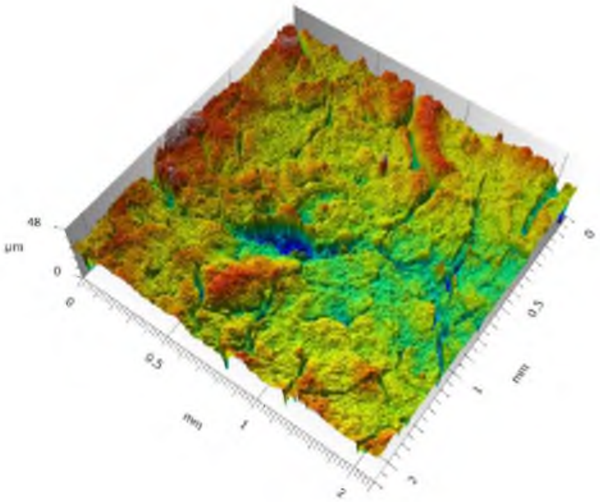


Fig.7

Fig.8

Figure 7. False color 3D surface topography map of the unflown tile; top black surface coating, in pristine condition. Note: Pitting of surface is visible as downward dropouts.
Figure 8. False color 3D surface topography of the flown tile; top black surface coating, exposed to atmospheric reentry conditions and additional post-flight testing. Cracking of the top surface coating is visible. Red color represents high points on the topography; blue represents low points.

The bulk material of the unflown and flown tiles below the top surface coatings, showed well defined refractory silica-based fibers. Their fibrous characteristics appeared to be unchanged in the flown tile. (fig.9, 10). The material alterations were observed on the burnt surfaces where the fibers were directly exposed to additional heating during post-flight testing (fig.11).

Fig.9

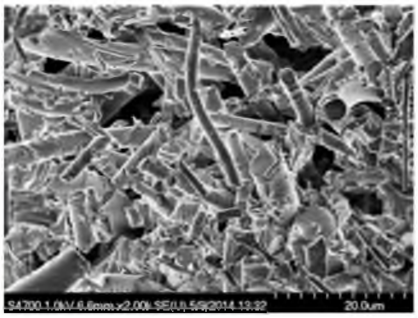


Fig.10

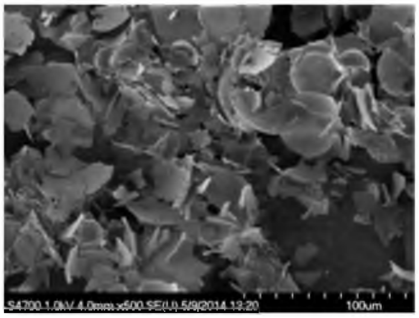


Fig.11

Figure 9. The bulk material in the interior of the Atlantis flown tile was not changed during reentry, it showed well preserved fibrous features. Micrograph taken with an Olympus DX100 Digital Microscope, in the extended focus imaging mode, scale: 200 µm.
Figure 10. SEM micrograph of the bulk material from the Atlantis flown tile shows hollow, silica fibers. SEM Hitachi S4700, scale: 20 µm.
Figure 11. SEM micrograph of the same fibers exposed to extreme heat in post-flight testing shows altered materials that have lost their characteristic fibrous features. SEM Hitachi S4700; scale: 100 µm.

The elemental qualitative analysis carried out using the SEM-EDS of the flown sample of Atlantis tile was in agreement with technical NASA references pointing to the presence of Si and Al in the bulk material (fig.12).

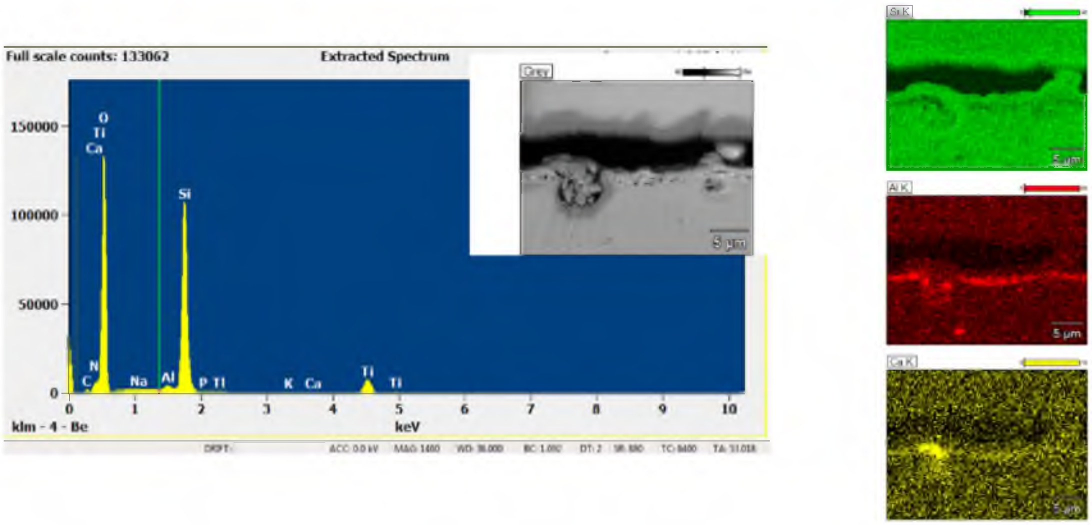


Figure 12. Spectral mapping of elements present in a sample taken from the Atlantis flown tile. The inset shows the cross-section of the examined samples', black coating and white bulk material. On the right is mapping of the most prominent elements Al and Si in the examined sample. The presence of Ca, prominent on the spectrum, is attributed to artifacts acquired during sample preparation.

Conclusion

A multivariate approach to the analysis of complex space heritage material enables one to extrapolate information about the impact of atmospheric reentry on the surfaces of HRSI tiles. The minimal changes observed on the surface and in the bulk indicated resilience of the materials that were designed to be reused in multiple reentries. The application of various analytical instrumentation provides complementary information about the tested materials. The complexity of aero-space materials and their unique characteristics required instrumentation that show expanded range of measuring capabilities encompassing the unique features of the examined materials. Surface metrology techniques are superior in the investigation of cultural heritage artifacts allowing their examination without the need to extract material samples from the original artifacts. This report presents results of preliminary examination; the work will continue once more samples of STS flown tiles became available.

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