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Numerical and experimental approach for the design of CMC and UHTCMC reusable structures: results of AM3aC2A project

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Abstract

The development of reusable space vehicles is a fundamental goal for reducing the cost of space access. In the last decades, Ceramic Matrix Composites (CMC) and Ultra High Temperature Ceramic Ceramic Matrix Composites (UTHCMC) emerged as particularly promising candidates for the development of lightweight hot-structures capable of performing structural roles in harsh environments with temperatures in the 1000°C÷2000°C range and in the presence of oxidizing agents. The primary structure of re-entry and hypersonic vehicles, and structural parts of space propulsion systems are among the main application scenarios considered in the studies carried out on such materials. However, a consolidated design approach for the aforementioned structural applications has not yet been identified. The experience gained in the aeronautic field for the application of composite materials suggests the adoption of a damage tolerance design philosophy, based on the assessment of the structural capability to withstand the operational loads even in presence of damages difficult to be detected. The project AM3aC2A (Multi-scale Approach for Material Models of CMC and UHTMC in reusable Component for Space) funded by Italian Space Agency was dedicated to developing numerical tools and experimental protocols for damage tolerant CMC and UHTCMC hot structures. The results of the project that will be presented include the characterization of the material beyond the elastic range anda series of material models developed at the meso-scale level, which make possible the prediction of the mechanical properties of structural details with complex geometries and possible defects. For the UHTCMC material, the fundamental role of thermal residual stress originated during manufacturing was pointed out and modelled. The tolerance to impact damage and the detectability of impact-induced damages was investigated experimentally. Finally, the residual strength after exposure to relevant environment in Plasma Wind Tunnel was assessed. These results strongly indicate the possibility of developing damage tolerant CMC and UHTCMC structures and provide the tools for effective design procedures for the development of reusable structural elements for space applications.

Keywords: Ceramic Matrix Composites, Ultra High Temperature Ceramic Matrix Composites, Material Characterization, Damage Mechanics, Plasma Wind Tunnel

1. Introduction

The development of a new generation of vehicles for a cost-effective access to space is fostering the research for engineering solutions that can maximize the reusability of the vehicles, and the maintenance effort required to guarantee safety in each mission.

A fundamental issue is represented by the extreme operational conditions experienced by the structure of re-entry vehicles as well as by the structural part of propulsive systems, characterized by complex loading, high temperature, and oxidizing environment. For such applications Ceramic Matrix Composites (CMC) and Ultra High Temperature Matrix Composites (UHTCMC) are promising candidates to build thermal shields that can combine structural roles and thermal protection functions [1-3].

CMC based on SiC matrix may have operational temperature beyond 1500 °C [4] and presents remarkable stiffness-to-weight ratios and good strength-to-weight ratios that depend on the manufacturing procedure adopted. Their cost also depends on the technology used for the production. Although very high properties can be obtained by processes such Chemical Vapor Infiltration (CVI) and Polymer Infiltration and Pyrolysis (PIP), a particularly appealing trade-off between cost and properties can be achieved by adopting Liquid Silicon Infiltration (LSI) [5].

Ultra-High Temperature Ceramic Matrix Composites are obtained by combining an ultra refractory ceramic, typically composed of borides and carbides of early transition metals. In particular, ZrB_2 - SiC matrix can be reinforced by slurry impregnation and sintering, as shown in [3,6], obtaining materials characterized by very high stiffness and noticeable toughness that can operate at temperatures close to 2000 °C.

The effective use of these types of materials in space vehicles requires the development of experimental characterization protocols and of numerical approaches to support the engineers in the design of those structural parts in reusable space vehicles exposed to the most extreme operational conditions. For such reason, a series of activities have been performed within a project aimed at reducing the gap between the result achieved by material scientists and the engineering application in real-world, cost-effective, and highly performing structures for a new generation of space vehicles.

In this paper, the results of AM3aC2A project (Multi-scale Approach for Material Models of CMC and UHTMC in reusable Component for Space funded by Italian Space Agency) are presented. The project was organized to improve the comprehension of material behaviour and to develop numerical and experimental protocols considering three fundamental areas: (i) the assessment, the evaluation, and the improvement of technological processes, (ii) an integrated experimental and numerical approach for the characterization of the materials, the analysis, and the prediction of the structural response, (iii) the development of criteria for the design and damage tolerant structures, with a focus on the exposition of relevant environment conditions.

1. Assessment of material responses and improvement of technological processes

The basic characterization of the materials consisted of a series of tensile, fracture, and bending tests aimed at evaluating the development of damages, the consequent non-linear response, and the strength of the materials produced by applying state-of-the-art and improved technological processes.

The experimental campaigns clarified some of the fundamental aspects of material responses. For the LSI-produced C/SiC CMC, produced by Petroceramics S.p.A., the tests pointed out the progressive degradation of matrix-dominated properties under the action of increasing stress. The results presented in Fig. 1, summarize the response of four lay-ups of carbon fabric plies reinforced by SiC matrices: *T0* ($[0]_{20}$ lamination sequence), *T45* ($[45/-45]_{10s}$ lamination sequence), *T30* ($[30/-30]_{10s}$ lamination sequence), and a quasi-isotropic lamination sequence (TQI with a $[0/45/90/-45]_{5s}$ layup). The increment of the non-linearity of the response, as the fibres are increasingly oriented at an off-axis angle with respect to the applied load, is originated by the anelastic phenomena in the ceramic matrix.

