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Space Heritage: The Apollo Heat Shield; Atmospheric Reentry Imprint on Materials' Surface.

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ABSTRACT

The heat shield is part of a thermal protective system (TPS) essential in shielding the cargo of a spacecraft during reentry to the earth's atmosphere. The ablated surface of the heat shield is a testimony to the harsh reentry environment, evidenced in melting and charring among other phenomena that occur during reentry at velocity of 9-11 km/sec. The aim of this study was to extrapolate information about atmospheric reentry from the surface of the ablated material. A sample of the heat shield from the test vehicle of the Apollo Program, AS-202, was the subject of the analysis.

For the preliminary studies, selected investigation modes from the Global Optimal Strategy model, developed to identify wear of engineering surfaces, were applied: examination of structure, optical observation, physico-chemical characterization and surface morphology. Instrumentation used included: microscopic surface analysis with Extended Depth of Field composite images (EDF), Fourier transform infrared spectroscopy (FTIR), attenuated total reflectance (ATR), confocal scanning laser microscopy and laser scanning microscopy. The Apollo Program testing vehicle AS-202 (1966) ablated specimen sample was obtained from the collection of the National Air and Space Museum (NASM), Smithsonian Institution, Washington DC. The authors combine their diverse experiences in tribology and in artifacts' museum conservation so as to contribute to the space heritage material science. This study represents one of the building blocks of a larger project, the Fundamental Model of public outreach and perception (FAM-pop) of complex aerospace technologies.

INTRODUCTION

One of the most difficult technical problems in the early years of space exploration was the design of the space vehicles so that they could withstand reentering the earth's atmosphere. The ship and its cargo had to be protected from the intense aerodynamic heating associated with the supersonic flight speeds.

Numerous materials were considered as potential candidates to be used in the ablative Thermal Protective System (TPS). The entire assembly consisted of the top ablative layer and a stainless steel substructure (Fig.1,2) The top layer of the heat shield thickness varied following the requirements of lunar return. It ranged from 1.7" to 2.8" along the three-piece heat shield (forward, crew, and aft compartment heat shield) according to the heat load expected from a lunar return trajectory [1]. The final selection was a thermal setting composite, an epoxy resin matrix reinforced with silicon-oxide fibers. A successful return of the Apollo 11 from its lunar exploration mission was the best testimony of the TPS protective qualities. Understandably, that material served as base for developing the new generation TPS in for the future lunar expeditions.

The surface morphology, structure, and chemical composition of both ablated and non-ablated surfaces were investigated using various analytical techniques, and shed light on the complexity of materials in TPS systems and the forces that affect them during atmospheric reentry. Evaluation of data generated by the techniques used in this investigation indicated their suitability for the future studies of space heritage artifacts.

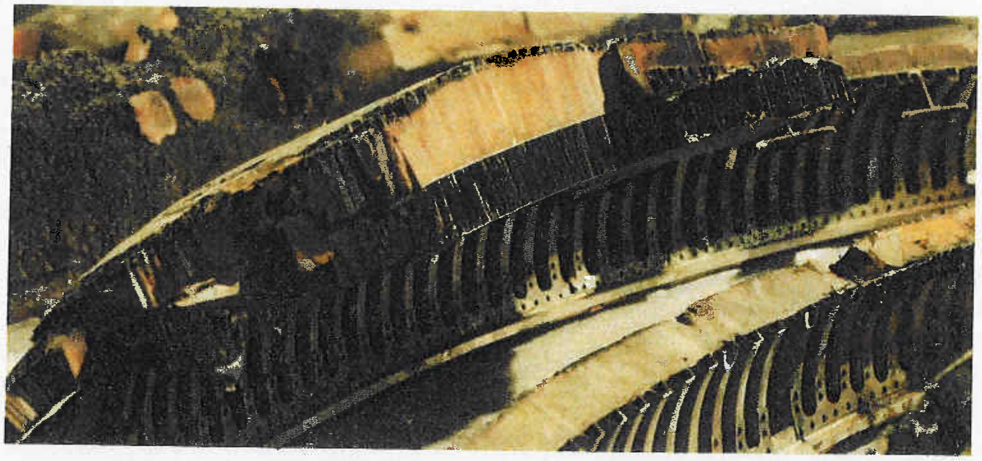
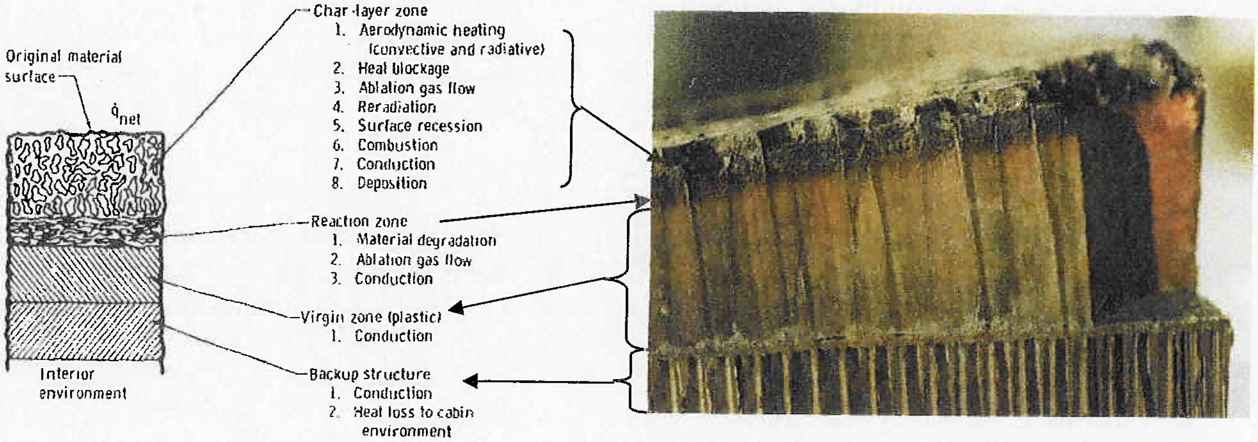


