Development, Application and Testing of High Performance Ceramic Matrix Composites based on C/SiC

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Abstract

Ceramic matrix composites (CMCs) reinforced with continuous carbon fibres feature high specific strength over a large temperature range and high damage tolerance compared to monolithic ceramics. Hence, CMCs are promising candidates for high performance structures in e.g. aerospace technologies. In order to achieve a high level of material- and product maturity at comparable low cost, adequate textile processing techniques and accompanying non-destructive investigation methods are indispensable. Especially the usage of Computer Tomography enables the non-destructive and contact-free inspection of complex geometries. In addition, a cost-effective hot testing method for small-scale components in representative harsh environment completes the field of material development.

1. Introduction

Ceramic matrix composites (CMCs) are a leading group of materials for a wide range of high performance applications in aeronautics, space exploration, energy, nuclear, and transportation industries. In the field of aeronautics and space exploration systems, these materials are utilized especially for applications in hot and highly loaded sections and harsh environments such as nozzle extensions for space propulsion, orbital thrusters, Thermal Protection Systems (TPS) for re-entry vehicles, or components for ram- and scramjet engines. Materials for suchlike applications have to withstand high aerodynamic and thermo-mechanical loads during operation. Figure 1 depicts the superiority of CMCs compared to commonly applied metals regarding the ratio of strength to weight at temperatures beyond 1,000 °C.

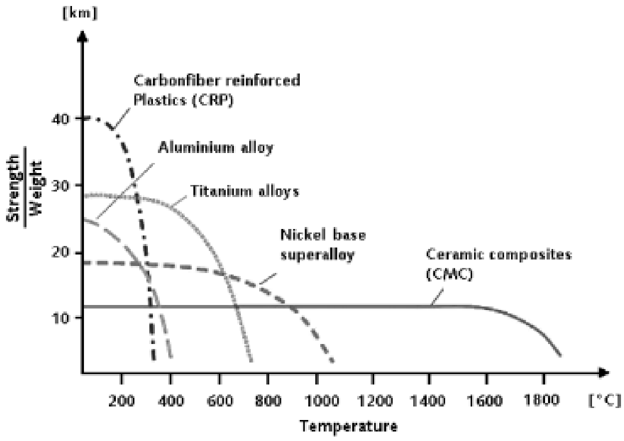

Figure 1: Ratio of strength to weight as a function of temperature [1]

Besides cost reduction, performance improvements are key drivers for the development and application of advanced materials in propulsion systems. Higher combustion efficiency and performance are reached by higher combustion temperatures and consequently lead to higher material working temperatures of up to 1,900 °C and beyond. Currently used materials in this field, which not only withstand high operating temperatures but also achieve high specific impulse, are high melting noble metals, such as Platinum-Iridium or Platinum-Rhodium, which are, however, rarely and costly. The main advantages of CMCs are low density, high resistance to thermos-shocks and high temperatures, stability to chemical attack, creep resistance and low thermal expansion coefficients. Airbus Defence and Space (Airbus DS) has been working on this key technology since the early 1990s. Airbus DS focuses on non-oxide CMCs based on carbon fibers. Manufacturing techniques, testing methods and space – relevant applications are presented in the following. Emphasis in this paper is placed upon the production accompanying non-destructive inspection via Computer Tomography (CT) [2][3].

2. Materials and Process Technologies

Due to the mentioned advantages of CMCs not only research institutes, but more and more engine manufacturers increase their efforts to push the maturity level of these materials for use in rocket engine thrust chambers or nozzle expansions. Airbus DS is involved in the development and manufacturing of components for space propulsion applications by realizing cost-effective process routes and establishing quality assurance criteria such as non-destructive investigation (NDI) methods. The currently most sophisticated CMC material at Airbus DS is carbon fibre-reinforced silicon carbide (C/SiC) named SICARBON® manufactured via Polymer Infiltration Pyrolysis (PIP). It is used in development programs for high performance orbital thrusters and applied for thermo-stable satellite structures. Further available CMC materials are carbon fibre-reinforced carbon (C/C) named CARBOTEX® using Rapid-Chemical Vapour Infiltration (r-CVI) and furthermore carbon fibre-reinforced carbon with a silicon carbide surface finish (C/C-SiC) using Liquid Silicon Infiltration (LSI) named CARBOTEX$^{SI®}$.

