



Development of multi-layered thermal protection system (TPS) for aerospace applications



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ABSTRACT

Multi-layered UHTC composites for use in an extreme oxidation environment were fabricated by co-sintering. The multi-layered composite system consists of three layers and was designed with (i) an outer surface with the capacity to withstand heat fluxes in excess of 25 MW m^{-2} , (ii) an intermediate layer with the ability to curtail diffusion of O_2 and (iii) a bottom layer with better performance at high temperatures. One design has MeB_2 (where $\text{Me} = \text{Zr}$ or Hf) as the outer or top layer, MeC_xO_y as the intermediate layer and MeB_2/SiC as the bottom layer. Since little is known about MeC_xO_y ceramics, a detailed study has been carried out to synthesise and characterise them focussed on controlling MeC_xO_y stoichiometry. During co-sintering, the outer MeB_2 layer thickness played a crucial role in providing a crack-free component. Indicative calculations of the residual stresses confirm that a compressive stress due to bending of the disks can stop crack growth through the MeB_2 layer when its thickness is increased to 3 mm, and that the tensile stress in the bottom layer is also reduced. The cross-section microstructure of the optimised multi-layered UHTC composite shows a crack-free seamless interface.

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1. Introduction

Improved technologies and materials capable of operating at ultra-high temperatures under the conditions of aerodynamic heating is important in development of hypersonic flight vehicles and in rocket engineering. To reduce aerodynamic drag and increase lifting force and maneuverability, hypersonic flight vehicles must have profiles with sharp leading edges [1–5]. As a result, the leading edges are subjected to extreme heat flux at the stagnation point ($>20 \text{ MW m}^{-2}$); correspondingly, the temperature of the surface can be greater than 2800 °C [6], which exceeds the exploitation temperature of current materials [1,2,4,5,7–9].

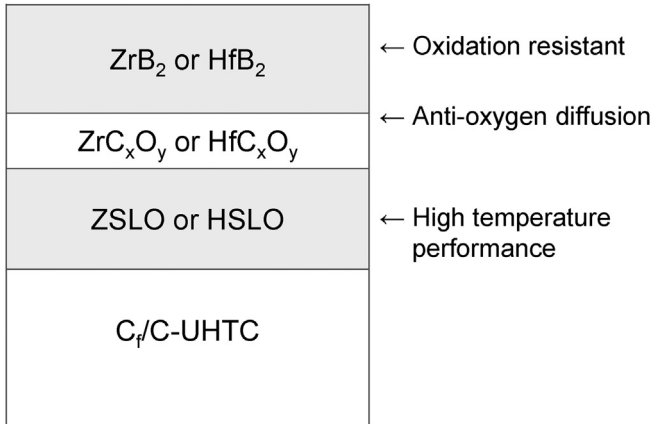
The current state-of-art in developing materials for leading edge components ranges over several areas. The first is connected with the modification of classical ceramic composites such as C_f/C and C_f/SiC by Ultra High Temperature Ceramic (UHTC) materials [10–13], by using ceramic matrices and/or protective antioxidant coatings, with the purpose to significantly increase their oxidation resistance without significant loss of the excellent thermo mechanical

properties of the base materials. However, at very high temperatures, the protective layer of SiC on C/SiC composites becomes chemically active causing rapid ablation of the material and the levelling off of the protective properties of SiC with respect to the reinforcing carbon fibres. The use of materials with a high thermal conductivity ensures a more rapid redistribution of the excessive heat and favours its removal from the line of total stagnation of the air stream in the case of components with sharp leading edges. C/C composites are attractive materials for hypersonic flight vehicles but they oxidise in air at relatively low temperatures $>500 \text{ °C}$ and hence need a thermal protection system (TPS) to survive aero-thermal heating. In both C/C and SiC based composites, development of the TPS represent a key issue for the successful re-entry of space vehicles and substantial improvements in the operational efficiency and reliability are needed, as well as cost reduction [7,14,15].

Another method of reducing the oxidation of C/C composites is to coat them with a durable multilayer Zr/Pt coating that forms ZrPt_3 as it anneals. The coating was adherent and provided oxidation resistance to the substrate. Preliminary results showed that the coating provided protection to graphite and a phenolic resin/graphite fibre composite from a 2800 °C flame. This protection is due to the oxidation resistance of the ZrPt_3 , and to the reflectivity of

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ZSLO – $ZrB_2/20$ vol.% SiC + (5–10 vol.% La_2O_3)
 HSLO – $HfB_2/20$ vol.% SiC + (5–10 vol.% La_2O_3)

Fig. 1. Conceptual diagram of a possible multi-layered UHTC component consisting of a range of potential outer protective and intermediate layers.