Fig. 1. Cross-section of the actual AS-202 heat shield remnants in NASM collection (NASM A 197314230000.)



a)

b)

Fig. 2a. Schematic diagram of a charred heat shield ablator (Courtesy , NASA TN D-5969) [2].
Fig.2b. Cross section of the ablated heat shield, top layer. The corrugated, backup substructure is only partially visible.

ATMOSPHERIC REENTRY

Atmospheric entry or reentry is the movement of natural or human-made objects, respectively, into the atmosphere of a planet from outer space. For our planet this is considered an altitude above the Karman Line (100km) [3].

The space vehicle reentering the atmosphere experiences an impact of various factors such as surface pressure, convective, catalytic and radiative heat, shear heating, vibration, turbulence, just to name a few. In that environment the heated surface interacts with the boundary gas layer moving around the outer shell. Consequently the temperature varied in different places on the heat shield, ranging from 1500°C to 2000°C and higher in relation to the angle of reentry. These high temperatures lead to internal physical-chemical transformations of the composite material accompanied by ablation, which is the erosion of a solid body by a high-temperature gas stream moving with high velocity that melts or chars. The pyrolysis that occurs during ablation results in two new phases: a polymeric phase containing solid products of decay (as a rule, carbon), i.e., the char, and a gas–phase containing gaseous products of decomposition of the polymer. There are two types of ablation, surface and volumetric, depending on the forces impacting the surface and the type of ablative material, discussion of which is beyond the scope of this paper [4].

It is critical that when the char forms it is not dislodged from the material, otherwise the resulting surface loss would facilitate heat transfer compromising effectiveness of the entire thermal protection system [5]. The study focuses on the ablator's surface, the only available witness of the events of atmospheric reentry. The objective of the AS-202 test vehicle was the testing of the heat shield for the eventual Apollo 11 mission.

SAMPLE DESCRIPTION

The sample of a heat shield from the test vehicle AS-202 available for this study was the top, outer part, called ablator. The top layer of the TPS ablator is charred while retaining an un-ablated (virgin) material below it. The sample has a honeycomb hexagonal matrix structure produced by injecting a polymer composite into a hexagonal matrix of fiberglass impregnated with a basic nylon-phenolic resin and dip-coated with polyester according to NASA's technical notes. The composite used to fill the cells is a proprietary product, Avcoat, a thermal setting phenolic- epoxy resin reinforced with silicon—oxide fibers and phenolic microballoons. The phenolic microballoons are not defined or described. The reinforcing fillers serve to optimize the properties of the composite material, such as heat conductivity, ablation rate, elastic modulus, etc. The depth of each cell in the honeycomb matrix is considerably greater than its width, to ensure their retention within the matrix. [6] Once the cells were filled, the honeycomb was bonded to the stainless-steel shell of the spacecraft with HT-424 adhesive; tape-like fashion. The stainless-steel substrate, in three parts, was assembled on the spacecraft. The final surface finish consisted of a thin layer of epoxy-base pore sealer and a moisture-protective plastic to ensure sealing of the porous ablator. Plastic coating was stripped off before launching of the space vehicle. [7,8]

