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Development of an Italian Technology for CMC Control Surface for Re-entry Applications

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Abstract 100 words

Re-entry systems are subject to the harsh environment during hypersonic atmospheric re-entry phase. In order to withstand the high aerothermal loads, thermal protections systems are necessary to protect the vehicle and hot structures are mandatory for aerodynamic control surfaces. Thermo-structural challenges are quite severe. Indeed, large thermal gradients are induced by the outer surface of the vehicle and the internal environment, including cold structure and avionics as well as at the connections between hot control surfaces and the much cooler actuators inside the vehicle.

C/SiC is widely selected as the best option for TPS and hot structure control surface providing the lowest weight and thinnest cross-section, minimal thermal expansion mismatch problems, and a good thermal margin. Different processes are commonly used: Chemical Vapour Infiltration (CVI), Polymer Infiltration and Pyrolysis (PIP) and Liquid Silicon Infiltration (LSI).

CIRA has recently set up a research and development project with Italian industrial partners, with the aim of working on design and development of hot structures based on ceramic matrix composites (CMC) technology (C/SiC). The proposed technology is based on LSI process used by team members for high performance braking systems furnished to prestigious automotive marques. The advantages of the LSI w.r.t CVI processes are shorter manufacturing times, and the use of low cost raw materials, leading to the most cost efficient CMC. LSI C/C-SiC is characterized by medium tensile strength and high shear strength compared to materials derived from CVI and PIP.

Despite some commonalities between brakes and thermal protection systems environments, different thermo-mechanical requirements ask for an ad hoc development of the process. In this respect, the team is assessing the following aspects:

- CFRP "green" manufacturing with long C-fibres or C-fabrics instead of short fibres preforms as normally used for braking devices;
- Innovative manufacturing techniques;
- Additional surface treatment for oxidation protection in low-pressure environment.

The development and realization of CMC hot structures requires significant know-how in structural analysis and design, material processing, manufacturing and qualification of components. The development team thanks to their own heritage covers all these aspects.

The present paper describes all the activities on going for the development of an Italian technology for CMC control surfaces with the aim to demonstrate a sufficient technology readiness level to be considered for SPACE RIDER, the next space system of ESA with re-entry capability for in-orbit experimentation.

1. Introduction

CIRA has recently set up a research and development project with Italian industrial partners, with the aim of working on design and development of hot structures based on ceramic matrix composites (CMC) technology (C/SiC), code-named SHS-CMC.

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SHS-CMC project focuses on the application of the CMC technology on the control surface of a re-entry vehicle, which is characterized by severe thermal structural challenges such as large thermal gradients between cold and hot parts of the structure. Moreover, the brittleness of monolithic ceramic is unacceptable for loaded structures such as control surfaces: the key to the toughness is the presence of the fibers. Nevertheless unlike traditional CFRPs, the interface between the fibers and the matrix has to be weak in order to allow toughness and reliability by energy dissipating mechanisms such as crack deflection, crack splitting and crack stopping [1].
Different processes are commonly used to manufacture C/SiC: Chemical Vapour Infiltration (CVI), Polymer Infiltration and Pyrolysis (PIP) and Liquid Silicon Infiltration (LSI).
The proposed technology is based on Liquid Silicon Infiltration (LSI, Figure 1), used by team members for high performance braking system furnished to prestigious automotive marques. The advantages of the LSI w.r.t CVI processes are shorter manufacturing times, and the use of low cost raw materials, leading to the most cost efficient CMC. LSI C/C-SiC is characterized by medium tensile strength and high shear strength compared to materials derived from CVI and PIP [2].

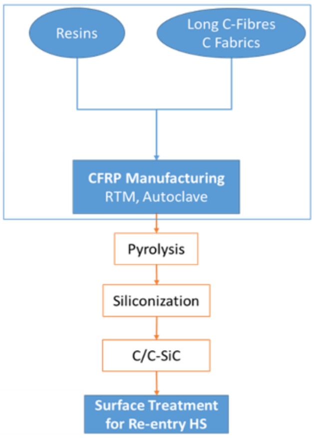

Figure 1: LSI manufacturing process

The final objective of SHS-CMC project is to reach TRL 5/6, to be spent in more challenging projects such as ESA-SPACE RIDER [3]. The challenge is to succeed in manufacturing a long-fiber LSI component, having a simplified geometry with respect to ESA-SPACE RIDER flap, but representative as regards manufacturing issues. Moreover, the component must have an acceptable level of strength at very high temperatures (up to 1700°C) and an anti-oxidation coating, which prevents it to be burnt away during the atmospheric re-entry.
The work logic of the project is depicted in the left of Figure 2, where red boxes indicate the material and process set-up phases, propaedeutic to reach the target TRL.