the Zr/Pt coating, which reduces the heat load on the substrate for short time and high-temperature applications. Biamino et al., [16] developed SiC-based TPS for C/C composites to decrease the thermal conductivity through the thickness of SiC multilayers. The first system included the presence of SiC layers containing pores after sintering. The second system involved depositing an external insulation coating made of yttria-partially stabilized zirconia (YPSZ) by means of spray combustion synthesis. The presence of porous layers in the architecture of the specimen can be effective in decreasing the thermal diffusivity. By inserting highly porous layers in the multilayer it has been possible to halve the thermal diffusivity through the thickness, that goes from 0.53 to 0.27 cm^2/s at room temperature and from 0.15 to 0.08 cm^2/s at 900 °C [16]. The presence of highly porous layers however degrades the mechanical properties, with the flexural strength being lowered from 342 to 250 MPa for a sample with halved thermal conductivity, even allowing for triggering of crack deflection mechanisms. On the other hand, the deposition of an external insulating coating of mainly dense or slightly porous layers allows reduction of the through thickness thermal diffusivity. Corral and Loehman [17] fabricated TPS using multilayers of high temperature ceramics such as ZrB_2 and SiC to protect C–C against oxidation. Their

approach combined pre-treatment and processing steps to create continuous and adherent high temperature ceramic coatings from infiltrated preceramic polymers.

An alternative approach is to make highly dense UHTC composites with high thermal conductivity, oxidation resistance and other high temperature mechanical properties such as flexural strength, hardness and fracture toughness [18–30]. This will allow efficient heat removal from the regions adjacent to the points or lines of the total flow stagnation, which will work as separate segments of a complex composite system [31,32], whose different parts will bear different types of load-thermal, oxidation, shock, shear, etc. An additional argument in favour of the selection of the design of the thermo loaded parts of the flight vehicles in the form of an assembly of several segments is that the UHTCs are brittle ceramic materials; therefore, from the viewpoint of reparability, structures are preferable that have a limited size or volume of components and, therefore, have a reduced probability of the appearance of defects, which can decrease the strength. Interfaces between the segments and the locations of junctions with other components are important from the viewpoint of construction [33]. Such segmental structures have been developed within the NASA program of the “Next generation launch technology” (NGLT) [32].

The UK research programme on the development of UHTC components for aerospace applications is expected to include C/UHTC composites on which layered structures of protective UHTC monoliths will be attached to enhance protection against oxidation and heat flux. Fig. 1 shows a multi-layered UHTC concept consisting of different layers combining various refractory ceramics with various functions, which fulfil different roles but provide better performance as a whole system. In the proposed design, the outer MeB_2 accommodates high heat flux, the intermediate layer MeO_xC_y acts as an anti-oxygen diffusion layer as it is expected to have lower oxygen mobility than MeO_2 or MeC , and the bottom substrate layer helps to increase the high temperature strength of the component. Special attention should be given to the thickness of the coating layers in order to limit cracking and the quantity of the oxidation products.

- (1) Candidate materials for an oxidation protective top layer to withstand high heat flux

The outer layer should have a high melting point exceeding 3000 °C, should serve as an oxidation protective layer and also should withstand high heat flux. Many studies have examined the oxidation behaviour of UHTCs from intermediate temperature to ultra high temperature using techniques such as furnace oxidation

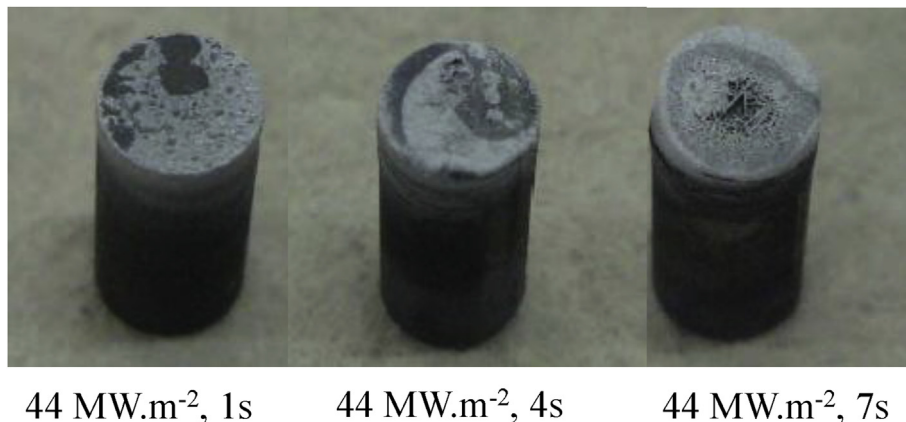


Fig. 2. Optical images of monolithic HfB_2 samples (10 mm long \times 5 mm diameter) following exposures to a heat flux of 44 MW m^{-2} for 1–7s as labelled. Some exposed to 15s not shown as it could not be detached from the graphite holder.

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