# High and Ultra High Temperature Ceramic Matrix Composites for Hypersonic Systems

**Mario De Stefano Fumo**
Italian Aerospace Research Centre
Via Maiorise, 81043, Capua
ITALY

m.destefano@cira.it

**Diletta Sciti and Luca Zoli**
CNR-ISSMC
Via Granarolo 64, 48018, Faenza
ITALY

diletta.sciti@issmc.cnr.it

**Lorenzo Cavalli**
Petroceramics Spa
Viale Europa 2, Stezzano
ITALY

cavalli@petroceramics.com

**Alessandro Airoldi**
Politecnico di Milano
Via La Masa 34, 20156 Milano
ITALY

alessandro.airoldi@polimi.it

## *ABSTRACT*

*Although the first hypersonic flight was achieved over 70 years ago, there has been increasing interest from a broader audience due to modern engineering advances that are poised to revolutionize defensive capabilities, sub-orbital travel, and rapid access to space.*

*When vehicle speeds pass supersonic conditions and enter the hypersonic regime (conventionally fixed to Mach 5) the physics of external aerodynamic flows become dominated by aerothermal heating rather than aerodynamic forces. Aerodynamic compression and friction in stagnation and off-stagnation points create high enthalpy gas dynamics that impart additional physical phenomena from the energy exchange of a superheated atmosphere. This superheated atmosphere results in high heat fluxes (some orders of magnitude greater than the 1.4 kW/m$^2$ from the sun); extreme thermal gradients (changing from -170°C to 3,000°C across distances of order 1 cm); high stagnation pressures (~105–107 Pa); and destructive plasma from gas ionization, which can strongly accelerate materials oxidation.*

*All of these formidable phenomena must be accommodated by materials in the principal subsystems of a hypersonic vehicle: aeroshell/primary structure, leading edges, control surfaces, acreage thermal protection, propulsion, and guidance systems. Developing engineering materials for hypersonic vehicles has become the focus of cutting-edge research and these materials are presently rate-limiting steps for* *the resilience of structures during operation in extreme environments, adding complexity and cost to material system development.*

*For these reasons, the performance of future defence platforms is highly reliant upon the emergence of materials able to withstand repeated operation at very high temperatures (>1,500°C) while subjected to high stresses from aerothermal and manoeuvre loads, severe thermal gradients, extreme thermal shocks, and particle impacts while also enduring exposure to high speed, sometimes ionized, reactive gas flows. Examples include components for the hot sections of turbine or scram jet propulsion systems, rocket nozzles, hypersonic leading edges, thermal protection systems of re-entry vehicles and aerothermal structures of high-speed interceptors.*

*During the last thirty years in Europe, C/SiC solutions have been developed during different re-entry spacecraft projects (X-38, EXPERT, IXV) with the operative requirement of a single mission at temperatures up to 1700° C.*

*Another more recent solution is the material class of the Ultra High Temperature Ceramics Matrix Composites (UHTCMC). These materials are mainly based on matrices of metal borides reinforced with carbon fibres and aim to reach operating temperatures above 2,000°C. Recent works demonstrated their potential for use as thermal protections and hot structures for hypersonic vehicles and re-entry systems.*

*The design of high temperature ceramic matrix composites (CMC) and UHTCMC structures for reusable systems will solve a series of significant critical issues due to the complex behaviour of the orthotropic materials characterized by multiple modes of damage often interacting. Furthermore, the degradation of the mechanical characteristics of the material, subject to mechanical and thermal cycling conditions in space environment and hypersonic flight in oxidizing environment. For these reasons, the design approach is presently based on very conservative criteria and, in parallel, extensive experimental activities are needed to certify materials and components.*

*This report presents numerical modelling supported by materials characterization and experiments conducted in a relevant environment. In particular, modelling activities have been carried out with the objectives of: providing suitable macroscale models describing the mechanical behaviour of the involved CMCs and confirming an understanding of the mechanical, thermal and chemical phenomena at work during operation. Thanks to plasma wind tunnel facilities available at the Italian Aerospace Research Centre, testing in a relevant environment in dedicated facilities allowed closing of the circle by means of verifying the initial requirements with the identified materials solutions.*

## *RESUME*

*Bien que le premier vol hypersonique ait été réalisé il y a environ 70 ans, l'intérêt pour ce domaine s'est considérablement élargi ces dernières années grâce aux avancées de l'ingénierie moderne, qui sont en train de révolutionner les capacités de défense, les voyages suborbitaux et l'accès rapide à l'espace.*

*Lorsque la vitesse des véhicules dépasse le régime supersonique pour entrer dans le domaine hypersonique (conventionnellement fixé à Mach 5), la physique des écoulements aérodynamiques externes devient dominée par l'échauffement aérodynamique plutôt que par les forces aérodynamiques. La compression aérodynamique et la friction aux points de stagnation et hors stagnation génèrent des dynamiques de gaz à haute enthalpie, entraînant des phénomènes physiques supplémentaires dus aux échanges d'énergie dans une atmosphère surchauffée. Cette atmosphère surchauffée entraîne : des flux thermiques très élevés (plusieurs ordres de grandeur supérieurs aux 1,4 $kW/m^2$ du soleil) ; des gradients thermiques extrêmes (variant de -170 °C à 3000 °C sur des distances de l'ordre du centimètre) ; des pressions de stagnation élevées ($\sim 10^5$-$10^7$ Pa) ; et un plasma destructeur issu de l'ionisation des gaz, qui peut fortement accélérer l'oxydation des matériaux.*

*Tous ces phénomènes redoutables doivent être pris en compte par les matériaux des sous-systèmes principaux d'un véhicule hypersonique : coques/aérostructures primaires, bords d'attaque, surfaces de contrôle, protections thermiques de surface, propulsion et systèmes de guidage. Le développement de matériaux d'ingénierie pour les véhicules hypersoniques est devenu un domaine de recherche de pointe, représentant actuellement un facteur limitant pour la résilience des structures dans des environnements extrêmes, et ajoutant complexité et coûts au développement des systèmes de matériaux.*

*Pour ces raisons, les performances des futures plateformes de défense dépendent fortement de l'émergence de matériaux capables de résister à des opérations répétées à très haute température (>1500 °C), tout en étant soumis à des contraintes élevées liées aux charges aérodynamiques thermiques et de manœuvre, à des gradients thermiques sévères, à des chocs thermiques extrêmes et à des impacts de particules, le tout dans des flux de gaz réactifs à haute vitesse, parfois ionisés. Parmi les exemples, on peut citer les composants des parties chaudes des turbines ou des systèmes de propulsion scramjet, les tuyères de fusée, les bords d'attaque hypersoniques, les systèmes de protection thermique des véhicules de rentrée atmosphérique, ainsi que les structures aérothermiques des intercepteurs à grande vitesse.*

*En Europe, au cours des trente dernières années, des solutions en C/SiC ont été développées dans le cadre de différents projets de véhicules de rentrée (X-38, EXPERT, IXV), avec l'exigence opérationnelle d'une mission unique à des températures allant jusqu'à 1700 °C.*

*Une solution plus récente est représentée par la classe des composites à matrice céramique à ultra haute température (UHTCMC). Ces matériaux sont principalement basés sur des matrices de borures métalliques renforcées de fibres de carbone, et visent à atteindre des températures de fonctionnement supérieures à 2000 °C. Des travaux récents ont démontré leur potentiel pour être utilisés comme protections thermiques et structures chaudes pour les véhicules hypersoniques et les systèmes de rentrée.*