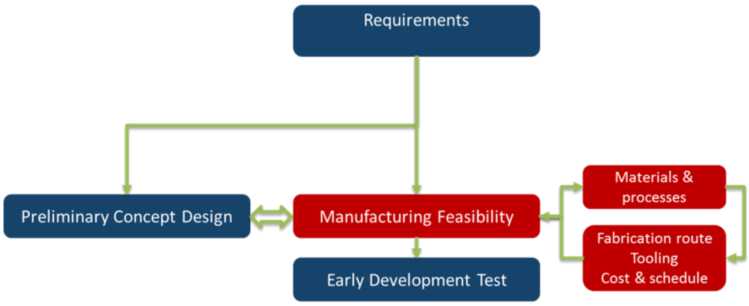

Figure 2: Work logic of the project

To accomplish the final task of reaching a proper level of maturity, the different technical activities are divided into the three main orthodox areas of Design, Manufacturing and Testing (Figure 3). So far, CIRA is concluding the first part of the three phases, highlighted by the dashed line of Figure 3, and presented herein.

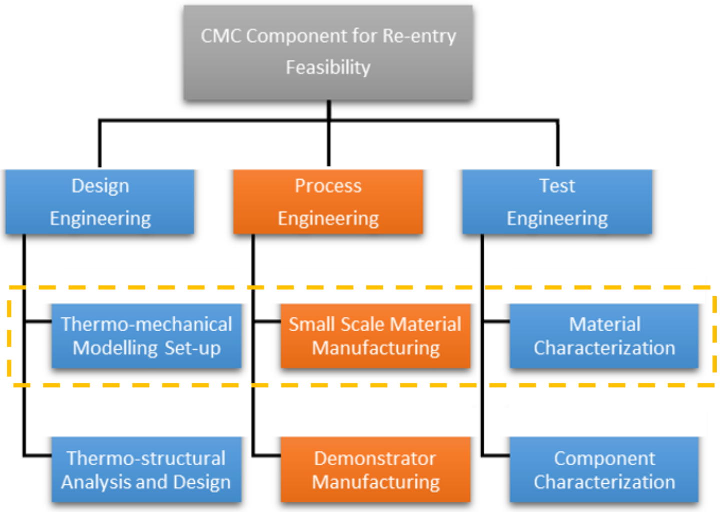

Figure 3: Project tasks

The first question to be answered at the very beginning of the SHS-CMC project, was the demonstrator layout concept to be manufactured. Since Italian research community has low experience in such materials for re-entry applications, the unnecessary geometric complication characterizing the ESA-SPACE RIDER body flap has been neglected. CIRA heritage in ESA-EXPERT project gave the answer: the ESA-EXPERT flap geometry [5] has the right degree of complexity from the manufacturing process viewpoint. It has dimensions, thickness and structural features (curvature fillets, undercuts, stringers, holes) which will be present on SPACE RIDER flap too.
The demonstrator characterization will consist in tests in relevant environment, by means of CIRA SCIROCCO plasma wind tunnel, aimed at demonstrating the ability of the material of keeping functional and structural integrity during one or more load cycles (re-entry environment of ESA SPACE RIDER vehicle).
In order to be able to demonstrate all of above, small scale manufacturing, preliminary characterization of material and set-up of numerical models are fundamental ingredients.

2. Process engineering

For the manufacturing technique of the long-fiber CFRP preform of the demonstrator, hand-layup and vacuum bagging in autoclave has been deemed the simplest, cheapest and most robust solution, especially for this particular phase, in which mould design has yet to be assessed and adjustments must be considered to accommodate geometry contraction during pyrolysis.
The choice of the material has followed the same philosophy: a phenolic pre-impregnated fabric is the most manageable with respect to unidirectional fibres, although mechanical properties are lower. Due to economic aspects, in this preliminary phase of the LSI process set-up, high tenacity (HT) carbon fibres have been preferred (tensile modulus of about 240 GPa), postponing the improvement of mechanical properties by means of intermediate modulus fibres (IM, tensile modulus of about 290 GPa) when the whole LSI process will be completely tuned.
Different pre-impregnated fabric for CFRP preform manufacturing are under investigation. The differences lie in the resins, which anyway are thermosetting phenolic matrices, and in the weave style of the fabric. Resin weight ratio is in the range 40-42%.
Different compacting and autoclave routes, during the CFRP phase, are under investigation, since they have a crucial effect on the subsequent phase of pyrolysis. In fact, during this latter phase the thermal stresses in the CFRP can cause severe delamination, thus not allowing the silicon infiltration [6]. In particular, it has been found that one of the most critical parameter is the compacting pressure. Excessive compacting pressures have led to severe delamination and sometimes (depending also on the base material) to almost “explosive” phenomena during the pyrolysis cycle. The optimum compacting pressure, i.e. the maximum pressure which do not show delamination problems during pyrolysis, depends also on base material. Figure 4 shows one of the panels which delaminated during pyrolysis.

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Figure 4: Severe delamination occurred during pyrolysis

At the same time compacting pressure cannot be too low otherwise mechanical properties of the final CMC will be affected.
It has been also found that a heat treatment above the curing temperature of the CFRP after the autoclave curing is beneficial both to the subsequent pyrolysis and infiltration phases and to the mechanical properties, with an increase of about 15% in flexural strength with respect to not-heat-treated specimens. Images of two CMC specimens are shown in Figure 5. The heat treatment relaxes unavoidable internal stresses occurred during autoclave curing and begins to crack the CFRP matrix, thus preparing it to pyrolysis [7].