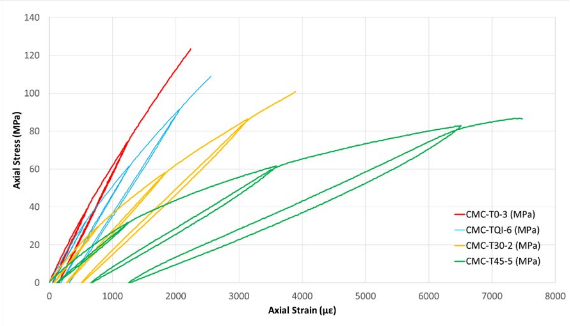

Fig. 1. Non-linear responses in the tensile tests of cross-ply, angle-ply, and quasi-isotropic laminates of LSI-C/SiC CMC

It can also be observed that the tests were performed by applying loading-unloading cycles to analyse the state of the material after the application of an increasing strain. The procedure was exploited to investigate the response of a protective SiC coating layer, which was obtained by using a special technological process to protect the laminates from the oxidation of carbon fibres that can occur at high temperatures. The results indicated that such coating does not experience degradation after the application of significant deformation in the tests, as exemplified in Fig. 2, referred to the surface analysis of a laminate in the pristine state and after the application of a strain corresponding to 0.66 times the failure stress.

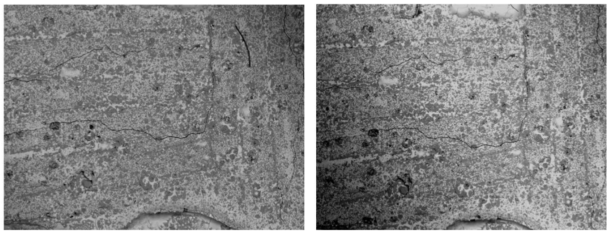

Fig. 2. Comparison of the protected surface of an LSI-C/SiC CMC laminate in the pristine state and after the application of mechanical strains

Bending tests confirmed the importance of the matrix damage in the failure of the material, which is characterized by a significant bending-to-tensile strength ratio, with a bending strength that can exceed the tensile one of a factor higher than 1.5.

The development of delamination in these CMC laminates was investigated through an extensive campaign based on Double Cantilever Beams (DCB) tests shown in Fig. 3 and End Notched Flexure (ENF) tests.

Fig. 3. DCB tests performed on a LSI-C/SiC CMC laminate

This experimental activity allowed the development of new methods to obtain pre-cracks in the material and obtained particularly interesting results. The interlaminar toughness of the material was found influenced by a fibre-matrix interaction that led to increase the resistance to crack advancement, as reported in [7].

All these experimental results provided the guidelines for the improvement of the LSI technological process, aiming at obtaining a more regular micro-structure of the material, exemplified in Fig. 4. Such evolution eventually led to improve the overall tensile properties of the material.

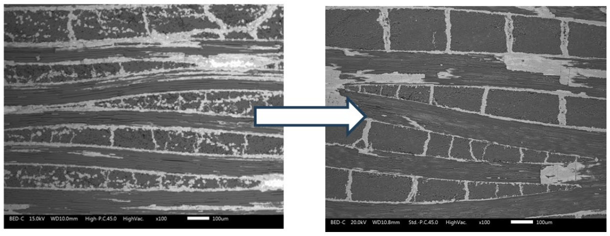

Fig. 4. Evolution of the LSI-C/SiC CMC material microstructure achieved by Petroceramics S.p.A. during the project

The activities performed on the UHTCMC material, produced by CNR-ISSM, evidenced a peculiar behaviour, characterized by an S-shaped response, which affects all the lamination sequences, as shown in Fig. 5.

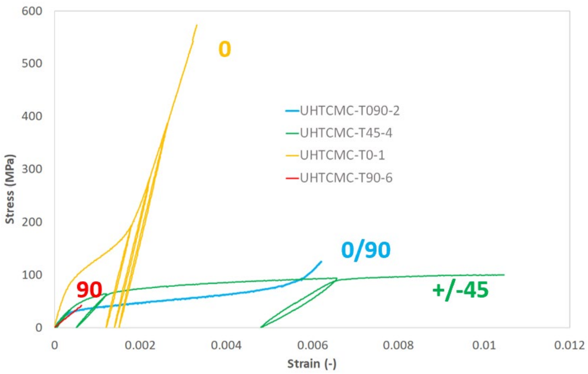

Fig. 5. Tensile response of Cf-ZrB2/SiC UHTCMC laminates with different lay-ups

The most interesting aspect of the responses reported in Fig. 5 is represented by the non-linear behaviour of the $[0]_8$ and $[0/90]_{3s}$ fiber-dominated lay-ups. An accurate analysis of the material microstructure and the comparison with previous bending experimental tests led to attribute such resposne to the presence of significant thermal residual stresses. Such stresses, orginated during the technological process are progressively released when the material is damaged with the application of a mechanical strain as described in detail in [8, 9].

Such response limit the elastic range of the material, but increases noticeably the damage tolerance and the toughness of the material. Efforts have been succesfully carried out acting on the material composition and the technological process to control such response, thus achieving a desired trade-off between the elastic limit and the maximum failure strains.

The activities performed in the project led to a substantial improvement in the scalability of the technological process for UHTCMC materials and allowed the production of elements with complex geometries and lay-ups as those shown in Fig. 6, which have been manufactured with innovative net-shape techniques using graphite moulds.

