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Infra-red and vibration tests of hybrid ablative/ceramic matrix technological breadboards for earth re-entry thermal protection systems

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ABSTRACT

A new thermal protection system for atmospheric earth re-entry is proposed. This concept combines the advantages of both reusable and ablative materials to establish a new hybrid concept with advanced capabilities. The solution consists of the design and the integration of a dual shield resulting on the overlapping of an external thin ablative layer with a Ceramic Matrix Composite (CMC) thermo-structural core. This low density ablative material covers the relatively small heat peak encountered during re-entry the CMC is not able to bear. On the other hand the big advantage of the CMC based TPS is of great benefit which can deal with the high integral heat for the bigger time period of the re-entry. To verify the solution a whole testing plan is envisaged, which as part of it includes thermal shock test by infra-red heating (heating flux up to 1 MW/m^2) and vibration test under launcher conditions (Volna and Ariane 5). Sub-scale tile samples (100×100 mm^2) representative of the whole system (dual ablator/ceramic layers, insulation, stand-offs) are specifically designed, assembled and tested (including the integration of thermocouples). Both the thermal and the vibration test are analysed numerically by simulation tools using Finite Element Models. The experimental results are in good agreement with the expected calculated parameters and moreover the solution is qualified according to the specified requirements.

1. Introduction

Most of the actual commercial re-entry or space exploration missions rely on state of the art pure ablative material such as PICA (Phenolic Impregnated Carbon Ablator) [1]. On the other hand, reusable thermal protection systems (TPSs) based on the integration of ceramic matrix composites (CMCs) systems have been brought to flight maturity by extensive ground and flight testing and are now ready for application on re-entry missions typical to values of 1 MW/m^2 and up to around 2.5 MW/m^2 maximum heat loads for a single use [2].

In order to withstand more and more severe oxidative and high temperature environment due to changing strategy and under increasingly demanding economical constraints, it worths to consider innovative solutions of TPS. Responding to new mission concepts for LEO or moon re-entry a new idea has been born extending the CMC based TPS to higher heat loads for such applications by integrating ablative material on the outer surface of the CMC [3,4].

The use of CMC will offer lightness, improved mechanical properties as well as higher robustness during the entry. Besides, the new moon or interplanetary missions planned causes higher heat loads during earth re-entry than ceramic or metallic TPS can withstand, since these heat loads are characterized by a peak profile. These high heat loads of the peak profile can be borne by the ablative material. For that a comparatively thin layer of ablative material is sufficient. The large integral loads will then be overtaken by the ablative/ceramic interfacial layer [5].

The aim is to fabricate the hybrid ablative/ceramic structure, through adhesive bonding based on commercial high temperature inorganic adhesives is employed. In our previous work [6,7] appropriate adhesives were selected based on the microstructural characteristics and the thermomechanical performance of the joints.

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2. Materials and methods

2.1. Materials

The Ceramic Matrix Composite (CMC) material C_f/SiC, so-called SiCARBON™, and the ablative materials ASTERM™ and CALCARB® were used for the fabrication of the breadboard specimens. Ceramic matrix composite consisting of continuous carbon fibers embedded in SiC matrix (C_f/SiC, SiCARBON™) was fabricated as plates by Airbus Defence & Space, Germany [8]. The fabrication of this material is based on the so-called Polymer Infiltration Pyrolysis (PIP) process. The infiltration of the carbon fibers 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 2.5 mm.

ASTERM™ ablative material, which was used for the Infra-Red test specimens, was fabricated by Airbus Defence & Space, France. It consists of carbon fibers (55–80%) and phenolic resin (20–45%) [9,10]. It is manufactured by impregnating compacted graphite felt with phenolic resin, followed by a polymerisation process and final machining. The manufacturing approach allows a wide range of final material densities from 0.2 to 0.55 g/cm^3 and its decomposition point is about 235 °C. Its porosity is between 75% and 80%. The thickness of the material used in the study was 3 and 4 mm. For the vibration test specimens, the ablative material CALCARB® (Commercial carbon substrate, similar to ASTERM™ or PICA preforms) was employed. It is made up from short cut carbon fibers, interconnected in a matrix produced by the carbonization of phenolic resin. Its bulk density is 0.18 ± 0.03 g/cm^3 [11]. This non-pyrolyzing material is very similar to the rigid precursor of ASTERM™ and serves as an ideal candidate to decouple the physico-chemical phenomena of heterogeneous pyrolysis gas chemistry and carbon fiber/char ablation for fundamental ablation experiments [12]. CALCARB® was also used in previous related studies [5,6,13] in which the thermal shock performance of ablative/ceramic structure was investigated.