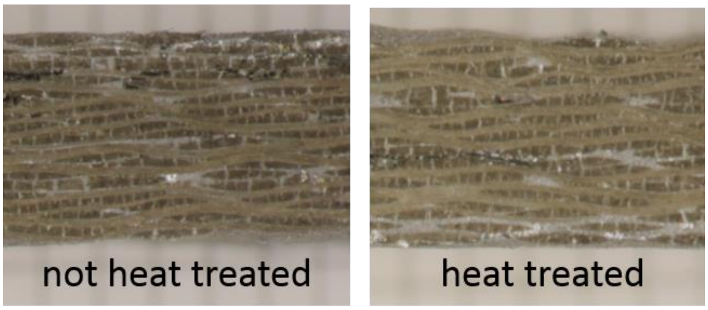

Figure 5: Two CMC specimens coming from heat treated CFRP and not

Mass loss during pyrolysis is 18% on average, whereas thickness contraction is about 10%. This not negligible value indicates that the mould design (CFRP phase) must take into account the geometry contraction in order to preserve final CMC geometry. Moreover proper kiln furniture have to be designed and installed in the pyrolysis oven in order to prevent distortions. At present, massive technical activities are running to cover all the above aspects.

2.1 Coating process

C/C-SiC ceramic composite needs an anti-oxidation coating in order to resist the re-entry harsh environment. Typically, it is made of a SiC layer. In the present project different chemical vapour deposition runs for the deposition of coating, by varying maximum temperature (temperature above 1300°C), time of exposure, pressure, cooling gas and duration have been set-up, in order to find the best process parameters as to guarantee a suitable coating thickness.
The deposition process scheme is shown in Figure 6. The samples have been exposed both directly and indirectly to Si vapours.

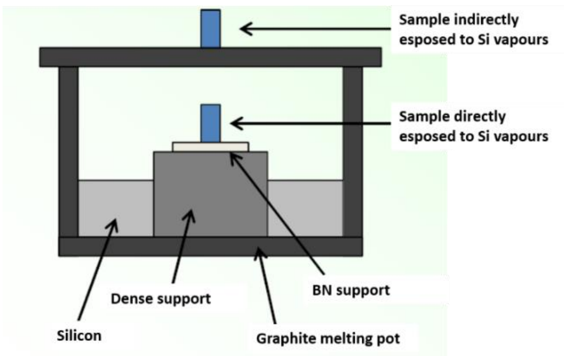

Figure 6: Coating deposition scheme

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The process has proven to be effective in laying an effective coating thickness. The quality of the substrate material influences the presence of cracks in the coating layer. These cracks are possible trigger sites for oxidation, thus have to be avoided. To date, optimization of the process is work in progress, along with coating characterization (explained later in the paper).
Figure 7 and Figure 8 show some surface and cross section SEM images of the coated samples.

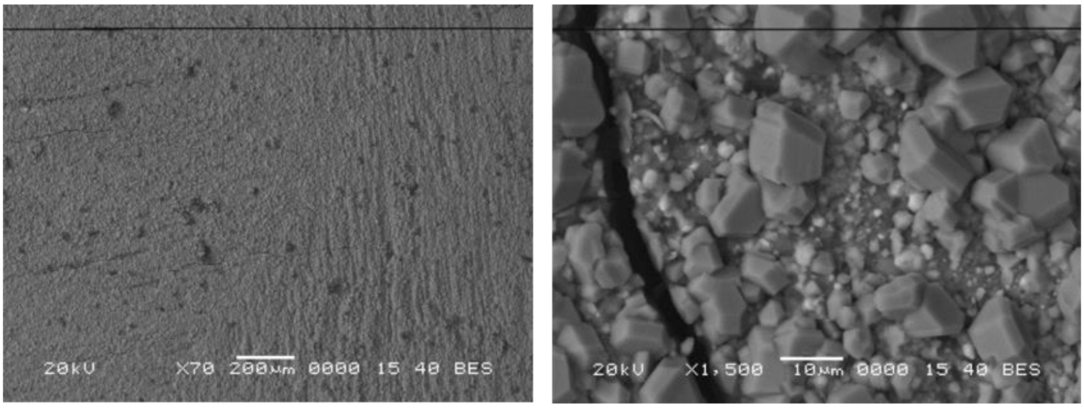

Figure 7: SEM of coated surface

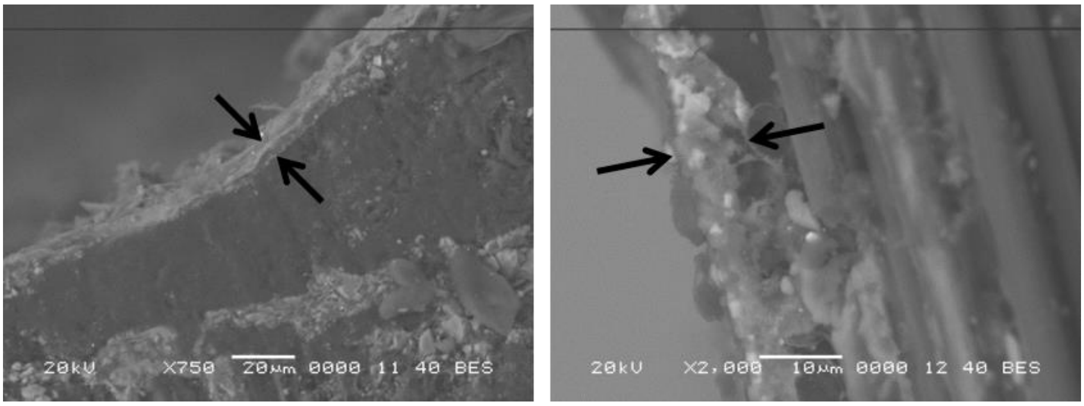

Figure 8: SEM coating cross section

3. Test engineering

Manufacturing and testing activities are in closed loop. To date, three types of material characterizations have been made: a) mechanical preliminary testing at ambient temperature (bending and tensile tests); b) thermo-physical characterization; c) plasma arc-jet testing of the coating.