2.1 Process Techniques

The C/SiC material is fabricated via the filament winding and PIP process. Additional surface coatings can be applied optionally. The PIP manufacturing process follows the polymer-route using liquid ceramic polymeric precursors for wet-infiltration of either the fibre roving or the preformed fibre lay-ups. This process technique for fabricating CMCs is widely recognized as a versatile method for fabrication of large and especially complex-shaped structures. In comparison to other ceramic composite fabrication processes, the PIP process offers greater flexibility. By application of low temperature during the forming and moulding steps, the PIP approach enables the initial use of existing equipment and processing technologies derived from Carbon Fibre Reinforced Plastic (CFRP) - production. The infiltration of a matrix preform with a liquid ceramic precursor yields a high-density matrix and therefore offers many possible reinforcement geometries [4][5]. The ability to fabricate preforms featuring the desired geometry and architecture is of primary importance in the CMC production. There are a number of processes currently used for manufacturing the SICARBON® material enabling the required shapes such as filament winding (shown in Figure 2) or manual lay-up of prepregs.

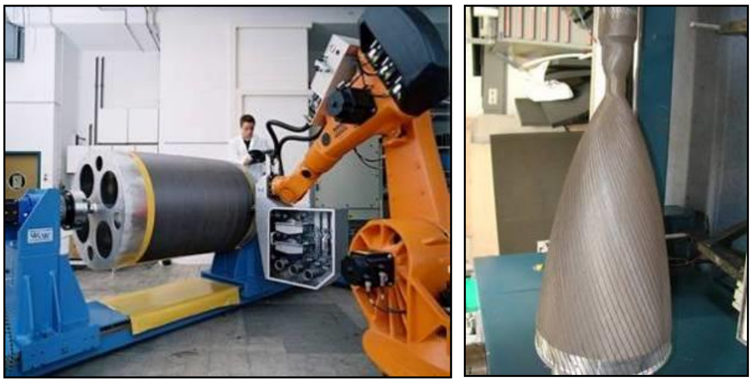

Figure 2: Filament winding facility for geometries > ∅ 1.5 m / 3 m length (left)
Filament winded orbital thruster made of C/SiC (right)

However, the subsequent required high temperature treatments can lead to reactions between matrix and fibres which can adversely affect the strength of the composite. Therefore, carbon fibres have to be modified in order to build a protective interface between the SiC matrix system and the fibre. Finally, the pre-ceramic composite undergoes a high temperature treatment to generate the ceramic state of the material. Depending on the application, an optional environmental barrier-coating or a thermal protection coating has to be applied.

The typical Airbus Group overall PIP-process is presented in Figure 3 and consists of the following single steps [6]:

1. Coating of fibres to create a weak fibre-matrix interface (ductilizing of fibre matrix bonding).
2. Infiltration of the fibres / the fibre architecture with a pure polymer-system or a powder-filled slurry system via fibre winding or lay-up of prepregs
3. Lamination of prepregs or joining of infiltrated parts as well as forming if necessary.
4. Curing with temperature and pressure in the autoclave.
5. Ceramization by pyrolysis (high temperature process in vacuum or inert atmosphere).
6. Multiple re-infiltration with a pre-ceramic polymer and following pyrolysis
7. Optionally coating with an external protection system