HEAT SHIELD TESTING PROTOCOL

In our preliminary investigation of the TPS ablated surface the principles of Global-Optimal Strategy (GOS) model were followed. This Model was developed by the scientists at the Laboratoire de Tribology et Dynamic des Systems, Lyon, France, for identification of wear mode of engineering surfaces [9]. Although it is a holistic approach, inclusive of all possible modes of surface evaluation, for this investigation only some diagnostic techniques were selected:

1. Optical examination of surface structure, high-resolution surface microscopy captured as Extended Depth of Field composite images (EDF)
2. Physico-chemical surface material characterization, using FTIR and ATR spectroscopy
3. Surface metrology, with confocal and laser scanning microscopy (LSM)

The selection of surface metrology technique was based on its representing the most immediate link between the human visual perception and quantification of topographical metrology of a studied surface. That aspect has been utilized in the formulation of a Fundamental Methodology for Public Outreach and Perception of complex aerospace technology (FAM-pop). [10]

Optical microscopy

'Total observation' , the first step in the GOS Model of surfaces evaluation provides information about surface characteristics, such as its homogeneity or heterogeneity, gross marks observed or types of inclusions, among other features. Information obtained from the optical examination serves as a base for selecting the surface metrology technique. The high-resolution stereo-microscope used in the optical examination was Olympus SZx12 and camera QImaging U-TVO.5xc-2. The resulting images were generated in the Extended Depth of Field mode (EDF). The surface was clearly heterogeneous, fibrous material was packed in cloth-like matrix and charred. (Fig. 3 a, b) Features, such as microballoons, were revealed during optical examination. (Fig. 4).

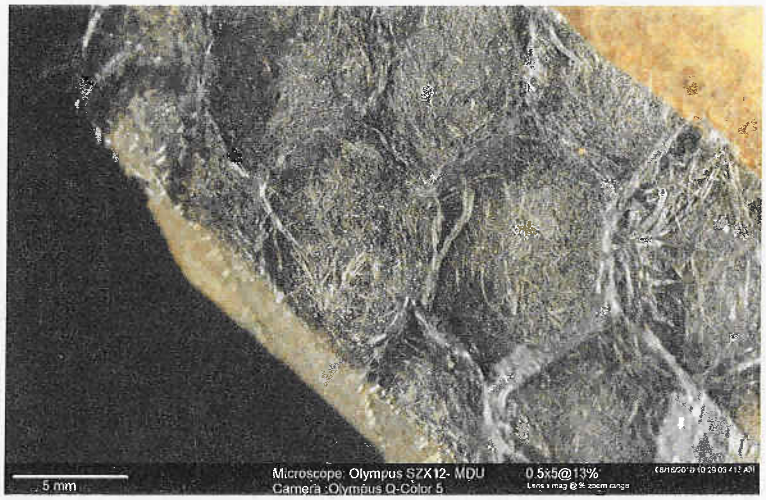


Fig. 3a Close-up of the ablated surface, showing its roughness and fiberglass embedded in charred material and configuration of the hexagonal matrix, test vehicle, AS-202; surface analysis microscope, Olympus LEXT OLS4000, EDF image.

Fig. 3b The fiberglass matrix impregnated with nylon-phenolic resin cloth; surface analysis microscope, Olympus LEXT OLS4000, EDF image.