3.1 Mechanical characterization

Mechanical preliminary testing at ambient temperature (bending and tensile tests on coupons, Figure 9 and Figure 10) have been carried out to understand if minimal strength and ductile fracture characterize the production batches. ASTM standards have been adopted, sometimes customized if necessary ([8], [9]). Mechanical properties are in line with literature data on LSI CMC [1].

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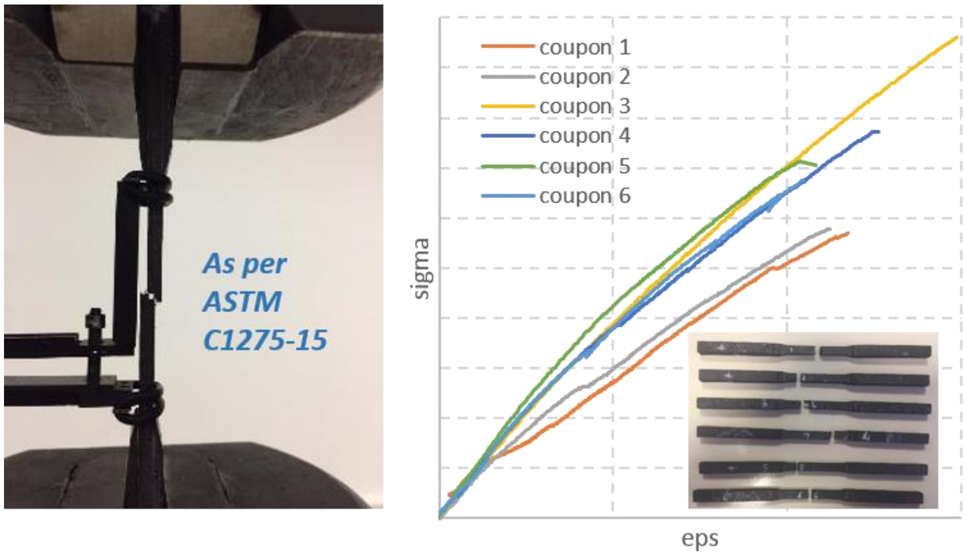

Figure 9: Tensile test

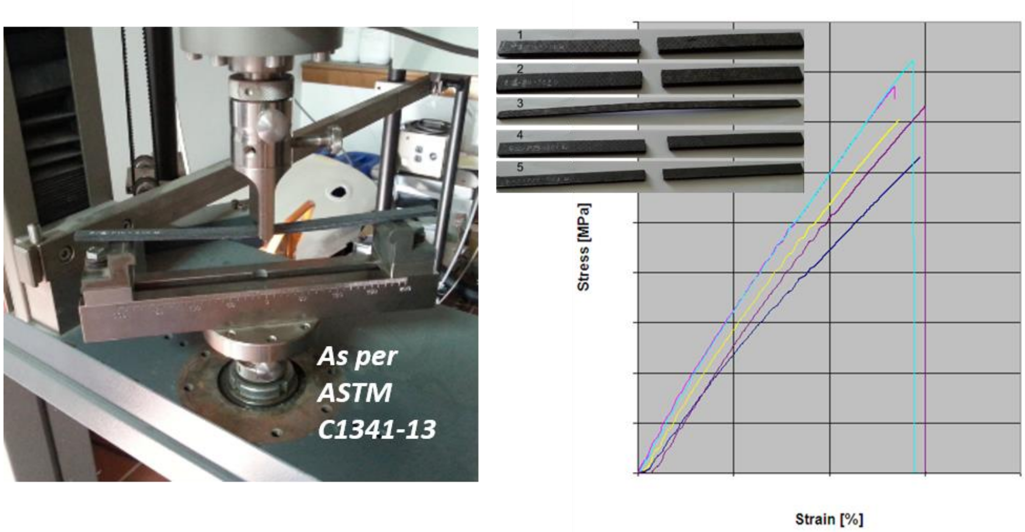

Figure 10: 3-point bending test

3.2 Thermal characterization

Thermo-physical properties of material such as specific heat, thermal diffusivity and thermal conductivity through the thickness have been determined by means of LFA according to ASTM E1461-01.. The values are in line with literature LSI CMC data (Figure 11).