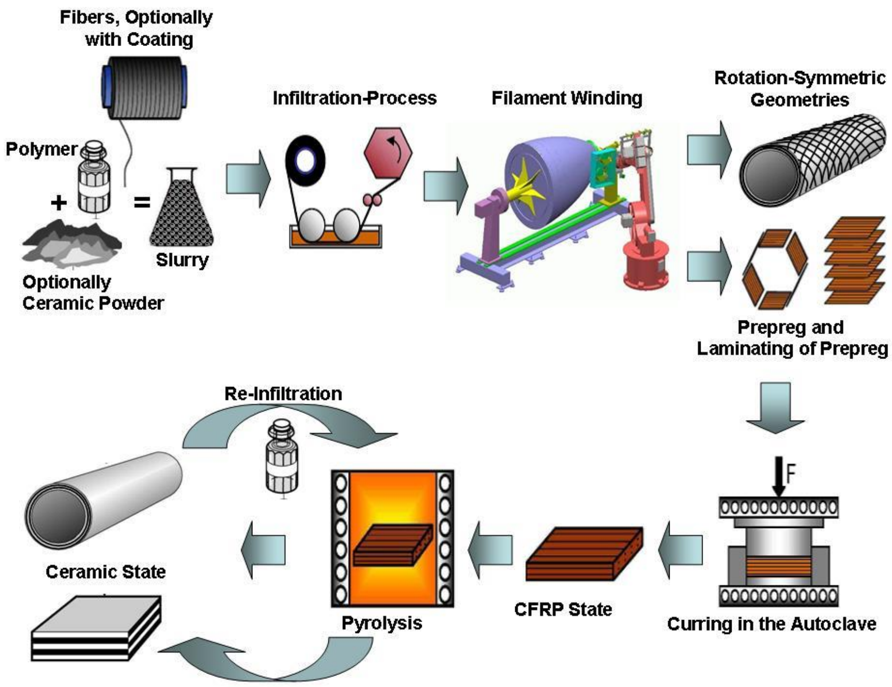

Figure 3: Schematic flowchart of the Polymer Infiltration Pyrolysis (PIP) process

SiC is also an integral part of the CARBOTEX$^{SI®}$ Surface Infiltrated (SI) material which is used for the so called PTAH-SOCAR composite structure of hypersonic propulsion components. Materials in such applications interact with hydrocarbon combustion products from combustion of kerosene and air and consequently require oxidation protection. The CARBOTEX® development was initiated with the objective of combining multidirectional (3D) textile structures with cost-effective infiltration methods. For active cooling systems several circuits have been compared to ensure high performance of the combustion chamber walls regarding the required combustion environment and the specified fuel mass flow. Therefore, different cooling channel configurations have been envisaged, such as series of rectangular shaped channels, or the so called pin-fin circuit, which was approved to be superior to mechanically machined channels in terms of cooling efficiency. Finally this resulted in the so called PTAH-SOCAR technology [7].
The textile preforms for these actively cooled applications are braided in a pre-defined fiber architecture (e.g. layer amount, fiber angle etc.) on a robot assisted circular braiding device and act as skin material for the components. To generate the typical sandwich structure of such actively cooled components comprising the typical pin-fin structure a distance-tooling has to be applied and the 2D textile-perform has to be sewed / stitched with a robot assisted sewing (tufting) device in the orientation perpendicular to the layer orientation prior to the carbon infiltration. In addition, this process step acts as the 3D reinforcement of the material. The braiding and tufting steps during textile processing are illustrated in Figure 4.
The PTAH-SOCAR-like component gets densified via the above mentioned r-CVI process, a special type of the CVI process, which has several advantages like cost and time savings as well as tuneable process routes concerning material quality and performance.

Figure 4: Robot-assisted circular braiding device (left)
Robot-assisted tufting device for multidirectional (3D) textile structures (right)

For the r-CVI a special high temperature tooling is used to guarantee a representative and reproducible densification process. Prior to the LSI process with the corresponding Si-slurry-coating (including an adapted curing cycle) a cleaning step is executed. Finally, the high temperature surface siliconization is performed yielding the final C/C-SiC state of the material (CARBOTEX$^{SI®}$). Depending on the application an optional diffusion barrier-coating can be applied. The entire CARBOTEX$^{SI®}$ manufacturing process with its specific process steps is shown in Figure 5 [2][3].