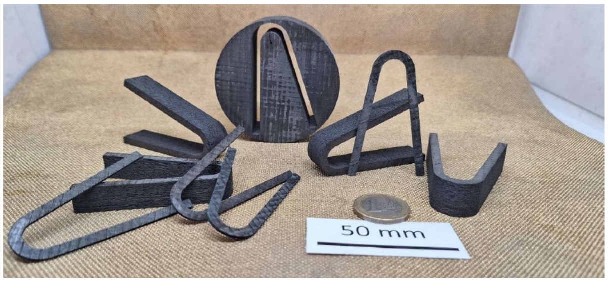

Fig. 6. Net-shape elements produced by CNR-ISSMC by using innovative manufacturing techniques

2. Numerical models of CMC and UHTCMC materials developed at the meso-scale

2.1 Characteristic of bi-phasic approaches for modelling composite non-linear response and failure

The experimental results provided the basis for the development of numerical approaches at the meso-scale level of the laminates. In these models, laminates are modelled with a finite element approach at the homogenized ply level, typically using one element per ply in the thickness directions. Material constitutive laws have been developed to capture the non-linear response that characterize the behaviour of laminates, which must be reliably understood and predicted to design damage tolerant structural elements for reusable space vehicles.

For both the LSI-C/SiC CMC and Cf-ZrB2/SiC UHTCMC considered in the project one the selected approaches was based on bi-phasic material models.

Such approach involves the decomposition of the material responses into idealized phases representing fibre-dominated and matrix-dominated properties. It should not be considered an actual micromechanical model since the two phases are idealizations that share the same material volume in the finite element mesh. The stress response is formulated considering the average stress of the plies, so that, in the elastic range, the constitutive law for a fabric ply with reinforcement fibres in two directions is represented as in Eq. 1.

$$\begin{bmatrix}\sigma_{11}\\ \sigma_{22}\\ \sigma_{33}\\ \tau_{12}\\ \tau_{23}\\ \tau_{31}\end{bmatrix} = \left(\begin{bmatrix} D^f_{11} & 0 & 0 & 0 & 0 & 0\\ 0 & D^f_{22} & 0 & 0 & 0 & 0\\ 0 & 0 & 0 & 0 & 0 & 0\\ 0 & 0 & 0 & 0 & 0 & 0\\ 0 & 0 & 0 & 0 & 0 & 0\\ 0 & 0 & 0 & 0 & 0 & 0 \end{bmatrix} + \begin{bmatrix} D^m_{11} & D^m_{12} & D^m_{13} & 0 & 0 & 0\\ D^m_{21} & D^m_{22} & D^m_{23} & 0 & 0 & 0\\ D^m_{31} & D^m_{32} & D^m_{33} & 0 & 0 & 0\\ 0 & 0 & 0 & D^m_{44} & 0 & 0\\ 0 & 0 & 0 & 0 & D^m_{55} & 0\\ 0 & 0 & 0 & 0 & 0 & D^m_{66} \end{bmatrix} \right) \begin{bmatrix}\varepsilon_{11}\\ \varepsilon_{22}\\ \varepsilon_{33}\\ \gamma_{12}\\ \gamma_{23}\\ \gamma_{31}\end{bmatrix} \tag{1}$$

It can be observed that the idealized fibre phase carries only the stress in the reinforcement directions, while the idealized matrix phase represents an effective medium that account for the additional stress contributions in the same direction and for the stress components in all the other directions. A similar approach can be found in the binary models developed for CMC in [9,10] and in the hybrid-biphasic models conceived for polymeric matrix composites in [12]. In the case of a unidirectional material the stiffness matrix of the idealized matrix phase is characterized by a single non-null coefficient D^f_{11}.

The advantages of the bi-phasic approach are various and depends on the details of model implementation. They allow a clear separation between the representation of the inelastic responses originated by matrix cracking or pseudo-plasticity in the matrix [10-12] and they can be exploited for modelling of in-plane and out-of-plane damage phenomena in laminates with a single constitutive law [12]. Moreover, they make possible the introduction of self-equilibrated thermal residual stress states in the materials originated by the different response of fibre and matrices [9].

All the material models presented in this section were implemented in VUMAT material subroutines linked to the Simulia/Abaqus Explicit solver.

2.2 Application of bi-phasic approach to LSI-C/SiC CMC

The development of a bi-phasic material model of the LSI-C/SiC composite considered in this work was carried out with the following objectives:

1) modelling the non-linear response of the different lay-ups shown in Fig. 1;
2) achieving a reliable prediction of the ultimate stress obtained in the tests, including the bending-to-tensile strength ratio;
3) representing the damage evolution in the matrix and the fundamental role of matrix damage in the final failure of the ceramic composite material.

Such objectives were achieved by applying a Continuum Damage Mechanics approach with completely different evolution laws attributed to the idealized matrix and fibre phases [13]. The matrix law was characterized by the progressive evolution of a single damage variable, activated at a threshold that was represented in the stress space by a quadratic formula. An additional threshold was defined for the activation of a strain softening behaviour to represent the maximum stress state that can be carried by the idealized matrix phase before a decaying of its stress carrying capabilities. The surfaces representing the damage and the softening thresholds are presented in Fig. 7 in a section of the stress space.