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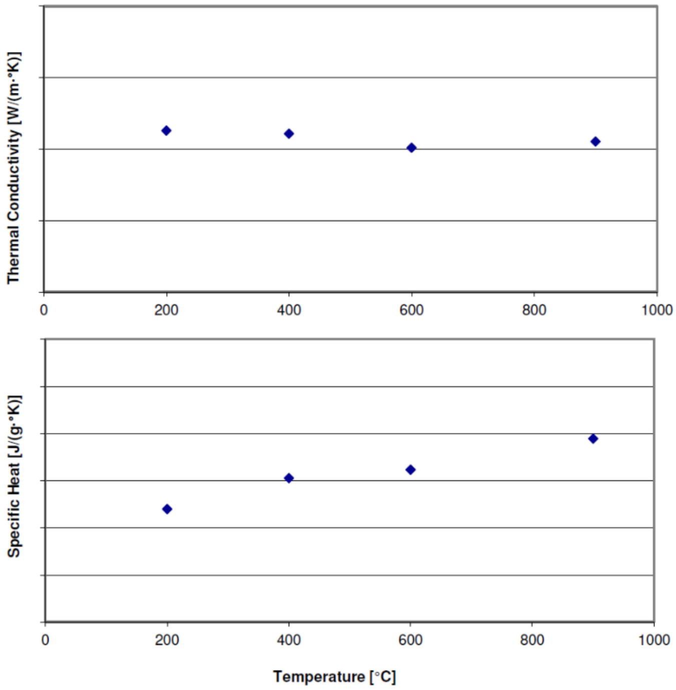

Figure 11: Thermal properties

As regards coating characterization, extensive experimental campaign has been carried out on the first production batch. Relevant environmental testing has been performed in two different plasma arc-jet facilities: SPES at the University of Naples (Figure 12, Figure 13) for lower enthalpy and GHIBLI at CIRA for higher enthalpy. Coated and uncoated flat disk samples have been subjected to heat fluxes, which made them reaching maximum temperature of the order of 1600°C for a test duration up to 300 s, with absence of active oxidation on coated samples, Figure 14 and Figure 15.

Figure 12: UNINA SPES arc-jet test set-up

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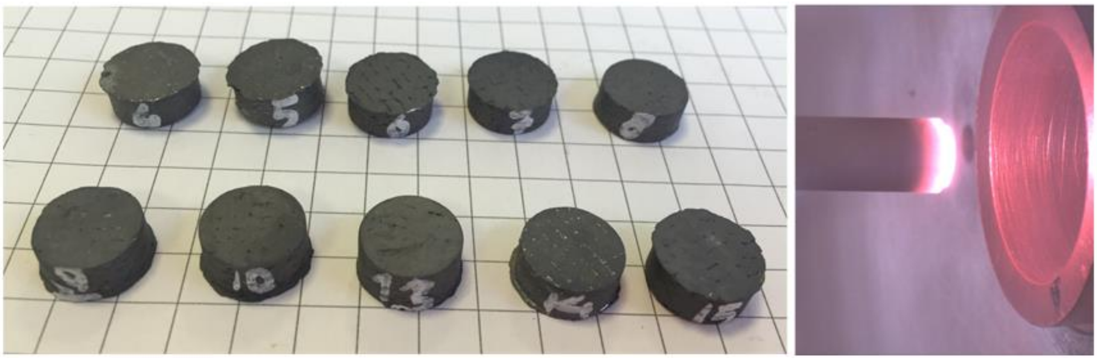