*La conception de structures en CMC et UHTCMC haute température pour des systèmes réutilisables doit résoudre une série de problèmes critiques majeurs, en raison du comportement complexe des matériaux orthotropes, caractérisés par de multiples modes de dégradation souvent interdépendants. De plus, la dégradation des caractéristiques mécaniques des matériaux soumis à des cycles mécaniques et thermiques dans l'environnement spatial et lors du vol hypersonique en milieu oxydant est une préoccupation majeure. Pour ces raisons, l'approche de conception repose actuellement sur des critères très conservateurs, et des activités expérimentales étendues sont menées en parallèle pour certifier les matériaux et les composants.*

*L'article présente des modélisations numériques alimentées par des caractérisations de matériaux et des essais en environnement représentatif. En particulier, les activités de modélisation ont été menées avec les objectifs suivants : fournir des modèles macroscopiques appropriés décrivant le comportement mécanique des CMC concernés ; confirmer la compréhension des phénomènes mécaniques, thermiques et chimiques en jeu pendant l'opération. Grâce aux installations de soufflerie à plasma disponibles au Centre italien de recherche aérospatiale, des essais dans un environnement pertinent, réalisés dans des installations dédiées, ont permis de boucler la boucle en vérifiant les exigences initiales avec les solutions matériaux identifiées.*

## KEY WORDS

Ceramic matrix composites; Hypersonic systems; Modelling

## MILITARY RELEVANCE

The hypersonic programs that are being pursued at various stages of maturity worldwide have mainly produced experimental missiles, except for the Zircon, which has been used by Russia in the current Ukrainian conflict. From the perspective of NATO member states, hypersonic weapons can defeat enemy Anti-Access/Area

Denial capabilities and could, if unmatched, provide an adversary with the means to coerce UE and NATO in times of crisis.

The performance of these systems is highly reliant upon the emergence of materials able to withstand repeated operation at very high temperatures (>1,500°C) while subjected to high stresses from aerothermal and maneuver loads, severe thermal gradients, extreme thermal shocks, and particle impacts, while also enduring exposure to high speed, sometimes ionized, reactive gas flows. Examples include components of the hot sections of turbine or scram jet propulsion systems, rocket nozzles, hypersonic leading edges, thermal protection systems of re-entry vehicles and aerothermal structures of high-speed interceptors.

## 1.0 HIGH TEMPERATURE MATERIALS TECHNOLOGIES

Although hypersonic technologies and systems have existed for more than 70 years, national and international interest in them has varied. At times, these technologies are viewed as critically important, and at other times they receive little to no interest beyond a small research community. Today, hypersonic vehicles receive remarkable attention as geopolitical forces return nations to a great-power competition. It is important to review what the classification "hypersonics" involves. Using the parameter Mach number, defined as the ratio of speed to the local sound speed, supersonic flight refers to vehicles flying faster than Mach 1. For supersonic flight, aerodynamic phenomena are strongly impacted by Mach number. Hypersonic flight is defined as a vehicle flying faster than Mach 5. Also, the term hypersonics is now used to describe all aspects of vehicles flying at these speeds.


**Figure 1: Examples of past, current, and potential hypersonic capabilities. (a) Apollo re-entry capsule and ballistic re-entry vehicles. (b) X-15 reusable research aircraft. (c) X-37B reusable spaceplane. (d) Space Shuttle and Falcon 9 first stage. (e) Standard Missile-3 interceptor missile. (f) HyFLY, X-51A, and Hypersonic Air-breathing Weapon Concept (HAWC). (g) Tactical Boost Glide concept vehicles and DF-17 missiles. (h) X-43A, Skylon and Falcon concepts. (i) Sänger and National AeroSpace Plane Program (NASP) concepts. (j) Electromagnetically launched railgun projectile [1].**

As flight speeds and associated energy levels increase, additional physical phenomena become important, and the term hypersonics was introduced to refer to these speeds. Regarding aerodynamics, important energy exchange mechanisms in the fluid occur due to excitation of vibration and electronic energy levels, dissociation of air molecules and their attendant chemical reactions, and ionization of the gas to create plasma. At sufficiently high speeds and altitudes, the boundary layer (i.e., the gas layer near any solid surface, where dissipative friction and heat transfer dominate) can grow quickly enough to have a significant impact on the entire flow-field. As these phenomena become more important, Mach number loses much of its significance as the aerodynamic phenomena become much more complex. Therefore, the term hypersonic materials refers to materials with potential application to hypersonic vehicles. Among others, important technologies include aerodynamics; propulsion; high temperature materials and structures; thermal protection systems; and guidance, navigation, and control.

## 1.1 Motivation and Rationale for High Temperature Materials Research and Development R&D Selection

Sustained hypersonic flight at high Mach numbers subjects a vehicle to a variety of high heat fluxes and heat loads that vary with position on the vehicle, ranging from those that can be sustained by current materials to those that exceed their capabilities, even for short durations. For the latter, designs depend on cooling by radiation or flowing fuel, or in a few locations the use of heat pipes. The ability of the system designer to close a vehicle design depends strongly on where on the vehicle it is necessary to use cooling, the efficiency of the cooling strategy, and the weight and thermomechanical performance of all the materials used, whether cooled or uncooled.

Specific deficiencies in existing materials depend on their location on the vehicle, as well as the operating mission, Mach number, and lifetime. Some of the most severe materials challenges are embodied in two examples:

1) Leading edges (nose, cowl lip, and control surfaces) are exposed to extremely high heat fluxes on small tip regions that are difficult to access for active cooling (preferred designs for low drag may have a tip radius in the order of millimetres and a wedge angle around 10°, leading to surface temperatures over 2,000°C). Without active cooling, the only apparent solutions are materials with a combined high temperature capability and high thermal conductivity (to spread the heat for dissipation by radiation or conduction to a heat sink). As illustrated in the property map in Figure 2, the major contenders for materials that fill this need are refractory carbides and borides or C-C composites with aligned high conductivity fibres.

   However, all of these materials are limited in application by their susceptibility to severe oxidation, as well as cracking induced by high thermal gradients. Advancing the science needed to overcome these limitations was one of the research goals of this program. This included understanding oxidation mechanisms and devising new approaches for improving the stability and life of refractory diboride materials at high temperatures.

2) In the propulsion flow path, where radiation cooling is not available, active cooling using fuel (hydrogen or hydrocarbon) is essential. For sustained flight at high Mach numbers, the system design requires a material with high strength-to-weight ratio (since the structures forming the propulsion flowpath have a large area) and capable of operating with high surface temperatures to minimize heat absorbed and thus the fuel required for cooling. Ceramic matrix composites (CMCs) (and carbon-carbon composites) are the only classes of materials so far identified as potentially capable of satisfying these requirements: as illustrated in Figure 2(c), the specific strengths of CMCs (C-SiC and SiC-SiC) and carbon-carbon at temperatures above about 1,200°C exceed those of competing refractory metals by about a factor of three. Thin skins are required with complex geometrical features, including internal channels for coolant flow (either regenerative or transpiration cooling) and features to allow attachment to the surrounding structure.

   However, the application of these materials is held back by several shortcomings:

   i) Surface temperatures remain limited to the range ~1,400°C–1,600°C, whereas higher temperature capability is desirable for durability, especially in regions susceptible to heat spikes from shock interactions or combustion instabilities;

   ii) Lifetimes are limited by oxidation, which is exacerbated by microcracking driven by high thermal gradients, especially at the highest use temperatures; and

   iii) A capability for high-fidelity modelling of damage and lifetime does not exist.