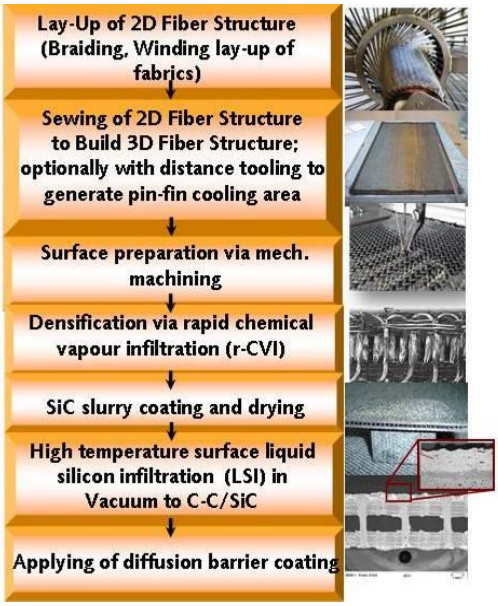

Figure 5: Schematic flowchart of the CARBOTEX$^{SI®}$ (C/C-SiC) via r-CVI & LSI

2.2 Non-destructive Investigation of CMC`s using Computer Tomography

Increasing material performance as well as enlarging of component dimensions and complexity is only achieved by using adequate inspection methods. Besides material development and manufacturing technologies, non-destructive as well as destructive inspection methods play a key role in the whole process chain to produce the desired performance and quality at reasonable cost. Regarding the significant anisotropic structure of ceramic composites, non-destructive testing is indispensable already during production. Possible production defects such as delamination or pores directly affect the lifetime and reliability of highly stressed components and consequently have to be detected timely. For that reason, various non-destructive inspection methods, namely Ultrasonic Inspection, Impulse Thermography and CT have been investigated and evaluated at Airbus DS regarding their particular suitability for CMC inspection. Due to its versatility and utility non-destructive investigation via CT turned out to be the method of choice for complex CMC parts. CT offers the possibility of visualizing the inner construction of components non-destructively and contact-free. Thus, this method is extremely advantageous for inspection of complex curved parts

such as combustion chambers. By application of the latest technologies and evaluation algorithms spatial resolutions of less than 1 µm are reached depending on the dimensions of the investigated component. Deviations in density can be detected and flaws can be characterized in terms of type, size and location. Besides detection of defects, the generated CT-data furthermore enable the virtual measurement of dimensions, for instance wall thicknesses. In addition, a comparison of the current geometry of a part can be compared with the target geometry by making an overlay of the reconstructed CT measurement data and the CAD model. Therewith, geometrical deviations can easily be visualized. Furthermore design parameters such as laid up fibre angle can be checked and secured. Consequently, CT is not only a method for non-destructive testing, but also a method for geometrical measurement allowing the inspection of various quality relevant aspects within one measurement job.
Currently three different CT technologies based on X-ray are in use for industrial purposes: Standard-CT with cone shaped X-ray beam, Helix-CT and Computer-Laminography. By generating multiple radioscopic images of an object from different positions and by using mathematical algorithms cutting planes of the object are calculated. Finally a three-dimensional volume model including its inner structure is reconstructed. Any arbitrary section plane in the object can be visualized. Regarding the standard technology a specific number of projections from the rotating part is produced (Figure 6).

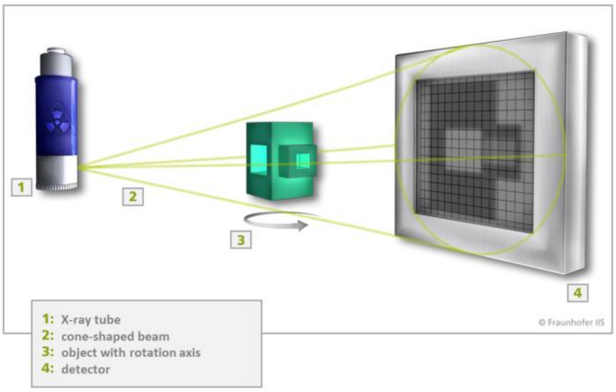

Figure 6: Standard industrial Computer Tomography based on cone shaped x-ray beam [8]

Since the material specific absorption coefficient correlates with the materials density, the radiation is attenuated resulting in a grey value distribution of every projection on the detector. The cone beam CT technology achieves sharp and precise results in sections which are penetrated by the central area of the X-ray beam. Due to scattering of radiation and edge effects typical failures, so called artefacts, occur in the re-constructed model resulting in diffuse and un-sharp imaging of edge areas. To reduce artefacts especially in edge areas two alternative CT technologies have been developed.

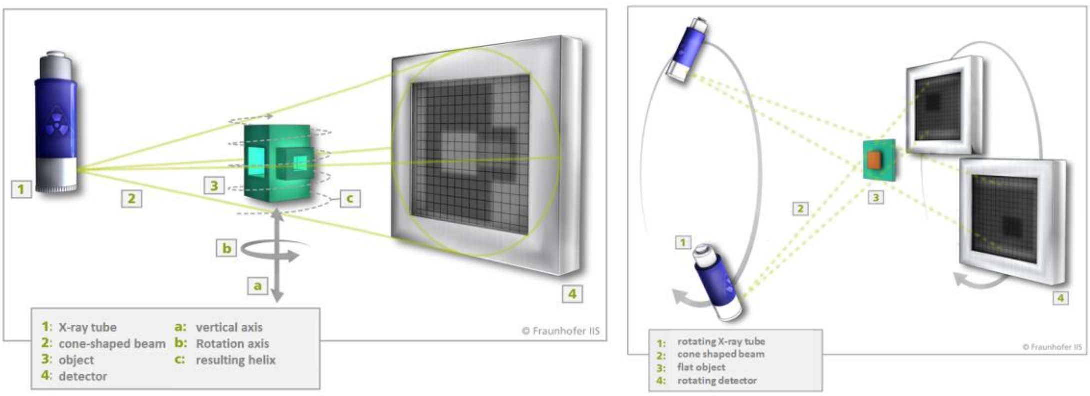

Figure 7: Helix CT (left) and Computer-Laminography (right) [8]