Figure 13: First batch of specimen and test

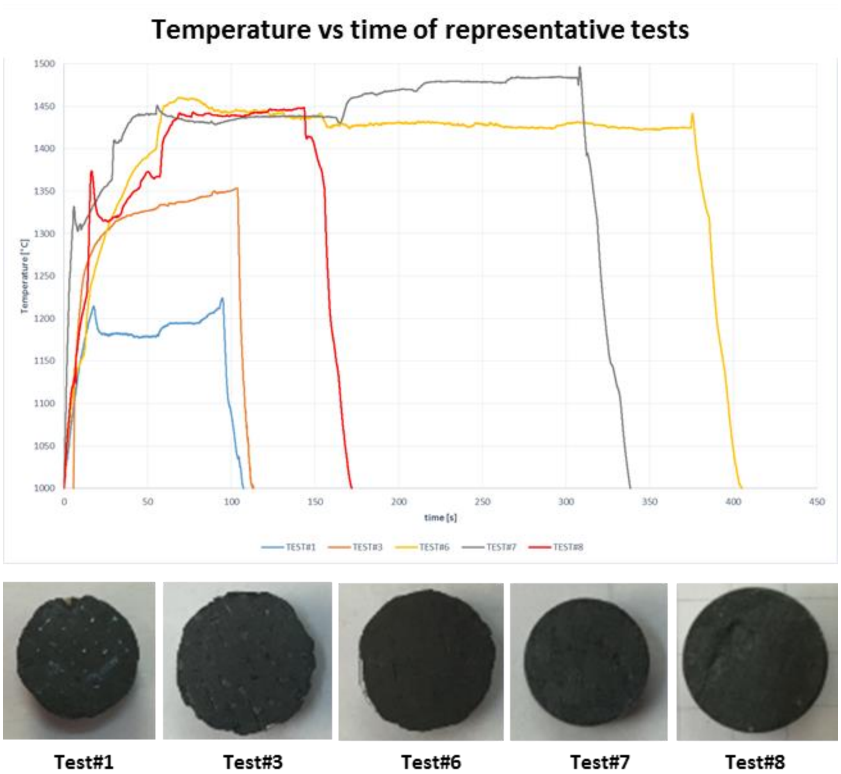

Figure 14: Temperature versus time of some representative tests

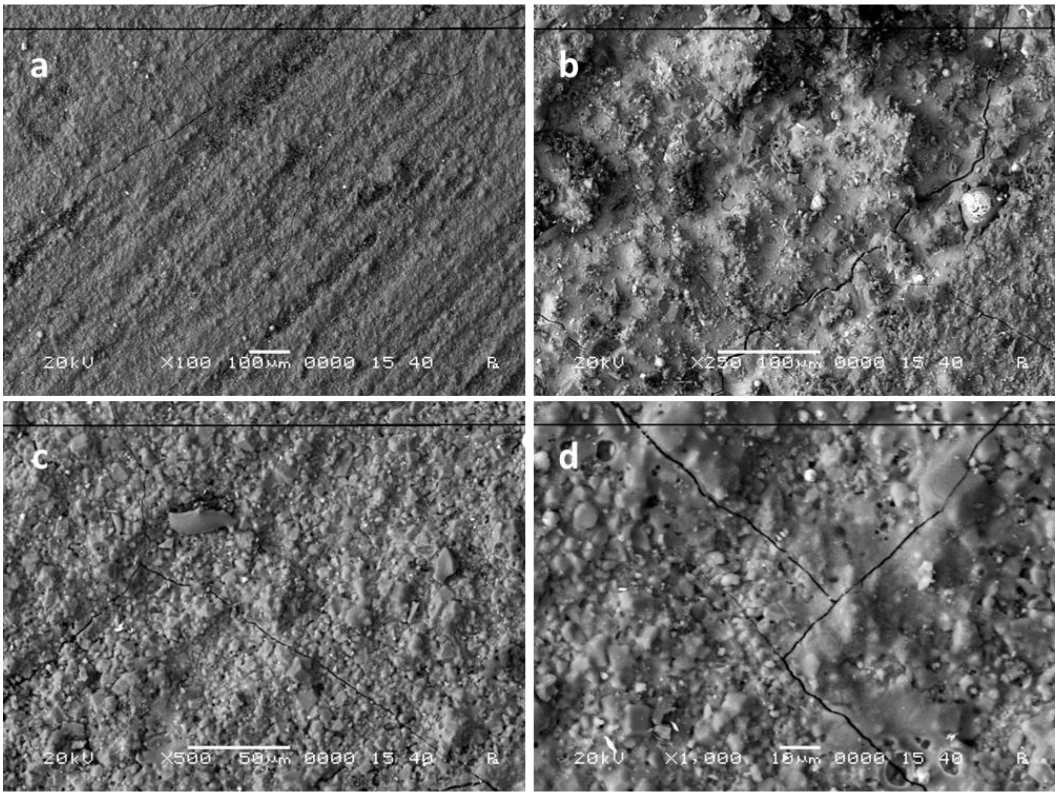

Figure 15: SEM images at different magnifications (x100 – x250 – x500 – x1000) of Test#7 sample

4. Design engineering

As explained in the preceding, the ESA-EXPERT flap structural concept has been chosen for the demonstrator to be tested in CIRA SCIROCCO plasma wind tunnel **Errore. L'origine riferimento non è stata trovata.**, since it has the right degree of complexity from the manufacturing process viewpoint. The aim of the test is to demonstrate the ability of the material of keeping functional and structural integrity during one or more load cycles (re-entry environment of ESA SPACE RIDER vehicle).
The technology demonstrator will be mounted on a model holder already available at CIRA, characterized by a cooled copper leading edge, Figure 16.

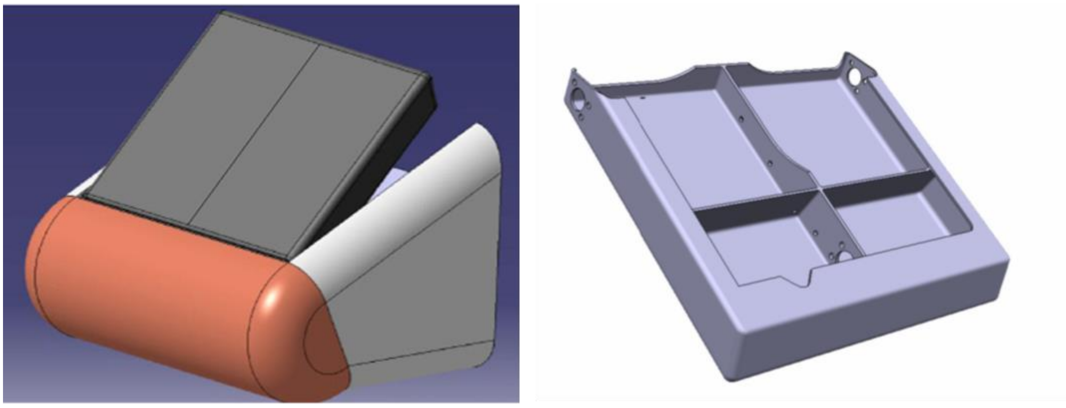

Figure 16: Control surface demonstrator concept

4.1 Thermo-mechanical models set-up and validation

A first thermal and thermo-mechanical model, hence including all the relevant heat transfer phenomena, was set up in [10] on a simplified geometry, Figure 17. The FE model was characterized by the use of quadrilateral plate elements (2D elements).

