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Thermo-mechanical performance of an ablative/ceramic composite hybrid thermal protection structure for re-entry applications

K. Triantou [a], K. Mergia [a, *], S. Florez [b], B. Perez [b], J. Barcena [b], W. Rotärmel [c], G. Pinaud [d], W.P.P. Fischer [e]

[a] National Centre for Scientific Research "Demokritos", Athens Gr-15310, Greece
[b] Tecnalia Research & Innovation, Donostia-San Sebastian E-2009, Spain
[c] German Aerospace Center (DLR), 70569 Stuttgart, Germany
[d] Airbus Defense & Space, Saint Médard en Jalles F-33165, France
[e] Airbus Defense & Space, Bremen D-28199, Germany

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Hybrid thermal protection systems for aerospace applications based on ablative material (ASTERM™) and ceramic matrix composite (SICARBON™) have been investigated. The ablative material and the ceramic matrix composite were joined using graphite and zirconia–zirconium silicate based commercial high temperature adhesives. The thermo-mechanical performance of the structures was assessed from room temperature up to 900 °C. In all the joints there is a decrease of shear strength with the increase of temperature. Analysis of the fractured surfaces showed that above 150 °C the predominant mode of fracture is cohesive failure in the bonding layer. The joints fabricated with the zirconia–zirconium silicate based adhesive present the best performance and they have the potential to be used as hybrid thermal protection systems for aerospace applications in the temperature range 700–900 °C.

1. Introduction

For future space exploration the demands for thermal shielding during very high speed atmospheric re-entry go beyond the current state-of-the-art Thermal Protection Systems (TPS) [1–3]. Therefore, the development of new thermal protection materials and systems at a reasonable mass budget is absolutely essential to ensure the feasibility of the envisaged missions.

In the present study the mechanical performance at temperatures up to 900 °C of a hybrid thermal protection structure consisting of an ablative material and a ceramic matrix composite is investigated. Hybrid TPS consisting of an ablative material and a ceramic matrix composite offer a wide range of advantages [4–7]. The usage of ablators in thermal protection for re-entry vehicles is re-gaining attention as a result of a worldwide change in space mission planning strategies with entry vehicles going back to capsule designs. The advantages of hybrid TPS arise from the employment of a thinner, than usual, ablative layer able to withstand very high surface heat loads while the tough ceramic composite underneath provides structural support. Thus mass saving can be provided in combination with the enhancement of the stability of the shield shape which is an asset for the aerodynamic performance of the re-entry capsule.

The ceramic composite used in this work was the SiCARBON™ which is a C_f/SiC ceramic matrix composite. Ceramic matrix composite materials (CMCs), in particular those consisting of a SiC-based matrix reinforced with carbon fibres (C_f/SiC), are considered as the most suitable structural heat-durable materials for hot structures and TPS of launch vehicles and spacecrafts [8,9]. CMC structures are able to sustain high operational temperatures and thermo-mechanical loads, conditions that are met during the re-entry of space vehicles. Hence, carbon-fibre reinforced ceramic materials are frequently used for the highly demanding performance space missions [8].

As the ablative material to be used for the TPS has to be as light as possible and able to withstand the high entry velocities and the high impact [10] encountered during re-entry, the ablative ASTERM™ which possesses these properties was employed in this work.

In order to fabricate the hybrid ablative/ceramic structure adhesive bonding was used, as adhesive bonding is most suitable for

* Corresponding author. National Centre for Scientific Research Demokritos, 15310 Aghia Paraskevi, Athens, Greece. Tel.: +30 210 6503706.
E-mail address: kmergia@ipta.demokritos.gr (K. Mergia).

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joining dissimilar materials. There is widespread use of adhesive bonding in aerospace structures due to the fact that adhesive joints provide more uniformly distributed shear strength than mechanical joining, higher lifetime under conditions of cyclic stress and superior fatigue resistance to other joining methods. In addition adhesive bonding offers little or no damage to the adherents, no need of additional components, very little extra weight to the structure and is in general cost-effective [11–14]. Further, the lack of rigidity of the ASTERM™ material and the low temperature of its decomposition onset make imperative the use of adhesive bonding.

2. Materials

2.1. Materials for the thermal protection structure

The composite sandwiched structure was composed from two materials, a structural heat-durable ceramic matrix composite (C_f/SiC) and an ablative material.

The C_f/SiC, so-called SiCARBON™, consisting of continuous carbon fibres embedded in SiC matrix was fabricated as plates by Airbus Defence & Space, Germany [15]. The fabrication of this material is based on the so-called Polymer Infiltration Pyrolysis (PIP) process. The infiltration of the carbon fibres with a pre-ceramic polymer-based and powder-filled slurry system is performed by Liquid Polymer Infiltration (LPI) via filament winding. The thickness of the C_f/SiC material used in the current study was 4.5–4.8 mm and its properties are depicted in Table 1.