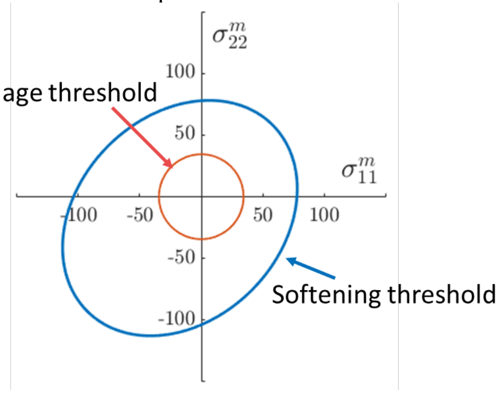

Fig. 7. Damage and softerning threshold surfaces for the idealized matrix phase in the LSI-C/SiC CMC material model

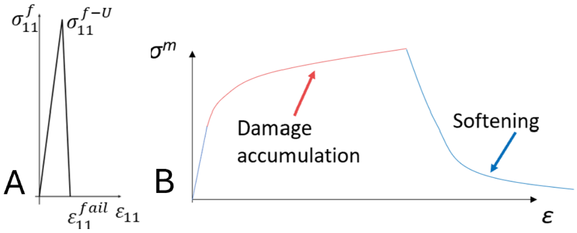

Fig. 8. Damage evolution law for (A) fibre phase and (B) matrix phase in the LSI-C/SiC CMC material model

For the idealized fibre phase, a brittle behaviour was modelled by rapidly degrading the stiffness after an ultimate stress level. The stress vs. strain responses obtained by applying the damage evolution laws of the idealized matrix and fibre phases are qualitatively represented in Fig. 8.

The appreciable results obtained by the bi-phasic model are summarized in Fig. 9. The bending-to-strength strength ratio was also captured by the model by introducing a statistical distribution of fibre strength properties in the numerical model of the bending test, as illustrated in the corner of Fig. 9-B.

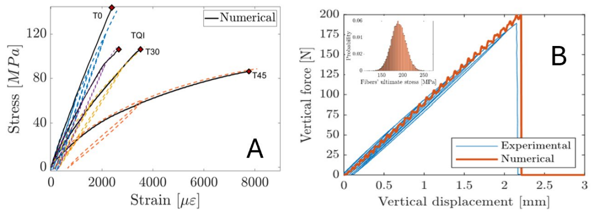

Fig. 9. Numerica-experimental correlation for the LSI-C/SiC CMC material model in (A) tensile and (B) bending tests

2.2 Application of bi-phasic approach to Cf-ZrB2/SiC UHTCMC

The bi-phasic approach was also applied to the UHTCMC material with the objective of representing the non-linear response of fibre-dominated laminates on a physical basis. Hence, the interpretation presented in the previous section, related to the combined effect of the thermal residual stress entrapped in the material and of the inelastic mechanism activated in the matrix phase, was implemented in the model.

According to such interpretation, described in detail in [8,9], the physical phenomena that originates the peculiar non-linear response of the material can be represented as in Fig. 10.

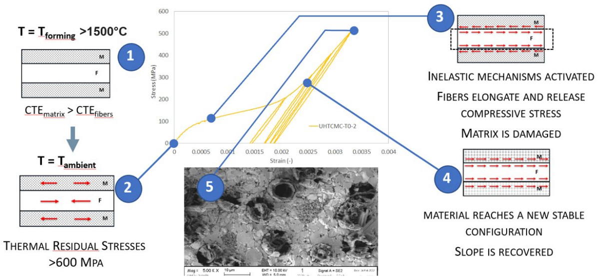

Fig. 10. Interpretation of physical phenomena that originates the response of Cf-ZrB2/SiC UHTCMC loaded in fibre direction

The effectiveness of a bi-phasic approach to represent the phenomena shown in Fig. 10 is illustrated by an unidimensional analytical model where the stress increment in the composite is obtained by adding the contribution of a linear elastic fibre phase to that of an inelastic matrix phase, with a tangent modulus depending on the applied mechanical strain, as in Eq. 2.

$$d\sigma_c = E_c^t d\varepsilon = \widetilde{E}_f \cdot d\varepsilon + \widetilde{E}_m^t(\varepsilon) \cdot d\varepsilon \tag{2}$$

An initial residual thermal stress is considered, which is characterized by a tensile stress acting in the matrix and a compressive stress acting on the fibre. If the tangent modulus of the matrix is degraded, the matrix phase can loose the capability of carrying the stress required to equilibrate the fibre stress and the compressive strain in the fibre is released thus leading to the elongation of composite without requiring the application of external mechanical stress. Then, the progressive unloading of the fibres reduces the stress acting on the matrix until the material reaches a new stabilzed state. At that point, the material recover almost completely the initial stiffness, as shown in the experimental tests. If the matrix degradation is properly calibrated in the model, the quantitative result shown in Fig. 11 can be achieved and a good correlation with the peculiar S-shaped experimental response is obtained. It should be remarked that the correlation does not depend on the specific values attributed to the initial thermal stresses, but on relation between the elastic modulus of the fibre phase and the tangent modulus of the matrix. When the latter becomes negative, the tangent modulus of the composite is reduced. In the limit case when the negative tangent modulus of the matrix is equal to the opposite of the elastic modulus of the fibres, as stated in Eq. 3, a perfectly horizontal plateau is achieved in the composite stress vs. strain response.