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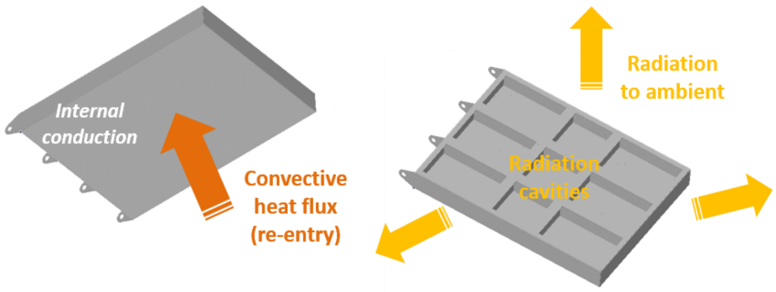

Figure 17: First thermal model on simplified geometry

Later in the project it was decided to move to a 3D-elements based thermal model, in order to catch the significant temperature gradients through the thickness, which characterize the thin-walled surfaces subjected to dramatically different loading conditions at their front and back sides. An alternative to this second modelling strategy was to investigate the feasibility of a mixed 2-D/3-D model, but it was deemed simpler and cheaper, from a modelling effort viewpoint, to continue with a totally 3-D elements model, also because of the fact that the same model will be used for thermo-structural calculation.
The ESA-EXPERT geometry has then been modelled with ANSYS Workbench. Material data on Keraman® C/SiC (MT Aerospace) have been taken from literature [12]. The demonstrator will have a geometry which is very similar to ESA-EXPERT flap, unless small thickness modifications arising from more challenging test loads. Figure 18 depicts the finite element mesh, which comprises also the cavity behind the flap, modelled with a plate.

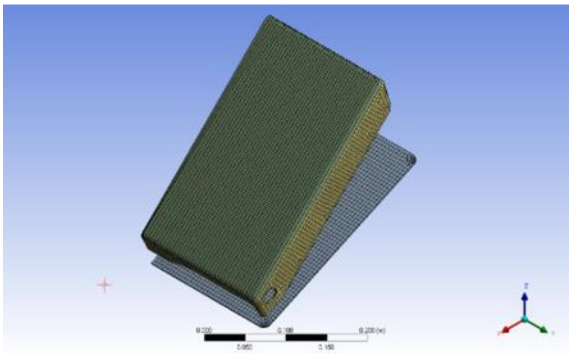

Figure 18: Finite Element Model 3D Mesh (half model)

The validation of the model has been made by means of results of SCIROCCO PWT test campaign on EXPERT Open Flap PL6/8 [13]. The test model was instrumented with the same flight qualified sensors (thermocouples and pressure) and measuring systems (IR thermo-camera from the inner side of the cavity under the flap collecting the back radiated flap energy). The correlation with FE model has been searched with thermocouples data.
Figure 19 shows the position of thermocouples botn on test article and on numerical model.

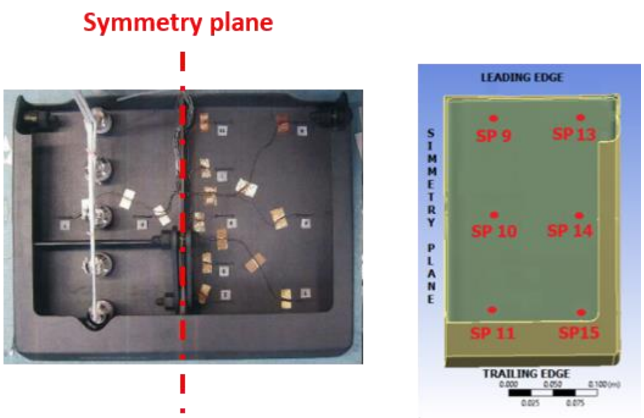

Figure 19: Thermocouples position on test article and on numerical model (half model)

Two test conditions (repeated two times each) have been used for experimental-numerical correlation. They are reported in Table 1.

Table 1: SCIROCCO PWT test conditions on ESA-EXPERT flap

Test #	H_0 [MJ/kg]	P_0 [bar]	arc heater air flow [kg/s]	arc heater Power [MW]	Flap qre [kW/m2]	Flap Tre [K]	Test Duration [s]
1-2	13,7	7,0	1,30	38	345	1396	110
3-4	11,3	6,5	1,30	28	270	1313	140

The numerical heat flux distribution, inputted on the thermal FE model, has been derived from stationary 3D CFD simulations.
Figure 20 shows respectively the thermal evolution at the thermocouples position on the rear flap surfaces for the two different test conditions. There is a very satisfying agreement in terms of global tendencies and maximum values for thermocouples with exception of SP13 and partly of SP9, probably due to a separation bubble on the leading edge, which is not caught correctly by CFD simulations. For this reasons further CFD analysis will be performed to improve the coupling between aerothermodynamics and thermo-mechanical model. Similar comments can be given for run 3. In this case numerical values tend to slightly underestimate the experimental values. Anyway, maximum differences are about 6 %. This is a very comfortable value indicating that the numerical thermal model is validated and can be considered as predictive.