The ablator, ASTERM™, was fabricated by Airbus Defence & Space, France and some properties of the material are presented in Table 1. It consists of carbon fibres (55–80%) and phenolic resin (20–45%) [16,17]. It is manufactured by impregnating compacted graphite felt with phenolic resin, followed by a polymerisation process and final machining. The manufacturing process allows a wide range of final material densities from 0.20 to 0.55 g/cm^3 and its porosity varies between 75 and 80%. The decomposition temperature of ASTERM™ is about 235 °C. The thickness of the material used in the study was 12.2–12.5 mm. ASTERM™ is a highly anisotropic material as it is reflected in the variation of its in-plane and out-of-plane mechanical properties (Table 1).

2.2. Adhesives

The selection of the most suitable commercial adhesives for the joints was based on a) the curing temperature that has to be below the temperature at which the ablator decomposition starts (150 °C) and b) the fact that they have to withstand the very high temperatures reached during the re-entry. These two factors imposed the use of inorganic adhesives. The presence of inorganic fillers in the composition allows for potential service temperatures up to 1650 °C, depending on the nature of the filler material (graphite, zirconia–zirconium silicate, Table 2).

A previous study [7] regarding the performance of selected high temperature inorganic adhesives at room and liquid nitrogen temperature showed that Graphi-bond™ 669 and Ceramabond™ 835 adhesives are the most promising ones for the envisaged application. These two adhesives, based on graphite and zirconia–zirconium silicate, are employed in this study. They were supplied from Aremco Products, Inc. and their properties are presented in Table 2 [18].

3. Mechanical tests

3.1. Structure fabrication for the mechanical tests

Joints of C_f/SiC + ASTERM™ + C_f/SiC using the adhesives 669 Graphi-bond™ and 835 Ceramabond™ (Table 2) were fabricated for the mechanical tests using the configuration depicted schematically in Fig. 1. This fabrication structure was chosen such so as to use one CMC piece to hold the structure and the other CMC piece to apply the load. The load was applied to the CMC piece next to the thermocouple.

The CMC/ablative-material joint was fabricated first (bottom Fig. 1) and then the second joint of ablative-material/CMC (top Fig. 1) with the second piece of CMC. The dimensions of the CMC pieces were 80 × 80 × 4.6 mm^3 and those of ablative material 60 × 60 × 12 mm^3. Both materials to be joined were ultrasonically cleaned using isopropanol and acetone and dried after each cleaning step. According to our previous analysis [7], the application of thinner on the ASTERM™ surface prior to bonding strengthens the ablative material and this results in an increase of both the shear strength and the ultimate shear strain. So, prior to bonding, a silicate thinner was applied on both sides of ASTERM™ to be bonded and subsequently it was cured. The thickness of the adhesive was controlled by a jig which at same time assured the parallel alignment of the pieces to be bonded. The adhesive was applied on both surfaces to be bonded (60 × 60 mm^2). The bonded sandwich specimens were cured at room temperature for 3 h and then at 120 °C for 7 h. During curing, a load was applied on the bonded specimens. The average bonding layer thickness, after adhesive curing, was 115 μm for the graphite adhesive and 215 μm for the zirconia–zirconium silicate adhesive, whereas the average initial thickness values were 440 and 380 μm, respectively. The difference in the thickness of the bonding layer (75% for graphite and 42% for the zirconia–zirconium silicate adhesive) is due to different shrinkage levels of the two adhesives as reported in Ref. [7] (Table 3). Representatives of the fabricated sandwich specimens are shown in Fig. 2. A K type thermocouple was placed at the one CMC/ablator interface in a grove inside the ablator as shown in Fig. 1.

3.2. Procedure and infrastructure for the mechanical tests

The bonded structure specimens (Fig. 2) were thermomechanically tested at the INDUTHERM test facility (Figs. 3 and 4). The specimen was held between the two holders on the hot (Figs. 3 and 4) and on the cold side. The tests were performed under vacuum of about 2 mbar. For the tests above 700 °C one additional cooling trap with liquid nitrogen was used in the active cooling part

Table 1
Properties of the base materials.

	Density (g/cm^3)	Porosity (%)	Flexural strength (MPa)	Compressive strength (MPa)	CTE ($\times 10^{-6}$ K^{-1})
SiCARBON™	1.8	8	500	590	3/5[a]
ASTERM™ (in-plane)	0.20–0.55	75	2.28 ± 0.08	1.71 ± 0.26	0.6[b]
ASTERM™ (out-of-plane)			0.65 ± 0.06	2.84 ± 0.18	9.2 ± 7.5%[c]

[a] Parallel/perpendicular to fibre orientation (RT to 1000 °C).
[b] RT to 180 °C.
[c] 60–180 °C.