The Helix-CT uses an additional translatory axis along the objects rotating axis (Figure 7 (left)). The resulting helix path ensures that every section of the object is penetrated once with the centre of the x-ray beam. Modified and adapted re-construction algorithms have to be applied to yield almost artefact free images.

To illustrate the improvement from Standard-CT to Helix-CT in terms of artefacts and sharpness the measurement of a CD stack is demonstrated in Figure 8. While the upper picture, obtained from Standard-CT, shows increasing artefacts with increasing distance from the centre, the lower picture obtained from Helix-CT shows high resolution also at the edges and an increase of sharpness in general.

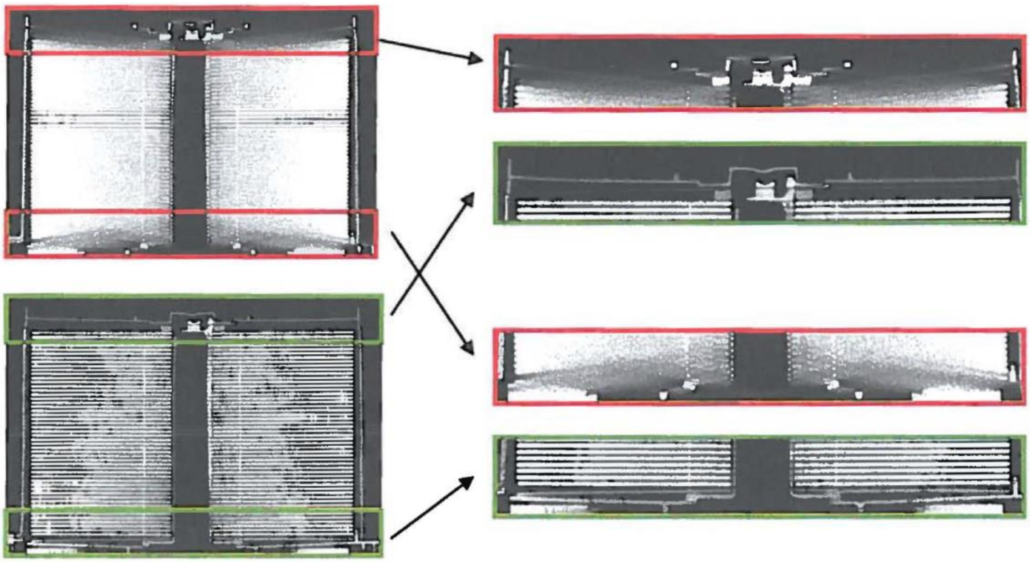

Figure 8: Section view of a CD stack via Standard CT (top picture) and Helix-CT (bottom picture). [9]

Another CT technology reducing the artefact generation is the so called Computer-Laminography shown in Figure 7 (right). By using two radiation sources and associated detectors which counter-rotate on co-planar trajectories it is assured, similar to the Helix-CT, that every section is penetrated once by the centre of the X-ray beam. This method is advantageous especially for flat components.
Typical applications for the advantageous domains of CT are presented in the following figures, namely visualization of complex inner structures (Figure 9), detection and evaluation of defects (Figure 10) and 3D measurement and comparison of current geometry with CAD target geometry (Figure 11). Nowadays CT inspections are an inherent process step of manufacturing CMCs at Airbus DS.