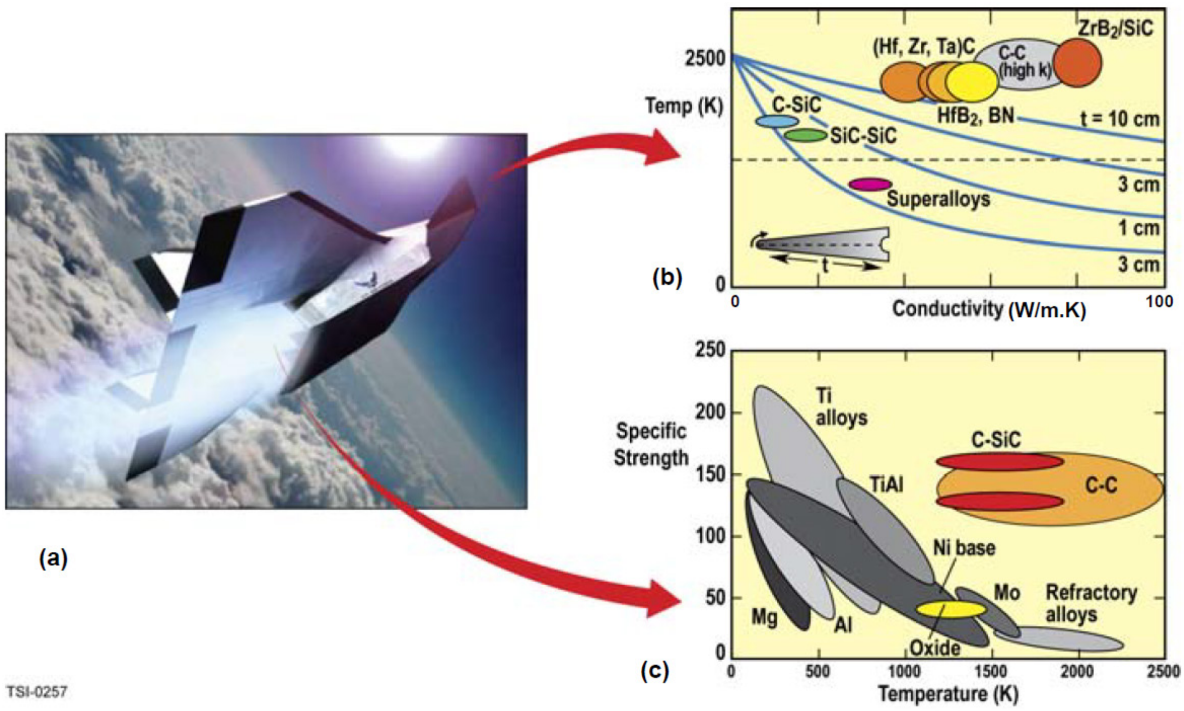


**Figure 2: (a) Visualization of hypersonic vehicle. Material property comparisons for leading edges (b) and large-area hot structures (c) [2].**

# 2.0 MATERIALS MANUFACTURING ROUTES

## 2.1 Ceramic Matrix Composites

Carbon-carbon (C/C) composites are types of carbon material that are made up of a reinforcement or filler (e.g., carbon fibres, granular carbons of a different nature) that may vary in their nature and geometry (e.g., unidirectional, bidirectional, and multi-directional, in the case of the carbon fibres) and a carbon matrix produced from polymers, resins, or pitches.

These composites are dense, lightweight materials with high mechanical performances, a high thermal resistance in a non-oxidant atmosphere, and inertia toward various chemical agents. In an oxidant atmosphere and at temperatures higher than 400°C–500°C these composites are reactive materials. The limit of C/C materials is the oxidation issue that requires the use of surface coatings to seal the surfaces in contact with oxygen, in particular at temperatures above 600°C.

In order to overcome this drawback of C/C materials and to increase the oxidation stability, another class of materials, so-called CMCs, characterized by an internal oxidation protection of carbon fibers, were introduced.

Different matrix types, such as SiCN and Al2O3 have been investigated. However, until now, all known industrial applications are based on SiC or SiSiC matrices, with current research activities mainly being focused on this class of materials.

Depending on the manufacturing route, the C matrix is either replaced completely by SiC matrix, leading to C/SiC, or tailored C/C preforms densified with an additional SiC or SiSiC matrix leading to C/C-SiC materials. Starting in the late 1970s, the development of C fiber-reinforced SiC materials was mainly driven by space applications. First components were used in thermal protection systems (TPSs) for reusable spacecraft, such as the nose tip and the wing leading edges of the Buran (1974–1993). Due to the development of low-cost materials and manufacturing methods, new industrial application areas outside aerospace could be successfully opened for these materials. The use of C/SiC brake disks in automobiles was an important milestone and breakthrough for this new class of materials.

In the manufacture of C/SiC materials, carbon fibres must be embedded within a SiC matrix. In practice, this is achieved by forming the SiC matrix inside a pre-shaped carbon fibre structure, or preform. The processes based on CVD, pyrolysis of preceramic polymers, and reaction bonding have been adapted for the buildup of SiC matrices in fibrous preforms, leading to three different manufacturing methods:

- CVI (Chemical Vapour Infiltration) = Deposition of gaseous SiC precursors.
- PIP (Polymer Infiltration and Pyrolysis) = Pyrolysis of Si polymers.
- MI (Melt Infiltration) = Reaction of molten Si with C.

In the CVI method, the SiC matrix is built up in one process step, using a gaseous precursor. In this process, a dry fibre preform is infiltrated with the precursor, and SiC crystals are deposited on the hot C fibre filaments, simultaneously. In contrast, using polymer precursors, several separated processes are needed to create the SiC matrix in the so-called PIP or LPI process (liquid polymer infiltration). In the first step, a carbon-fibre-reinforced plastic (CFRP) preform is manufactured by infiltrating the liquid polymer precursor into the fibre or fibre preform before curing to a thermoset polymer matrix. In the second step, the CFRP preform is pyrolyzed, converting the polymer matrix to a porous SiC matrix. Whereas in the PIP process, the SiC matrix is directly derived by the pyrolysis of the preceramic polymer matrix, two separate process steps are needed for the SiC matrix buildup in the MI or LSI (liquid silicon infiltration) process.

The Liquid Silicon Infiltration process has the advantage of being faster and cheaper than other processes, with the drawback of needing to obtain a material with lower mechanical properties to take into account for design of components. Recently CIRA and Petroceramics developed a proprietary material named ISiComp®. This material is based on carbon-fibre-reinforced silicon carbide, the so-called C/C-SiC, which is produced via LSI process. This process can be divided into three main steps:

- Manufacturing a CFRP part, where fibre fabrics impregnated with phenolic resin are formed through autoclave techniques.
- Pyrolysis of the polymer matrix above 900 °C, which leads to a porous C/C microstructure.
- Siliconizing above 1,460°C in vacuum.

Due to the high purity and the low viscosity, the molten silicon fills up the porous C/C green body by capillary effects. The infiltrated silicon then reacts with the carbonaceous matrix and some C-filaments, mainly at the surface of the individual dense C/C-areas, and is then converted to silicon carbide. Thus, the LSI process produces C/C-SiC composite materials consisting of C/C domains that bear mechanical loads, and a silicon carbide matrix that provides protection through multiple internal oxidation barriers. In addition, the C/C-SiC material is also protected against oxidation with an outer layer SiC coating based on a proprietary process for reusability purposes.