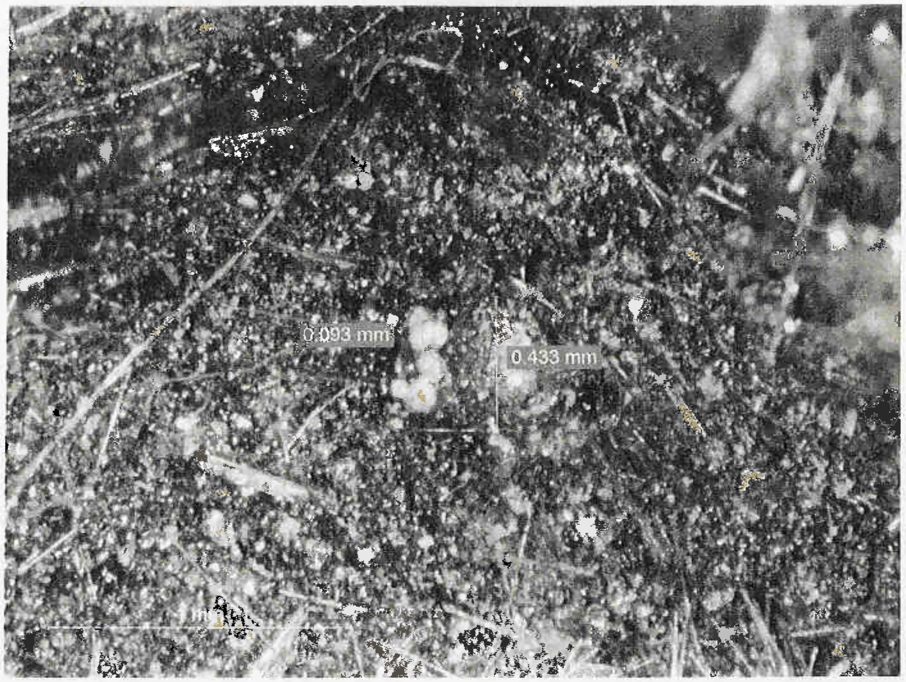


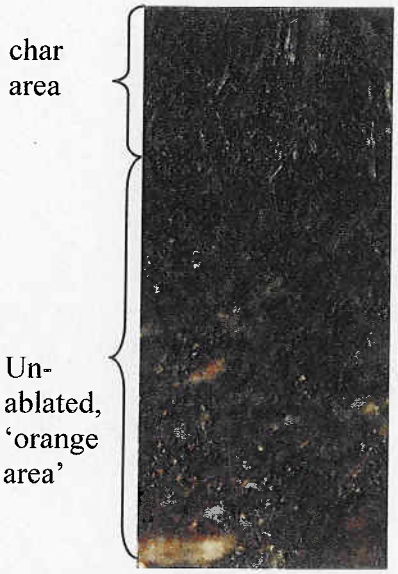
Fig. 4 Aerial view, showing 3D morphology of the ablated surface with a microballoon protruding from the surface's matrix. (Multi Focus Montage, courtesy, Melvin Wachowiak, Senior Conservator, MCI, Smithsonian Institution)

Physico-chemical characterization

Spectroscopic analysis, ATR-FTIR provided information about chemical composition of the examined material, type of organic compounds present, their concentration and distribution on the surface (chemical mapping).The analytical instrumentation used in the examination included (courtesy of ThermoFisher Scientific) a Nicolet iN10, Infrared Microscope, and a Nicolet iS10 FTIR with diamond ATR accessory. Three regions of the sample were examined, (i) the top, (ii) the charred surface and (iii) the un-ablated material; both the polymer and the honeycomb matrix that presented unique spectral features. FTIR was selected as it is particularly useful in the characterization of organic materials. Alterations in the ablated part were indicated by loss of absorption in the region of 1700 cm^{-1} and near 3000 cm^{-1} when compared with the un-ablated material (referred on the spectrum graph as 'orange area'). The spectral features are characteristic for C=O stretch near 1700cm-1 and CH stretch at about 3000cm-1, indicating a loss of organic compound in the charred area. (Fig.5) FTIR data cross referenced with the spectral library only in some cases correlated with NASA technical notes. The materials were identified as:

1. Charred layer: possibly carboxymethyl cellulose (57.65%)
2. Un-ablated matrix: phenol resin in the hexagonal cloth-like matrix (44.43%)
3. Un-ablated material: sebacic Acid (80.29%) or polyester (74.18%) depending on the analyzed site.

The found matches of some of the materials were too low quality to be considered as indicative of the presence of these materials. Among them, only the phenol resin and polyester are the closest match to the materials referenced by the technical and historic notes from NASA of that period (1965).[11]. Presence of carboxymethyl cellulose is interesting in this type of the heat shield because its use was mentioned as 30-50% by weight additive to thermal control coating in the later applications such as to a space shuttle shield.[12] Furthermore, other techniques may be needed to identify the rest of the material present in the charred section. Sebacic acid, used in the manufacture of certain synthetic resins and fibers, various plasticizers, and polyester rubbers may indicate a plasticizer in the ablator mass, or remnants of a sealer which was applied on the virgin material. Further analysis of the virgin material is necessary prior to reaching definite conclusion.