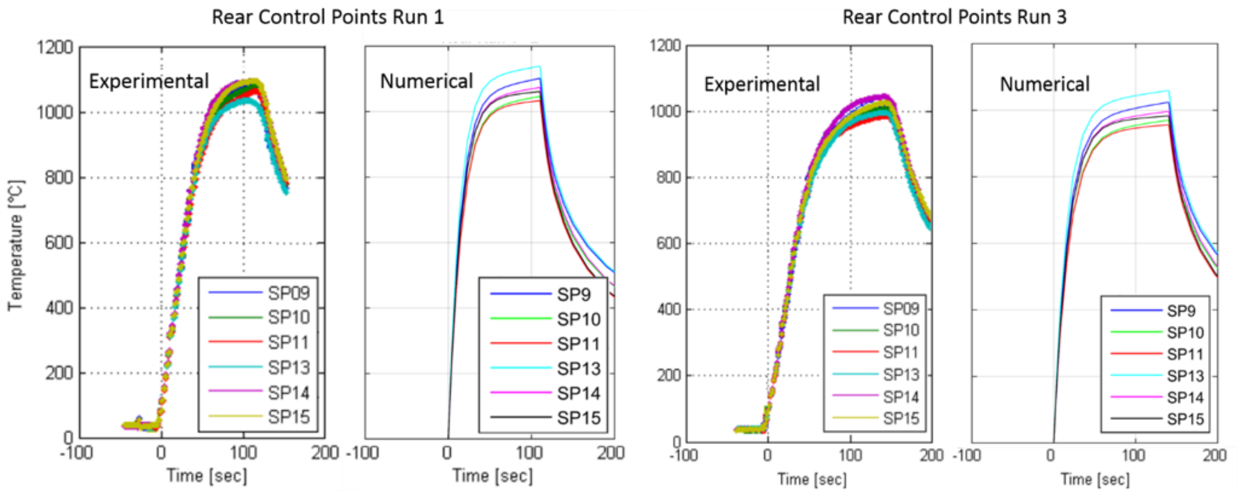

Figure 20: Experimental and numerical temperature variation at TC position for run 1(left) and run 3 (right)

4. Conclusions and perspectives

In the present paper the main ongoing activities on the development of a research and development project, with the aim of working on design and development of hot structures based on ceramic matrix composites (CMC) technology. The technology is based on LSI of pyrolyzed CFRP preforms, obtained by vacuum bagging of fabric layers. The technology demonstrator will be representative of the body flap of a re-entry vehicle, in particular due to CIRA heritage on ESA-EXPERT, it will have - unless minor modifications – the same structural layout and dimensions of EXPERT flap.
So far, different type of activities have been carried out, covering the three main areas of design, manufacturing, and testing.
From the process side, two aspects are covered: the first one is the whole process aimed at obtaining the CMC, the second one is related to the deposition of a proper thickness of anti-oxidation layer. As regards the first aspect, it has been experienced that a great impact on the final result comes from CFRP phase, both from base material and from curing conditions, especially autoclave compacting pressures. Moreover, the significant thickness contraction during pyrolysis has a huge impact on the mould design of the CFRP preform, and this is a work in progress activity together with complex samples manufacturing. Complex shapes are difficult to manage during pyrolysis, and sometimes require “non-orthodox” manufacturing expedients, such as cutting the carbon fibres prior to hand layup [6]. Future activities are aimed at assessing more and more complex geometries until demonstrator manufacturing.

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The production batches have been characterized from mechanical and thermal points of view. Tensile and bending tests at ambient temperature show performances in line with other LSI CMCs from literature. Further improvements are necessary to increase mechanical properties, especially tensile strength, by acting on minimization of flaws in the CMC. Moreover, when target mechanical properties are obtained, a full test campaign will be performed also to cover other aspect such as high temperature testing.
As regards the second aspect of the coating deposition, the main parameters of the chemical vapour deposition process have been assessed, showing that also the substrate quality influences the crack distribution on the coating. The process has proven to be effective in laying an effective coating thickness. Further runs are envisaged by changing the quality of the substrate and tuning of process parameters. Extensive experimental campaign have been carried out on the first production batch, to test the coating under relevant environmental conditions. Coated and uncoated flat disk samples have been subjected to heat fluxes, which made them reaching maximum temperature of the order of 1600°C for a test duration up to 300 s, with absence of active oxidation on coated samples. Further experimental campaign on samples will be performed when the coating process and the CMC process will be assessed, until final characterization of the technological demonstrator in SCIROCCO PWT.
Finally, as concerns the design of the demonstrator, a propaedeutic activity aimed at setting up validated finite element models for thermal and thermo-mechanical analyses has been performed thanks to CIRA heritage on ESA-EXPERT project. In particular, experimental validation has been obtained by means of temperature measured during the test campaign of EXPERT flap in SCIROCCO PWT. Future activities on this side will be aimed at designing the final experiment on the demonstrator, by evaluating the design heat fluxes which have to be reproduced in the plasma wind tunnel. The design heat fluxes will be more demanding of those tested in the past on expert flap, because will be representative of more challenging missions such as SPACE RIDER.

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