The choice of the raw material used was based on the simplest, cheapest and most robust solutions: a phenolic pre-impregnated fabric is the most manageable with respect to unidirectional fibres. The process was set up with both high tenacity (HT) carbon fibres and intermediate modulus fibres (IM), but ISiComp® makes use of IM fibres in order to achieve higher mechanical properties.

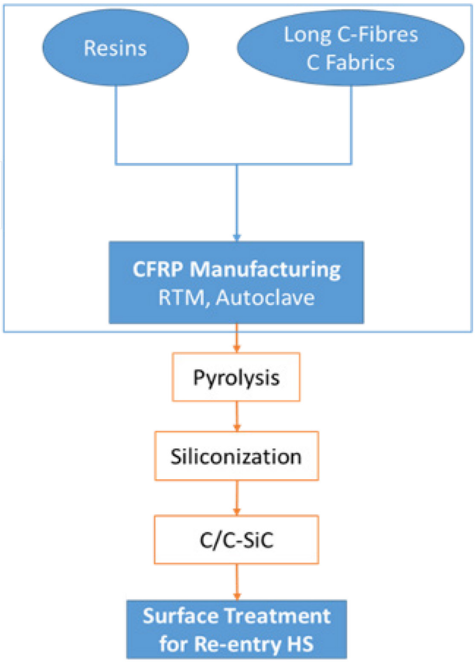


**Figure 3: LSI manufacturing process.**

Different pre-impregnated fabrics for CFRP preform manufacturing were investigated, which considered different phenolic resin formulations and different weave styles of the fabric. Furthermore, different compacting and autoclave routes, during the CFRP phase, were also investigated, since they have a critical 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 [3]. In particular, it has been found that one of the most critical parameters 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) also depends also on base material. Figure 4 shows one of the panels that delaminated during pyrolysis.

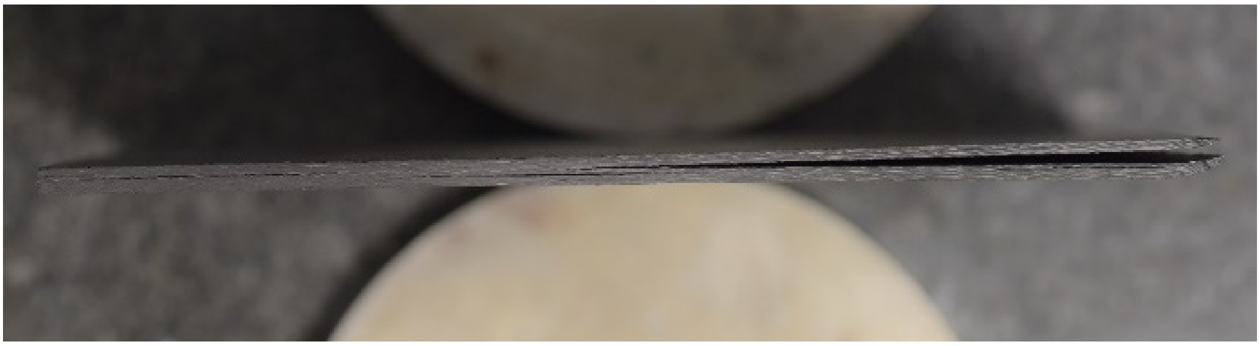

**Figure 4: Severe delamination occurred during pyrolysis.**

At the same time, compacting pressure must not be too low, as this would affect the mechanical properties of the final CMC. It has been also found that a heat treatment above the curing temperature of the CFRP after the autoclave curing step is beneficial, both to the subsequent pyrolysis and infiltration phases and to the mechanical properties. This heat treatment results in an increase of about 15% in flexural strength with respect to specimens that are not heat treated. 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 for pyrolysis [4].

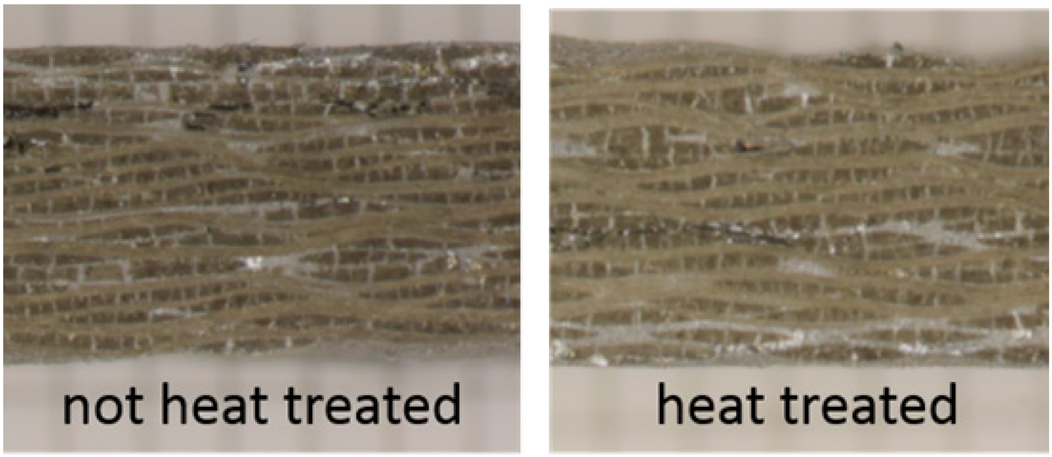


**Figure 5: Two CMC specimens: coming from heat treated CFRP and not.**

Mass loss during pyrolysis is about 20%, whereas thickness contraction is about 10%. Looking to complex shapes, this value is not negligible, highlighting that the mould design (CFRP phase) must consider the material contraction in order to preserve final CMC geometry. Moreover, proper kiln furniture has to be designed and installed in the pyrolysis oven to prevent distortions.

### 2.2.1 Coating Process

C/C-SiC ceramic composite needs an anti-oxidation coating in order to resist the harsh re-entry environment. Typically, it is made of a SiC layer. A dedicated physical vapour deposition has been developed and patented in order to find the best process parameters as to guarantee a suitable coating thickness (some tens of microns).

The deposition process scheme is shown in Figure 6. The samples have been exposed both directly and indirectly to Si vapours.

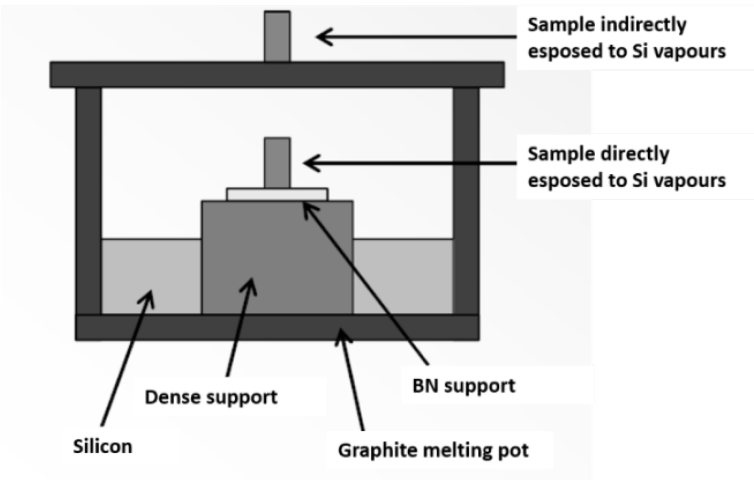


**Figure 6: Coating deposition scheme.**

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.

Figure 7 shows surface and cross section scanning electron microscope (SEM) images of the coated samples.