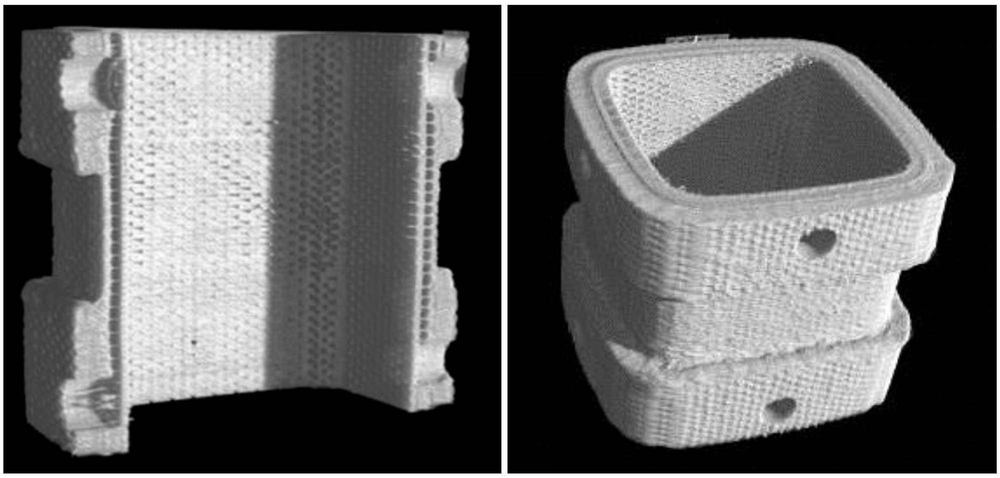

Figure 9: Visualization of inner structures via CT.

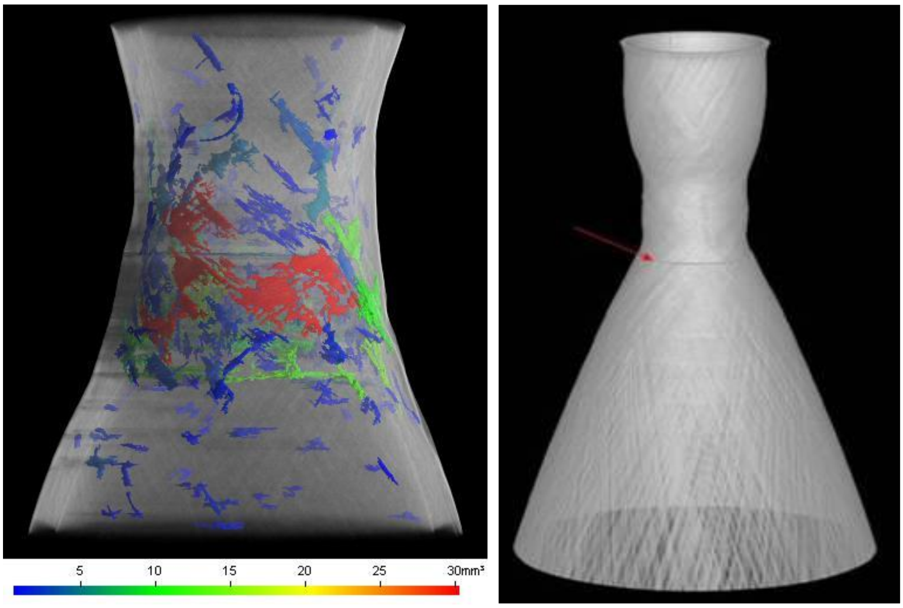

Figure 10: Detection of defects in complex shaped components.