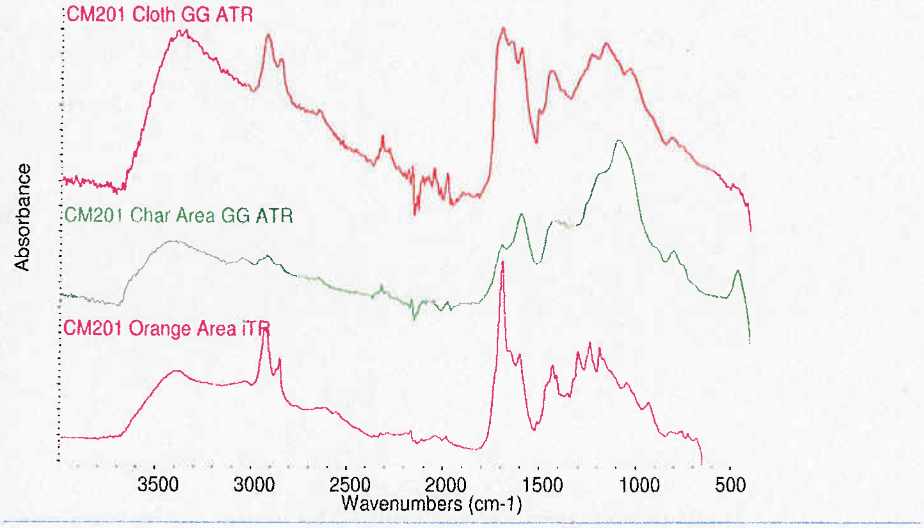


Fig. 5 Cross-section of the AS-202 heat shield and corresponding FTIR spectra of each region; charred, un-ablated (orange area) and matrix show molecular changes in each material as a result of exposure to the harsh environment during re-entry.

Chemical mapping (Fig.6) generated from FTIR analysis contributed information about distribution of chemical compounds; it is yet another tool of surface characterization. It served as a visual complement to the chemical, optical examination and interpretation of the technical historical sources. However, it did not provide a great deal of information about the causes which produced that particular outcome, or the ablation event itself.

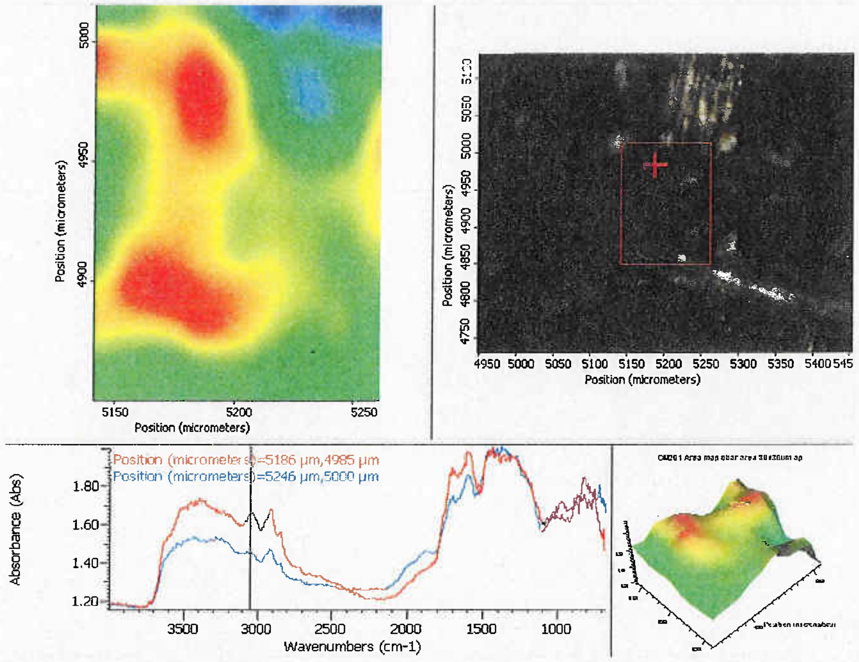


Fig. 6 Chemical mapping, illustrates intensity and distribution of chemical compounds of the charred area with fibrous matrix. The map was generated based on IR spectrum data collected from an 3mmx3mm area. (courtesy Thermo Fisher, J. Dorsheimer).