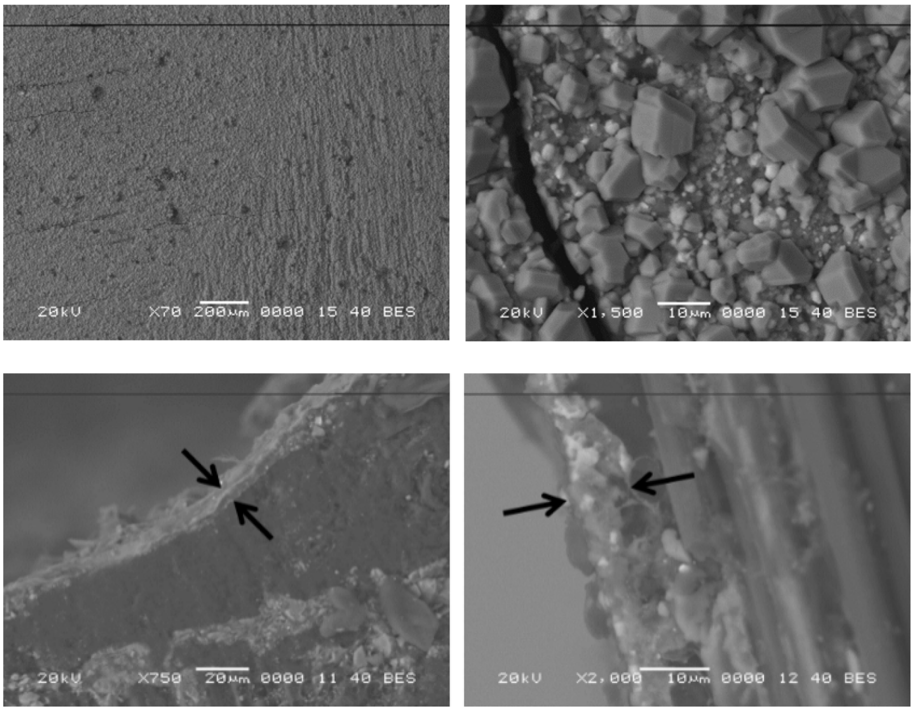


**Figure 7: SEM of coated surface and of the cross section.**

## 2.2 Ultra High Temperature Ceramic Matrix Composites

To overcome present technological limits, novel materials must be conceived that combine the best features of CMCs with those of Ultra High Temperature Ceramics (UHTCs), as depicted in Figure 8. The integration of these two classes of materials should mitigate the limits from each, leading to multiple benefits. In this respect, UHTCMCs show improved erosion/ablation resistance compared to pure CMCs, improved damage tolerance, and thermal shock compared to UHTCs and intermediate specific weight [5].

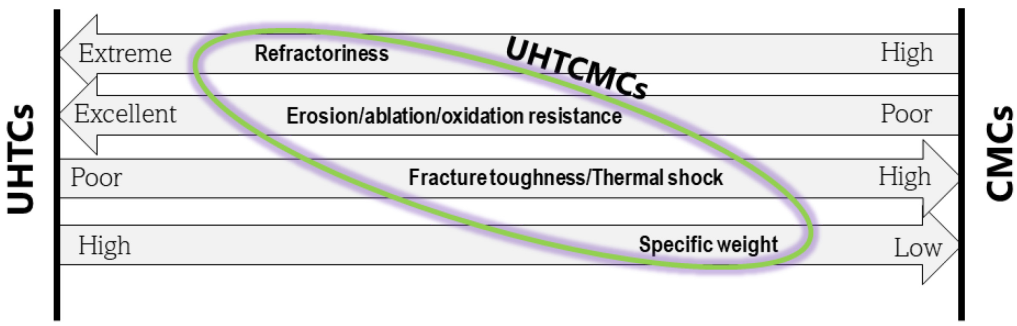


**Figure 8: UHTCMCs are hybrid composites that combine the best qualities of UHTCs (Ultra High Temperature Ceramics) with those of CMCs (Ceramic Matrix Composites).**

This new class of material can be investigated for their near-zero erosion and ablation at very high temperatures (well above 2,000°C). These properties make them especially suitable for application such as propulsion system nozzles, where they can improve the stability of the motor by minimizing erosion in the throat region. Additionally, their use in TPS materials can enhance spacecraft reliability and crew safety by improving manoeuvrability. Because these materials resist surface recession, aerodynamic performance remains consistent, and sharp leading-edge profiles can be maintained even under extreme conditions.

Although these two applications are different, they both require ultra-refractoriness (imparted by the ceramic phase) and damage tolerance (imparted by fibres). However, they operate in distinct, harsh chemical environments involving reactive particle-loaded flows, and must withstand a broad range of surface pressures and heat flux rates. The corrosive environment of a propulsion chamber is characterized by intense enthalpy gas flows of $CO/CO_2/H_2O$ vapour and the presence of condensed phases, such as particles. Re-entry, on the other hand, is characterized by high enthalpy reacting flows of fully dissociated gases (oxygen, nitrogen, etc.). This implies a need for different constituent materials, fibres and ceramic matrices, with suitable chemical/physical properties, compositions and architecture (fibre volume fraction, orientation).

Different key technologies can be considered for the manufacturing of UHTCMCs (such as Chemical Vapour Infiltration, Polymer Infiltration and Pyrolysis, Reactive Metal infiltration), however, hot pressing/spark plasma sintering (HP/SPS) are the best consolidation techniques for achieving high-density composites with elevated UHTC phase content (see Figure 9). The preferred matrix is essentially based on $ZrB_2$ enriched with secondary phases that possess different functionalities, such as sintering aids, self-healing agents, reinforcing and anti-oxidation agents.

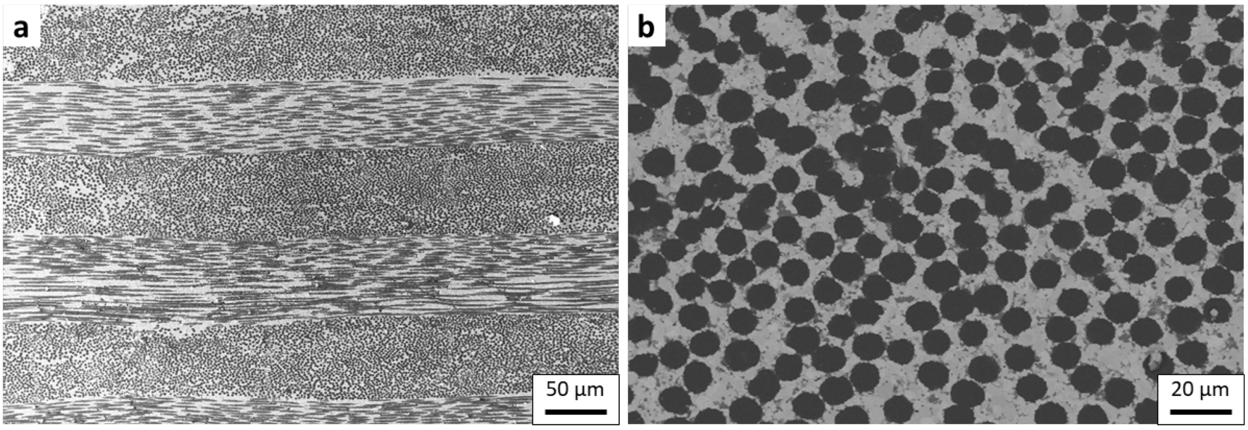


**Figure 9: a) Typical microstructure of UHTCMCs, b) detail of the cross section showing fibres embedded in the UHTC matrix.**

Components like nozzles and TPS components were manufactured and tested in relevant environments. Nozzle throats were tested under real operating conditions in small-scale solid/hybrid propulsion motors, with temperatures up to 3,000 °C, pressure up to 100 bar, thrust up to 2,500 N, and a burning time over 35 s. On the other hand, TPS tiles were manufactured and tested in arc-heated facilities, at different enthalpy and pressure levels representing different flight trajectory points (temperatures up to 2,500 °C and testing time of several minutes). [6], [7], [8].