$$\widetilde{E}_m^t(\varepsilon) = -\widetilde{E}_f \tag{3}$$

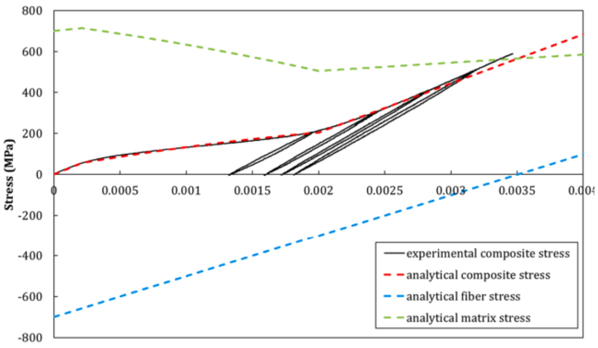

Fig. 11. Quantitative results obtained by applying a simplified bi-phasic monodimensional model to capture the response of the Cf-ZrB2/SiC UHTCMC material tensed in fibre direction

The numerical implementation in a finite element frame was carried out by attributing an elastic-plastic behaviour to the idealized matrix phase, based on a plastic flow rule associated to a Drucker-Prager yield criteria with a ductile damage driven by the plastic strain. Different Coefficient of Thermal Expansions were attributed to the two phases. The mechanical analysis was divided in two steps: the first simulated a temperature change in an unconstrained element and the consequent build-of thermal stress of opposite sign in the matrix and in the fibre, while the second step represented the application of a mechanical strain. Such approach was able of capturing both the response of $[0]_8$ and $[0/90]_{3s}$ fiber-dominated lay-ups as presented in Fig. 12.

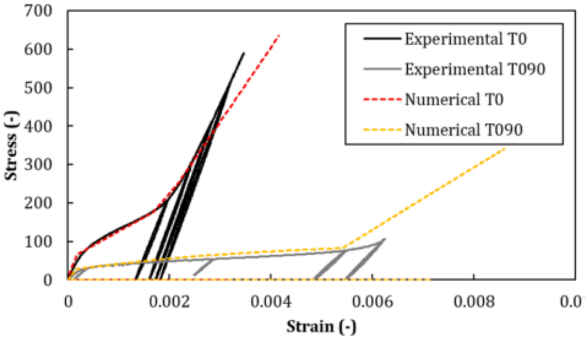

Fig. 12. Numerical-experimental correlation for a bi-phasic approach applied to the Cf-ZrB2/SiC UHTCMC material

2.3 Modelling of the internalminar response of LSI-C/SiC CMC by using Cohesive Zone Models

Cohesive Zone Models (CZM) represent nowadays a consolidated approach for out-of-plane damage onset and propagation in composite laminates [15].

The bi-phasic approach also represents an effective frame to implement CZM, as shown in [12] and [14] for polymeric and carbon matrix composites, respectively. For such a reason, CZM were applied to model the interlaminar response of the CMC under study in the project, by adopting the tri-linear response shown in Fig. 13-A.

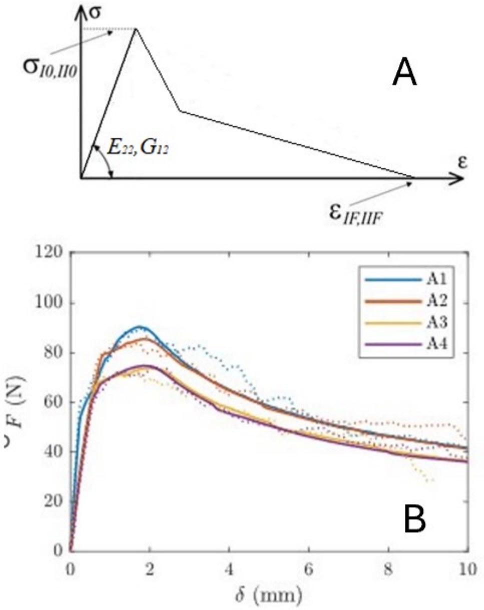

Fig. 13. Modelling of interlaminar response of the LSI-C/SiC CMC laminates: (A) tri-linear response attributed to Cohesive Zone Model and (B) example of numerical-experimental correlation

Numerical models of the DCB tests were created by using the Simulia/Abaqus Explicit solver. Due to the scattering exhibited by the specimens, the parameters of the numerical model were identified by an automatic procedure developed in [16], which was applied individually to each specimen as to obtaining a range of modelling parameters [7]. An example of the numerical-experimental correlation for a set of DCB specimens is presented in Fig. 13-B.

2.4 Elastic-plastic model of the Cf-ZrB2/SiC UHTCMC for engineering applications

The bi-phasic model discussed in the sub-section 2.2 was able of represent the bi-linear response of the material based on an interpretation of the physical phenomena occurring in the material, but the application to structural elements is complicated by the difficulties of simulating the build-up of the thermal residual stress during the manufacturing of complex geometries and lay-ups and the subsequent transfer of the stress-state into models developed for mechanical analysis with specific boundary conditions.

For analysing structural elements, an elastic-plastic orthotropic model of the Cf-ZrB2/SiC UHTCMC plies was developed by applying Hill plasticity theory corrected by a term depending of the hydrostatic stress, in a similar way to the approach presented in [17]. The model was implemented in a VUMAT subroutine and calibration was accomplished by developing

Simulia/Abaqus Explicit model of tensile tests on $[0/90]_{3s}$, $[+45/-45]_{3s}$ specimens and of three-point bending tests on $[0/90]_{5s}$ specimens. The numerical-experimental correlation achieved is presented in Fig. 14. The final failure in the numerical curves was obtained by introducing in the constitutive law a scalar damage variable driven by the equivalent plastic strain.