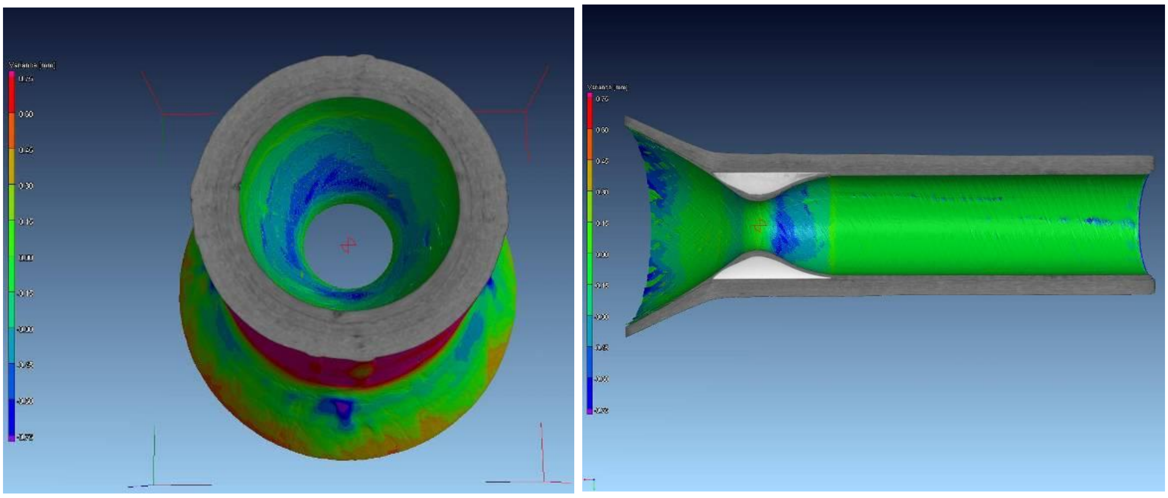

Figure 11: 3D measurement of contours and comparison with CAD data.

2.3 Destructive testing in ERBURIG hot testing rig

To complete the field of CMC material development, destructive testing of samples in representative combustion chamber-like environment has to be executed. Therefore, Airbus Group has established a facility for cost effective long term hot testing of small scale products. The so called Environmental Relevant Burner Rig (ERBURIG) is available in two different configurations, one using kerosene/oxygen as propellant (ERBURIG^K) and the other one using hydrogen/oxygen as propellant (ERBURIG^H). The facility is based on a conventional High Velocity Oxygen Fuel (HVOF) gun generally designed and applied for thermal spraying. The HVOF gun was modified and extensively characterized in order to gain a deep understanding of the produced gas flow parameters and gas compositions at various operating points. The test rig is mainly used for flat material samples, which are placed in the free jet stream. Depending on the operating point, hot gas temperatures up to 2,500 °C and velocities up to 2,000 m/s can be produced. Different material compositions or coatings can be tested very fast and cost-effective.

Figure 12: Hot testing of a flat material sample in ERBURIG[K] facility.

Besides flat material samples, micro combustion chambers are tested likewise by substituting the original ERBURIG combustion chamber. The influence of higher pressures on the material can then additionally be taken into account.

3. Space-relevant applications of CMCs

3.1 Launcher-, Orbital- and Hypersonic Propulsion

Up to now, Airbus DS has manufactured and successfully hot-tested several manufacturing demonstrators for launcher propulsion as well as for orbital propulsion. Noteworthy components made of SICARBON® via PIP process and filament winding are subscale (scale 1:5) nozzle extensions for the Vulcain main stage rocket engine, full-scale nozzle extensions for the Aestus upper stage rocket engine, and combustion chambers for a 400 N orbital thruster. Figure 13 (left) shows the hot test of the Vulcain subscale C/SiC nozzle extension on the F3 test bench in Ottobrunn. Combustion chamber pressures of 80 bar were applied and short-time (couple of seconds) temperatures of up to 2,000 °C have been measured on the hot gas wall. Figure 13 (right) shows the EAM orbital thruster during hot firing. The objective of this test campaign was regarding long-term behaviour resulting in 5,700 s accumulated test duration at a combustion chamber pressure of 11 bar.