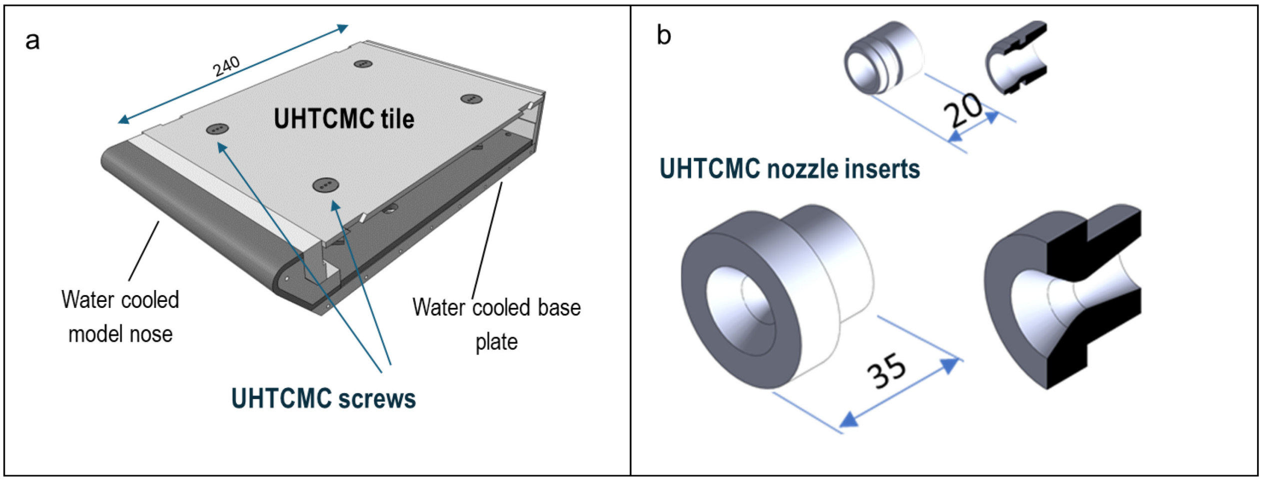


**Figure 10: Technical drawings of the breadboards, a) a tile assembled to a water cooled base plate through UHTCMC screws and b) small and intermediate nozzle prototypes. Quotes are in mm.**

## 3.0 NUMERICAL MODELLING APPROACH

Predicting the structural response of CMC materials in the presence of geometrical features, such as holes, cut-outs, highly curved parts, and macro-porosity, is one a challenging tasks when modelling of such materials. The presented results show the capability to idealize the most important failure mechanisms and to capture the quantitative response of multi-directional CMC laminates in tension and bending, thus providing a proof of its potential.

A test campaign was conducted with the aim of obtaining the basic properties of the orthotropic C/SiC fabric plies produced through the LSI technique and, at the same time, of providing validation experiments for the model to be developed. Figure 11 shows the stress vs. strain responses recorded in four types of quasi-static tensile tests on laminates with lamination sequence of $[0]_{20}$ (“T0”), $[45/-45]_{10ss}$ (“T45”), $[30/-30]_{10ss}$ (“T30”), and $[0/45/90/-45]_{5ss}$ (“TQI”). Moreover, bending tests were performed and a significant bending-to-tensile strength ratio of 1.4 was obtained, which can be attributed to the statistical distribution of strength properties and to toughness of the material.

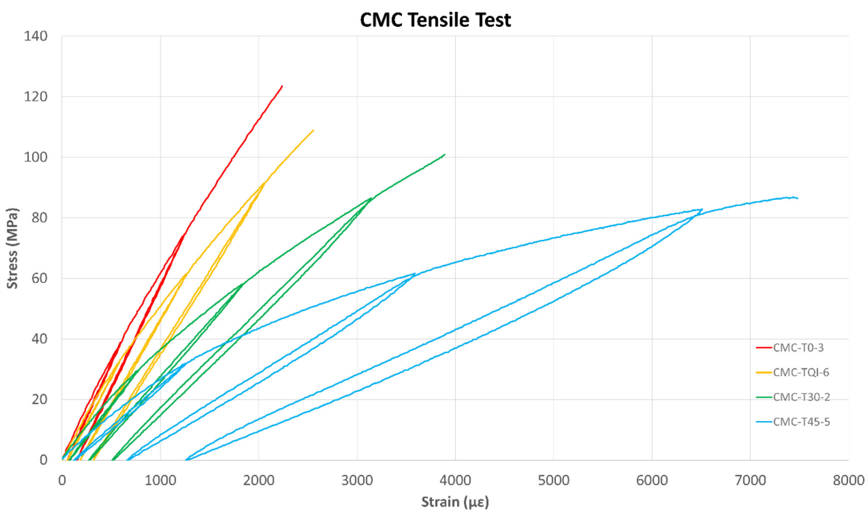


**Figure 11: Tensile test response in different CMC laminates.**

The development of a numerical model aimed to capture the most significant aspects of the mechanical response in non-linear range of the laminates, such as:

1) The non-linear curves presented in Figure 11;
2) The maximum load carrying capacity of the different lay-ups;
3) The bending-to-tensile ratio; and
4) The role of the matrix damage in the activation of the mechanisms leading to the final failure (see for instance [9]).

Considering such requirements, the numerical approach adopted moved from the technique developed in [10] and [11] for polymer matrix composites. The CMC-homogenized material model was decomposed into two idealized phases: fibre and matrix. The fibres were modelled through a layer of membrane elements, which carried stress only in the fabric reinforcement directions. On the other hand, the idealized matrix actually represented an equivalent medium that provided the additional stress contributions required to model the stress state in the composite, as in the binary models presented in [12]. Accordingly the idealized matrix was represented by solid elements. It is worth noting that, in general, the technique makes possible the representation of delamination without the use of zero-thickness cohesive elements, the development of different constitutive laws for matrix- and fibre-dominated responses, the representation of the interactions between matrix damage inside the plies and the delamination phenomena [9], [10].

The damage in the matrix plays a fundamental role in the model. A single scalar matrix damage variable is adopted, with an evolution that depends on the distance of the stress states from the Tsai-Wu shaped envelopes exemplified by theiso-damage surfaces that are shown in black and red colors in Figure 12(A). The model was implemented as a material subroutine in the Simulia/Abaqus explicit codes and was calibrated and validated by developing finite element models of the tests. Figure 12(B) and Figure 12(C) present the finite element models of the tensile and bending tests, respectively. The need to model the influence of matrix damage on the failure led to calibration of a new surface for the peak stress carried by the matrix phase, shown in green in Figure 12(B), beyond which an exponentially decaying strain softening regime was modelled. Such second surface and the limit stress carried by the fibre phase were not uniformly characterized in the models, but were statistically distributed in the elements of the models. The parameters of such distribution have been identified through a Monte-Carlo approach, considering the correlation with the ultimate strength in a selected set of the tests as main performance index.