Table 1
Identification, composition and properties of adhesives [14].

Tradename	669 Graphi-Bond™	835 Ceramabond™
Description	Black, viscous and odorless paste	Tan, viscous and odorless paste
FILLER, Major Constituent	Graphite	ZrO_2 - $ZrSiO_4$
Ingredients	Graphite, Aluminum oxide, silicate solution	Zirconium oxide, zirconium silicate, aluminum-silicate, silicate solution
Viscosity, cP	20,000–40,000	20,000–40,000
Density, g/cm^3	1.45–1.50	2.25–2.35
Temperature Use Limit, (°C)	760	1371
CTE (x 10^{-6} K^{-1})	7.6	7.2

The selection of the most suitable adhesives for the joints was based on a) the curing temperature that has to be below the ablator decomposition temperature (approximately 150–200 °C) and b) the fact that they have to withstand very high temperatures reached during the re-entry. These factors imposed the use of inorganic adhesives. For the joining of CMC and ablative material, the commercial inorganic adhesives Ceramabond™ 835 (ZrO_2-$ZrSiO_4$-based) and Graphibond™ 669 (graphite-based) were used. These two adhesives were selected after an initial screening of six commercial inorganic adhesives based on alumina, zirconia-zirconium silicate and graphite having high and low viscosities.

The screening was performed employing pull off tests and microstructure investigation of the interfaces. Furthermore, this selection was based on the results of the previous studies [5,6,13] employing thermal shock tests as well as mechanical tests at liquid nitrogen, room and elevated temperatures. The adhesives were supplied from Aremco Products, Inc. Their properties and composition are presented in Table 1.

2.2. Fabrication and design requirements of specimens for Infra-red tests

Breadboards were designed and constructed for the Infra-Red (IR) tests (Fig. 1), so as to reproduce as close as possible the temperature profile of the ablator/CMC dual layer interface and check the thermal behaviour of the hybrid tile. Four specimens were fabricated with graphite-based and ZrO_2-$ZrSiO_4$-based adhesives for the CMC-ablative material joining (two specimens for each adhesive). Depending on the used adhesive, different thickness of ablative material was used, 4 mm for graphite adhesive and 3 mm for ZrO_2-$ZrSiO_4$ adhesive in order to tune the maximum interfacial temperature to the maximum service temperature of each adhesive. As shown in Fig. 1, the IR specimen is fully representative of an operational tile and is composed of:

- ASTERM™ ablative material (thickness 3 or 4 mm)
- Graphite-based or ZrO_2-$ZrSiO_4$-based adhesive (0.2 mm)
- SiCARBON™ CMC material (2.5 mm)
- Internal Flexible Insulation (IFI) (40 mm)
- Four stand-offs made of Ti6Al4V grade 5 alloy
- Aluminum plate (2 mm)

The fabrication procedure includes the following steps:

- Brazing of the stand-offs to the CMC
- Placement of the thermocouples
- Placement of insulator material
- Joining of ablator to CMC

The first step for the fabrication of the specimens was the joining of four “U” shape Ti6Al4V stand-offs at the corners of the CMC plate using the technology developed in [15]. Due to the temperature limit of the Ti6Al4V, this design and material choice is particularly suitable for the breadboards and for the final component a material trade-off

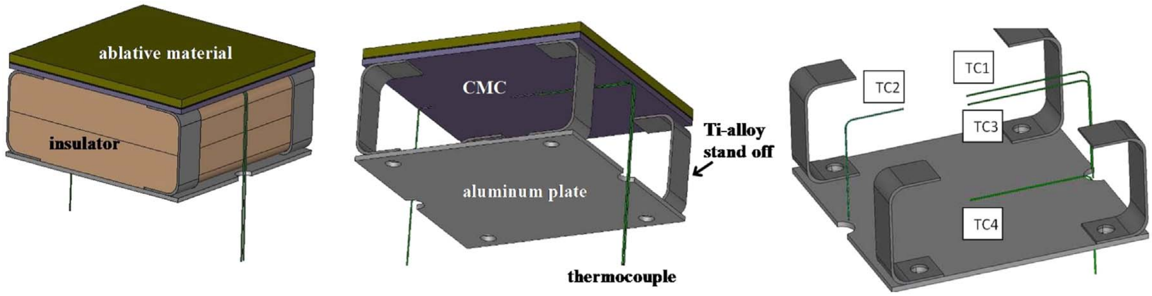

Fig. 1. IR specimen definition concept and its instrumentation with thermocouples (TC).