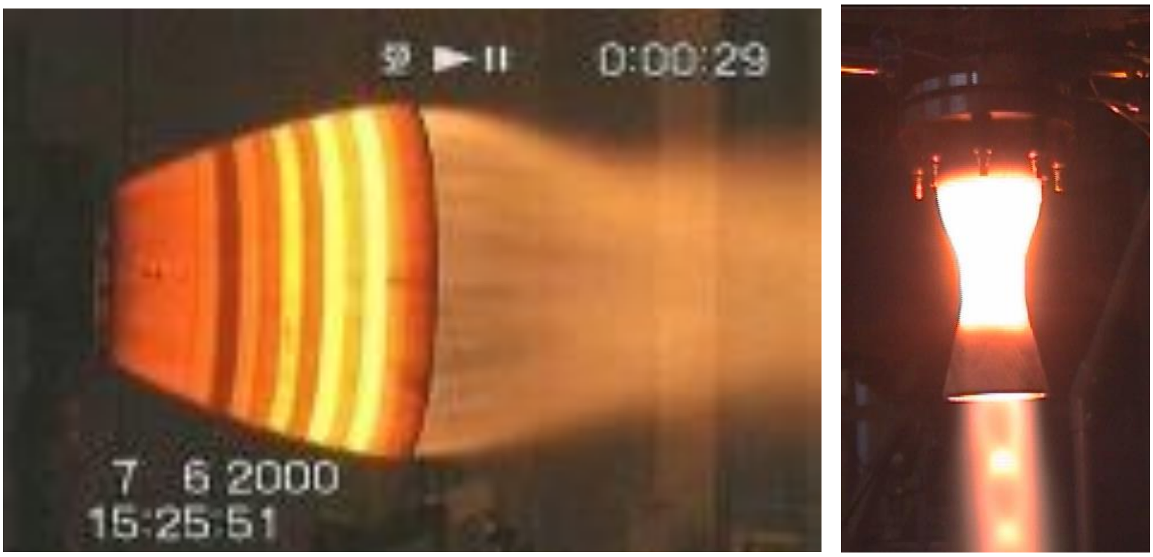

Figure 13: Hot testing of a Vulcain subscale nozzle made of C/SiC (left)
Hot testing of the European Apogee Motor (EAM) made of C/SiC (right)

In the field of CARBOTEX[SI®] material, various demonstrators for nozzle extensions and combustion chambers in PTAH-SOCAR architecture have been produced and successfully hot-tested, as well.

3.4 Thermo-stable Structures

For scientific space exploration missions to sun near orbits, where components are exposed to harsh environments, Airbus DS has developed thermo-stable support structures to carry the sub-reflector of high gain antennas and to guarantee the positioning of the sub-reflector with high accuracy. The arrangement comprising the High Gain Antenna, the sub-reflector and the tetrahedron built by a 2x3 array of C/SiC struts is shown in Figure 14 (left). The challenge was to build a stiff but lightweight structure, being able to withstand high acoustic and mechanical loads during launch and furthermore having a thermal expansion coefficient close to zero in a temperature range between -196 °C and 550 °C. Struts made of filament winded SICARBON® material with a specific fiber lay-up and an innovative clamping mechanism made of Titanium to build an interface to the surrounding structure (shown in Figure 14 (right)) fulfill the mentioned criteria. A verification of the design by conducting mechanical tests (tensile, compression, vibration) accompanied by non-destructive testing via CT has assured the acceptance for flight [10].

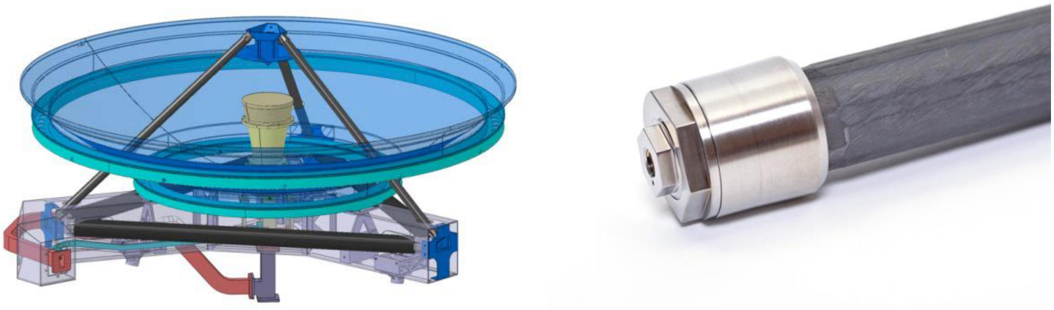

Figure 14: High Gain Antenna arrangement (left). Clamped fitting on C/SiC strut (right).

This application, achieving the acceptance for flight, shows the sophistication of the SICARBON® material and furthermore opens a wide field of applications for CMCs beyond propulsion and TPS.

4. Conclusion

Successful manufacturing and hot testing of manufacturing demonstrators for nozzle extensions for upper- and lower-stage engines as well as for combustion chambers made of C/SiC manufactured via filament winding and PIP show and approve the high potential of the CMC technology. The C/SiC material has reached a high level of maturity, proven by the fact that Airbus DS has already manufactured thermo-stable C/SiC struts as flight hardware for scientific space exploration missions to Mercury and the Sun. This has been enabled by continuous improvement of the whole manufacturing chain beginning at adaption of raw material and textile processing, high temperature treatments, mechanical machining and especially manufacturing accompanying measures related to quality assurance such as non-destructive inspection and dimension control via CT. The establishment of a flexible and cost-effective hot testing rig has been an additional important step to increase the understanding of damage mechanisms in CMCs. For future in-flight applications of highly loaded CMC components, decisive criteria are reproducibility, cost expenditure and the availability of lifetime models for damage prediction. These criteria will have to be satisfied for the qualification of a product.

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