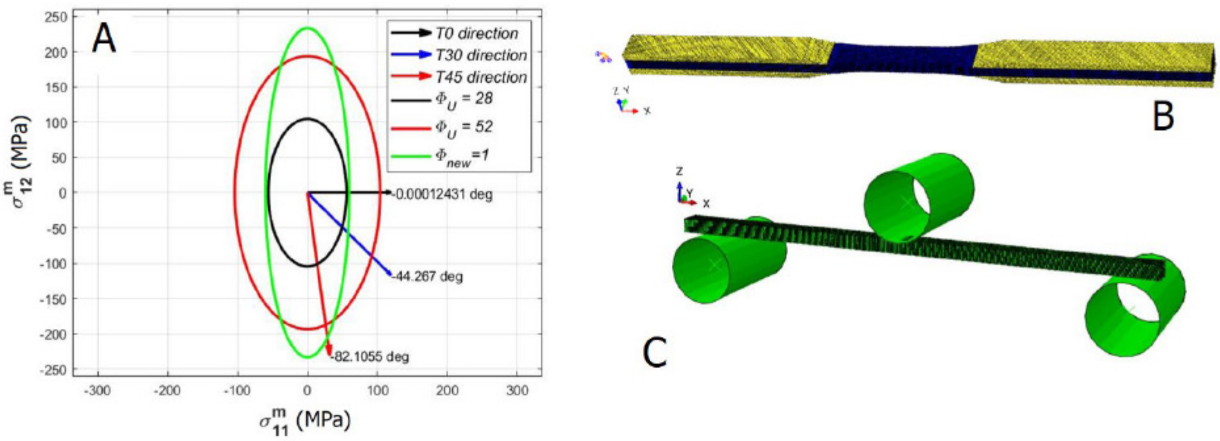


**Figure 12: Tsai-Wu shaped surface for matrix damage evolution (A) and FE models of tensile (B) and bending (C) tests.**

The bi-phasic nature of the approach, the choice and calibration of the damage evolution laws, and the statistical distribution of the ultimate strength of the idealized phases led to obtain the results presented in Figure 13, which indicate that all of the quantitative and qualitative aspects of the responses are captured. In particular, both the ultimate tensile strength in the “T0” test (Figure 13(A)) and the force vs. displacement response in the bending “B0” tests is obtained, thus indicating that the statistical distribution of properties can represent the bending/tensile strength ratio (Figure 13(D)). Moreover, the localization of fracture can be represented, as shown in Figure 13(E). For such localization, the integration of delamination damage in the analyses was found to play a fundamental role. Hence, the modelling approach was able to represent, without meshes at the sub-ply or microscopic levels, the fundamental quantitative and qualitative aspects of non-linear response and failure of the CMC material.

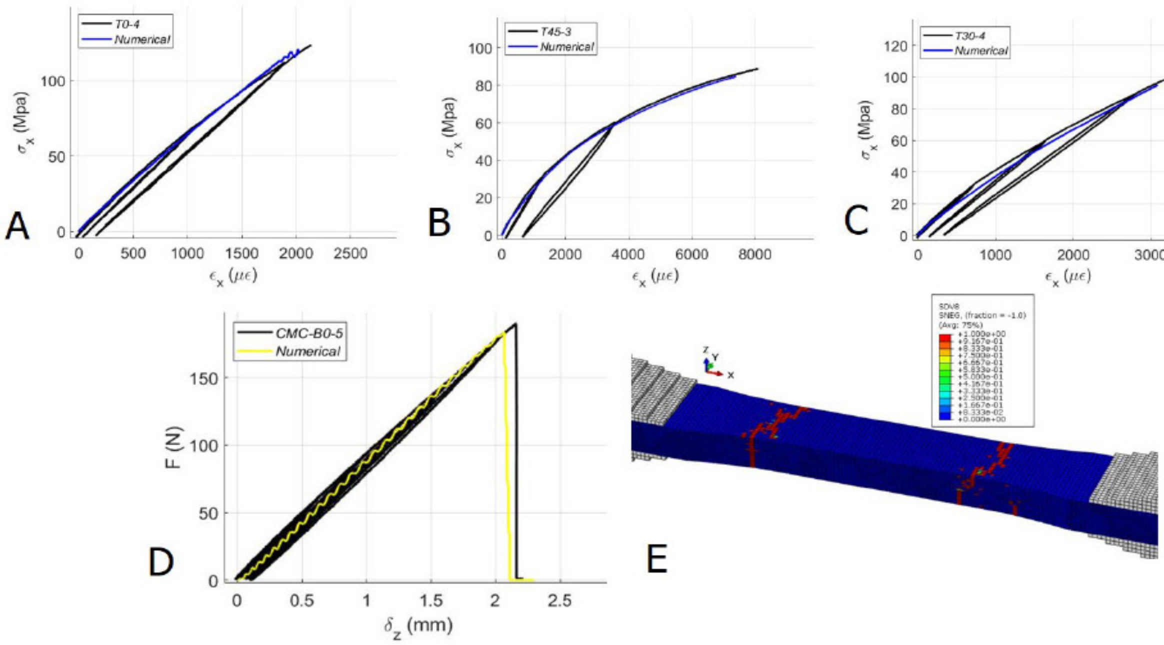


**Figure 13: Numerical-experimental correlation in test “T0! (A), “T45” (B), “T30 (C), “B0” (D) and numerical failure mode (fibre damage) in “T30” analysis.**

Modelling is less mature in the case of UHTCMCs; however, recent efforts resulted in the design and validation of a macroscale model reproducing some of the very peculiar features of these materials (e.g., decrease then increase of the tangent modulus with tensile stress), as discussed in [13].

## 4.0 TESTING IN RELEVANT ENVIRONMENTS

Looking to materials development testing in relevant environments is the key for fast and effective results. Unfortunately, testing in a hypersonic relevant environment is not easy nor cheap. Different types of hypersonic wind tunnels facilities exist and were used for experimentation. Each type of facility has its own limitations, such as test time and test model size. Each type of hypersonic facility also has its own unique advantages and disadvantages. Continuous running and blowdown facilities allow the longest running times, detailed measurements and best knowledge of freestream properties, but are limited to low stagnation enthalpies. On the other hand, reflected shock tunnels and especially expansion tunnels allow for high enthalpy conditions, but suffer from short test times and a limited variety of measurement techniques.

Hence, success in experimental hypersonics would result from testing in a variety of different facilities in order to benefit from the advantages of each facility.

For the aerothermal qualification of materials and components, it is fundamental to duplicate the heat fluxes experienced at leading edges. This can be achieved by properly coupling specific total enthalpy and total pressure. Clearly such parameters are strongly influenced by the capabilities of test facilities. Presently, the best options are given by a plasma wind tunnel, where hypersonic and high enthalpy flow can be achieved with lower pressure with respect to flight condition. This aspect is particularly important for ablative materials, where decompositions are strongly linked to pressure and shear stress whereas for rigid materials this effect is relatively negligible. This report provides one example of plasma wind tunnel testing in the SCIROCCO facility available at CIRA.

The SCIROCCO Plasma Wind Tunnel (PWT) is the world' s largest and most powerful hypersonic, high enthalpy, low pressure arc-jet facility in operation. It was built in the framework of the European Space Agency (ESA) program Hermes and has been operational since 2002. The design and engineering of the SCIROCCO facility represent the state of art of arc-jet technology.

**Figure 14: CIRA PLASMA TESTING complex aerial view.**

The main goal of the facility is to qualify large scale test articles, up to 600 mm in diameter, including TPS, hot structures, and payloads designed for space re-entry vehicles.