Surface metrology for evaluating surface wear aspects

Surface metrology is the study of surface geometry, its texture and roughness. Analysis of the measured patterns reveals how a particular texture was influenced by its history and how it influenced materials' behavior. Among a broad array of surface metrological tools two were selected as the most appropriate for measuring the heterogeneous surface of our sample; a confocal, scanning laser microscopy (Olympus, LEXT OLS4000) and laser scanning microscopy

(UBM Laser Microscope, Solarius Development Inc.). Both employ a non-contact, optical scanning to measure surface topography.

Surface metrology, widely used in the industry, to the authors' knowledge surface metrology has not been applied to the evaluation of space heritage artifacts; it is a novel application of a highly precised tool. The resulting image of surface morphology is obtained by complex mathematical formulations calculating differences in wavelength of the laser beam reflected from the measured surface.The preliminary study served to reveal new information about the structure of the surface and the causes which shaped it. For example, the presence of phenolic microballoons was barely mentioned in the technical literature and no characteristics or description was given. In the charred area of the sample, one microballoon was readily detected by laser confocal microscopy and measured to be 0.4 mm in diameter (Fig.7). The intact spherical shape of the microballoon revealed the material's characteristics, such as its resilience to wear during atmospheric re-entry.

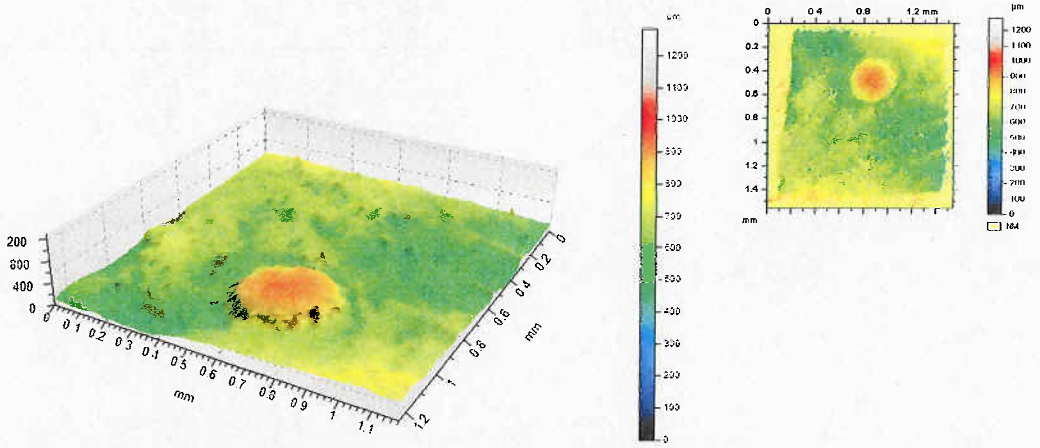


Fig.7 3D topographical mapping of the ablated surface illustrates a phenolic microballoon. Presence of this inclusion is indicated by color intensity, orange, pointing to its height and elevation above the softer and ablated surrounding. (The measurements were carried out at the Surface Metrology Laboratory, WPI, Worcester, MA)

Materials wear occurs at different rates during their exposure to the external, aggressive elements. For example, the most exposed, upper surfaces of the heat shield were wearing differently than the ones located in the deeper layers of each honeycomb cell.Application of just one of many available tools of surface metrology demonstrated a promising potential for these techniques in the investigation of cultural heritage surfaces. Further investigation of others surface metrological tools will be undertaken in the studies of various cultural heritage artifacts.

CONCLUSION

Information imprinted into the ablated heat shield, interpreted through various analytical techniques allowed a better understanding of the complexity of chemical and physical reactions occurring in materials and their causes, atmospheric reentry.

The analytical techniques employed in this preliminary study confirmed the scarce historical records of materials used, as in the case of the analysis of the unablated portion, as well as provided new information as for the microballoon. The characterization of the ablated heat shield requires more extensive and in-depth studies to establish the type of compounds in the un-ablated region and to trace the molecular changes in the charred region. The most revealing technique which provided new information, not only about the materials' structure and causes of their alteration, was surface metrology. Application of just laser confocal microscopy has shown a promising potential to enhance the understanding of complex phenomena occurring to the surface of the heat shield of space vehicle. The proposed regiment of surface examination, although applied to the space heritage artifacts, is flexible enough to be adapted in the examination of diverse cultural heritage material's surface. The derived information elucidated some aspects of a complex event contributing to a larger construct, transmission of the scientific technological concepts to the public.

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