The model was applied to capture the response of the material in the more complex stress-strain conditions than those of the tensile and bending tests. For such a reason the *ring-on-ring* tests presented in Fig. 15-A were performed, to induce a biaxial stress state in the plies of the laminates undergoing bending.

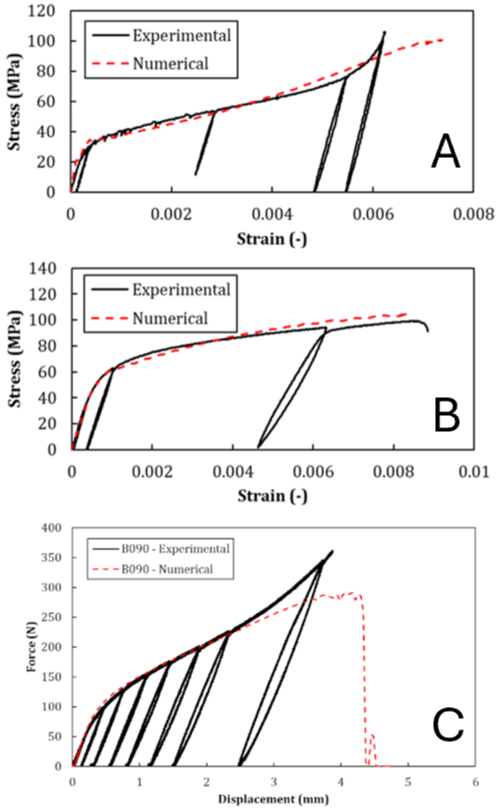

Fig. 14. Numerical-experimental correlation of the elastic-plastic model of the Cf-ZrB2/SiC UHTCMC material: (A) stress-strain response on $[0/90]_{3s}$ and (B) $[+45/-45]_{3s}$ tensile test, and (C) force vs. displacement response of $[0/90]_{3s}$ bending specimens

The finite element model of the disk was developed in Simulia/Abaqus Explicit and provided the quantitative correlation presented in Fig. 16-A. The contuour reported in Fig. 16-B represent the damage developed in the plies of the meso-scale numerical model.

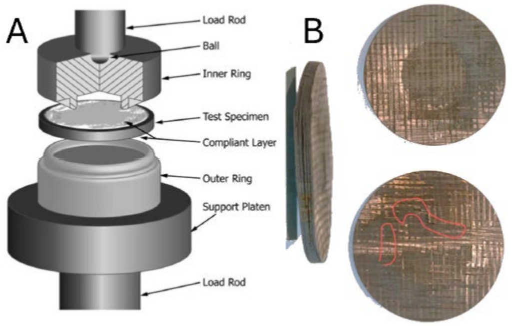

Fig. 15. (A) Layout of ring-on-ring tests and (B) Cf-ZrB2/SiC UHTCMC disk subjected to the test

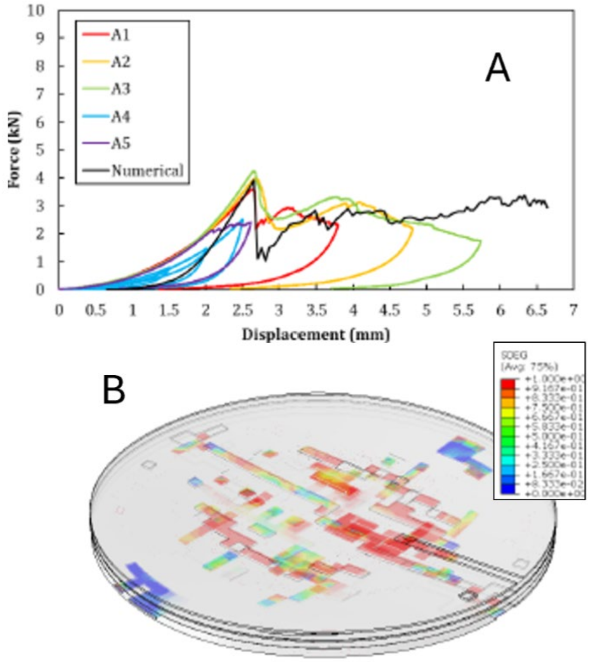

Fig. 16 – Numerical-experimental correlation for the ring-on-ring test case: (A) quantitative results and (B) contour of damage in plies

3. Strength criteria and experiments for the design of damage tolerant CMC and UHTCMC structures

3.1 Development of criteria for design at the structural scale by virtual testing

The numerical models presented in the previous section are being used to develop strength criteria for application at the structural scale. Virtual tests with different lamination sequences can be performed by using numerical models of plates endowed with the non-linear consitutitive laws validated at the meso-scale level, as in the example reported in Fig. 17-A.

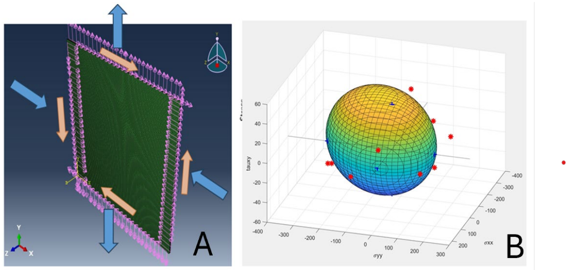

Fig. 17 –(A) virtual test on a multi-directional plate with complex load condition and (B) assessment of a Tsai-Wu criterion for the LSI-C/SiC CMC material

The stress state in the plies at the failure of the virtual plate analyis is examined. Results are reported in the stress-space to be compared with the failure surface defined through a Tsai-Wu criterion at the lamina level, which is calibrated by using both the original experimental results and a selection of virtual experiments (Fig. 17-B). The adequacy of the criteria can be verified and the best calibration options can be identified to guarantee a margin of safety without excessively conservative choices.