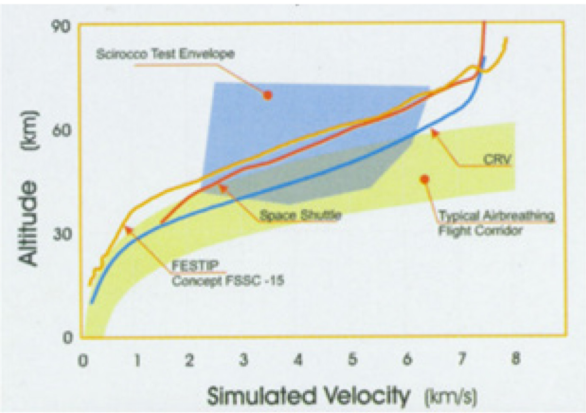


**Figure 15: SCIROCCO operative map.**

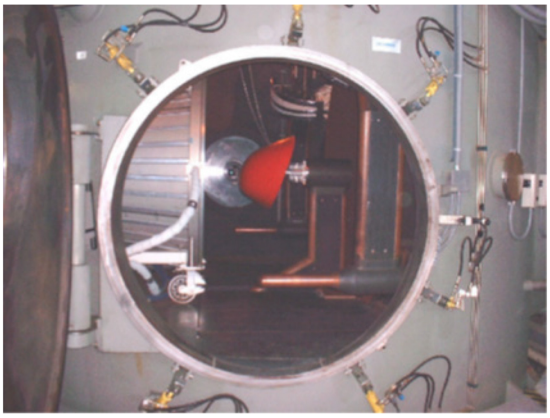

**Figure 16: SCIROCCO test chamber.**

SCIROCCO is powered by an arc heater of 70 MW maximum electrical power and is able to generate a plasma jet of up to 2 metres of diameter, at Mach 12, for a test duration up to 30 minutes. The test gas is a mixture of Air and Argon with a maximum mass flow rate of 3.5 kg/s. Inside the arc, the compressed gas is heated up to plasma temperatures in the range of 2,000 K–10,000 K.

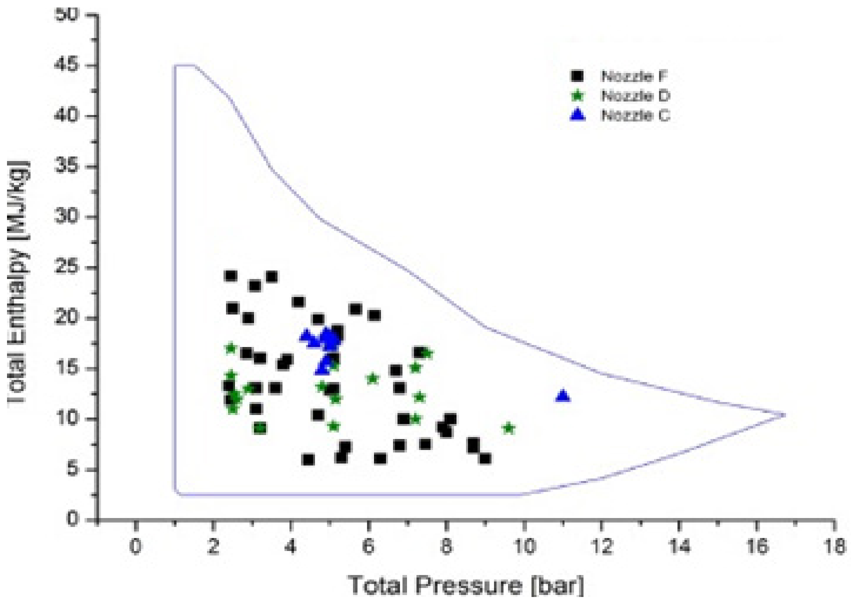


**Figure 17: SCIROCCO Performance map in terms of reservoir pressure and enthalpy.**

By operating on current and air mass flow, SCIROCCO reaches a steady operating condition characterized by fixed reservoir pressure (P0) and total enthalpy (H0); once the steady state is reached, if required, the flow can be qualified by means of a Calibration Probe—a 100 mm in diameter hemisphere copper water cooled probe—to measure pressure (PS) and heat flux (QS) at the probe stagnation point.

As an example, Figure 18 shows a test campaign as part of the AMACA project funded by Italian Space Agency for modelling and testing of UHTCMC and ISiComp® samples in hypersonic conditions.

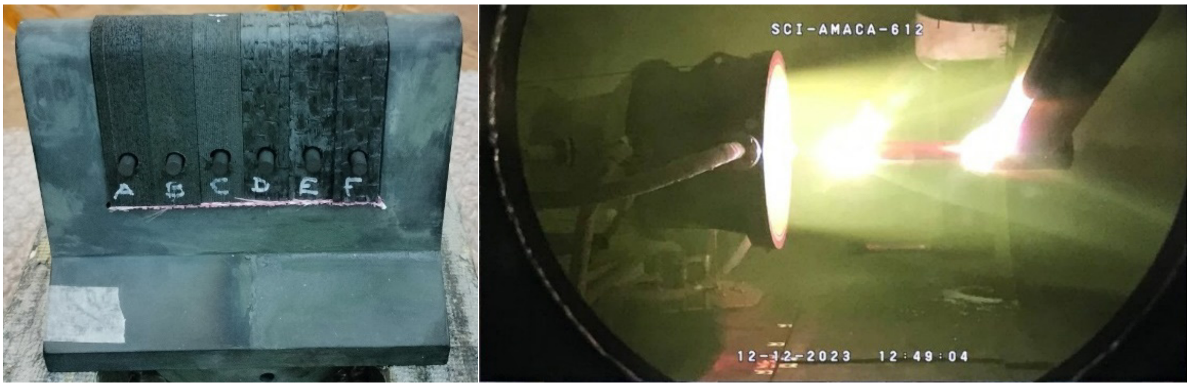


**Figure 18: Test article before and during test.**

Post-test analysis is fundamental in order to understand the behaviour of the materials under relevant aerothermal loads. Figure 19 shows infrared (IR) images taken during test, depicting the samples post-test and SEM.

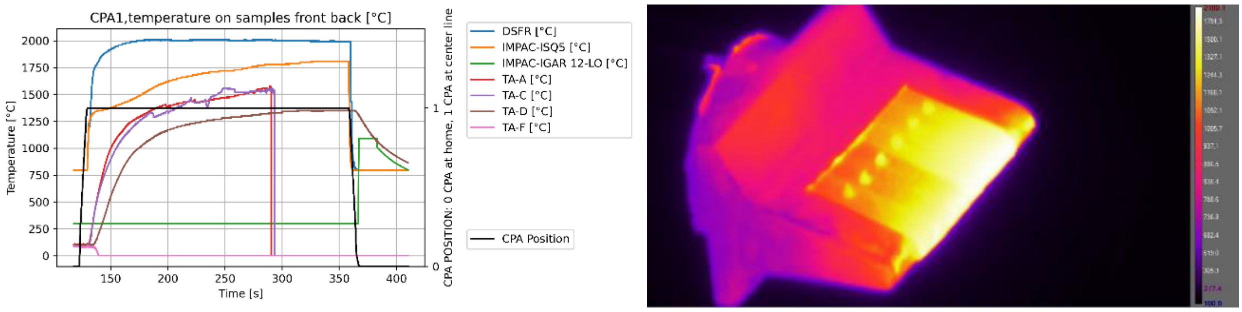


**Figure 19: Temperature measurements by pyrometers and thermocouples, and thermocamera.**

## 5.0 CONCLUSION

This report gives an overview of material research for high temperature applications focused on hypersonic systems. It covers manufacturing routes, modelling and relevant environmental testing of advanced high temperature materials as CMC and UHTCMC beyond coupon level.

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