3.2 Tolerance to damages induced by low-energy impacts

In the design of structural parts designed for reusable space vehicles, a paramount role is played by the evaluation of the damage tolerance of the materials, which is required to understand how the structural elements behave in the presence of degradations induced by external events.

In the project, the response to low-energy impact was first investigated. Such threat is not typical of space structures, but its importance could be emphasized in future vehicles, considering the use in multiple missions, the possibility of impacts with debris at landing, and the mishandling during maintenance and mission preparation operations.

In the experimental campaign, spherical impactors with different mass and kinetic energy (Fig. 18-A) were dropped on specimens, as exemplified in Fig. 18-B, referred to a Cf-ZrB2/SiC UHTCMC beam clamped at the end.

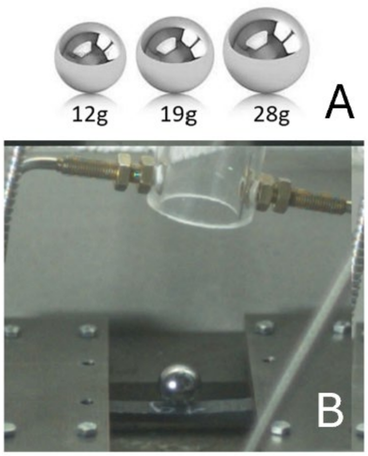

Fig. 18. Experiments for impact tolerance of CMC and UHTCMC beam: (A) spherical impactor (B) impact on a beam

The impacted specimens were then subjected to three-point bending tests to compare the results with the ones of pristine specimens.

The results of impacts on narrow beams confirmed that the peculiar non-linear response of Cf-ZrB2/SiC UHTCMC provides the material a significant impact tolerance, since a permanent bending is obtained and the ultimate strain at failure is reduced, with a small effect on the stress at the elastic limit and at failure (See Fig. 19-A).

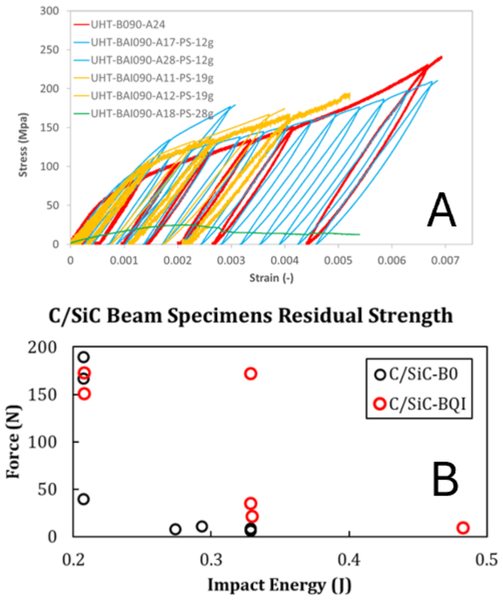

Fig. 19. Residual response of beams subjected to low-energy: (A) bending response of Cf-ZrB2/SiC UHTCMC beams and (B) residual bending strength of cross-ply (B0) and quasi-isotropic (BQI) LSI-C/SiC CMC

For the LSI-C/SiC CMC material, the results on the beams indicated the existence of a threshold impact energy beyond which a sudden drop of load carrying capability occurs (Fig. 19-B). Such threshold is apparently 30% higher for quasi-isotropic lay-up (B0) than for pure cross-ply specimens((BQI).

Considering the LSI-C/SiC CMC, impacts were also performed on 150 mm x 100 mm plates, by using heavier impactors than the previous spherical ones, to evaluate a detectability threshold (see Fig. 20). The plates were subject to three-point bending tests and the comparison with pristine plates indicated a loss of about 20%-30% of the strength for a damage at the threshold of detectability [18].

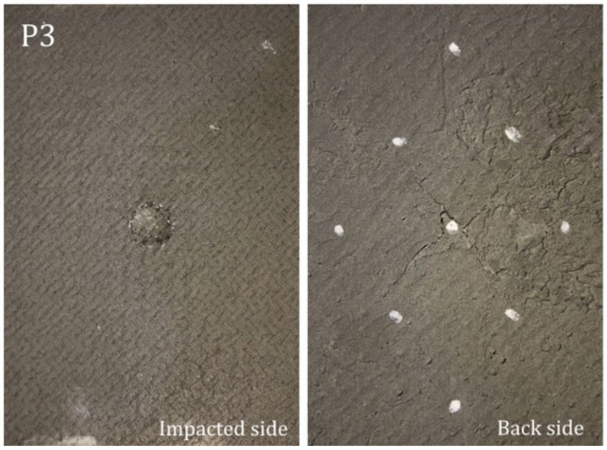

Fig. 20. Outcome of impact tests performed on LSI-C/SiC CMC plates

3.3 Exposition to relevant environment in arc-jet and plasma wind tunnel tests

Another area of investigation related to the damage tolerance of the composites under study was more specific to space applications and was focused on the effects of the exposition to high temperatures in an oxydizing environment.

A first test campaign was performed on beam specimens by using the Small Planetary Entry Simulator (SPES) facility of the University of Naples Federico II.

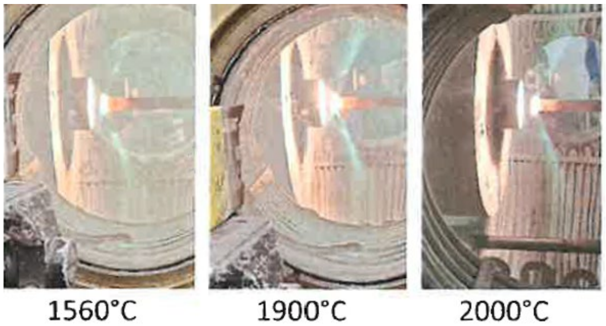

Fig. 21. Pictures taken at increasing temperature in the SPES facility during the test of a Cf-ZrB2/SiC UHTCMC beam

In the tests, exemplified in Fig. 21, the Cf-ZrB2/SiC UHTCMC specimens were exposed to plasma flow at a maximum temperature of 2150 °C for two minutes. A mass loss of 0.2% was detected. The residual strength of the specimens was measured in three-point bending tests and the maximum strength reduction was found lower than 30%, even after the exposure to subsequent thermal vacuum cycles. The tests performed on LSI-C/SiC CMC were carried out at a lower temperaure, about 1500 °C and did not lead to apparent degradation of the protective layer obtained on the specimens.

A second test campaign was carried out at the Plasma Wind Tunnel test facility Scirocco, at the Italian Aerospace Research Center (CIRA). The tests involved a series of wedge-shaped specimens produced both in LSI-C/SiC CMC and Cf-ZrB2/SiC UHTCMC materials.

Specimens were introduced in the plasma flow generated in the *Scirocco* chamber by means of a specifically designed sample holder, which was attached to moveable calibration probes, as shown in Fig. 22-A. During the tests, all the most relevant physical parameters were acquired and the temperatures were recorded by using dual-colour pyrometers, thermographic acquisition, like the one shown in Fig. 22-B, and thermocouples.

The wedge-shaped samples were then subjected to destructive opening and closing tests (see Fig. 22-C) and the results compared with those of the pristine specimens.

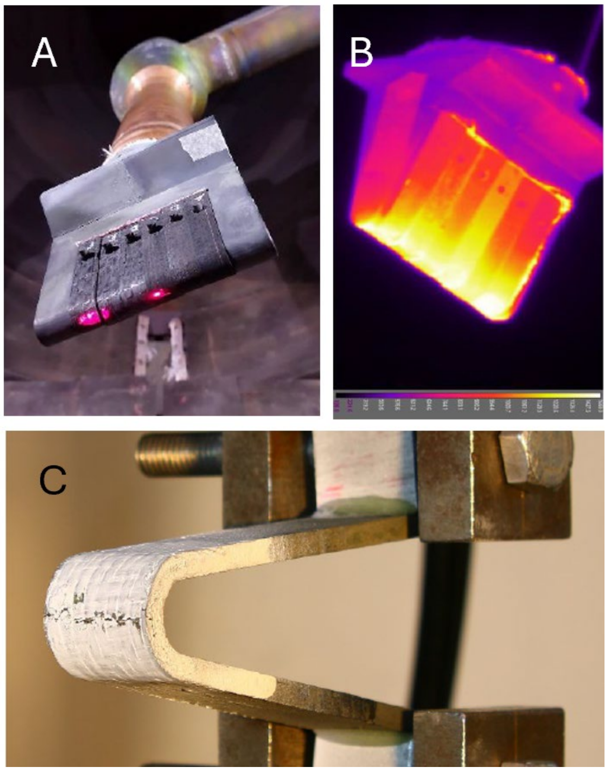

Fig. 22. Test campaing on wedge-shaped specimens exposed to re-entry conditions in Plasma Wind Tunnel: (A) Sample holder in Scirocco PWT chamber, (B) thermographic acquisition duing test, (C) destructive mechanical test

The tests reached temperatures well beyond the design temperature considered for the LSI-C/SiC CMC specimens, confirming the robustness of the protective layer until to temperatures of 1900 °C. A visible damage was produced at higher temperature, but the element still retained about 65% of the initial strength. The tests on Cf-ZrB2/SiC UHTCMC were performed at temperatures around 2000 °C for about 3 minutes.

The main results of these activities confirmed the possibility of the materials to work in the expected range of temperature but also disclosed the possibility of adopting a damage tolerant philosophy for the structural design. Indeed, it was evidenced that the exposure to heat fluxes exceeding the expected operational conditions produces clearly visible signs on the surfaces and that, even in these cases, the materials retain an adequate structural strength to complete the mission, thus limiting the safety risk for the payloads and occupants.

4. Conclusions

The large amount of data collected, of numerical tools developed, and of experimental protocols assessed in the AM3aC2A project represenst a valuable background for the further development of lightweight reusable space vehicles by using advanced ceramic composites materials.

The results confirm that the use of LSI-produced CMC material can be extended to significant parts of primary structures in re-entry vehicles, thus making available an appealing alternative to solutions where the structural role and the functions of thermal protection systems are completely separated. At the same time, the studies have enhanced the maturity level of UHTCMC materials and of the related production process, significantly increasing, at the same time, the understanding of their response and the role of thermal residual stress in their behaviour.

Further elaborations of the collected data and future activities stemming from the ones that have been summarized in this work will consolidate the technological know-how related to these materials and to the structural parts of space vehicles that will take advantage of their outstanding properties.

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Appendix A (Title)

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Appendix B (Title)

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Etc.

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