CMC MATERIALS FOR COMBUSTION CHAMBER APPLICATIONS

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ABSTRACT

CMC materials seem to be a promising candidate for cooled, heavily stressed parts of combustion chambers. For the cooled case, transpiration cooling with porous CMCs seems to be a technique with good prospects. To estimate the effect of hot gas flow onto cooled and uncooled CMCs, test campaigns at different combustion environments were performed. Within the framework of the multi-national and EU-funded project "Aerodynamic and Thermal Load Interactions with Lightweight Advanced Materials for High Speed Flight", short ATLLAS, some tests with transpiration cooled CMCs were conducted in a kerosene/GOX combustion chamber. Furthermore, tests with uncooled CMCs under GH2/GOX combustion environment were performed at DLR. This paper illustrates the design and hardware characteristics of the CMC samples investigated as well as first experimental results.

NOMENCLATURE

Acronyms and abbreviations

C/C	carbon fibre reinforced carbon
C/C-SiC	carbon fibre reinforced silicon carbide
CMC	ceramic matrix composite
CTE	coefficient of thermal expansion
DLR	German Aerospace Center
GH2	gaseous hydrogen
GOX	gaseous oxygen
OXIPOL	oxide based CMC based on polymers
TUM	Technische Universität München
WHIPOX	wound highly porous oxide CMC
WPS	Walter Pritzkow Spezialkeramik

Symbols

k_d	Darcy coefficient [m²]
k_f	Forchheimer coefficient [1/m]
L/d	ratio of length and diameter [-]
O/F	mixture ratio of oxidiser and fuel [-]
p	combustion chamber pressure [MPa]
T	hot gas temperature [K]
η	dynamic viscosity [Pa s]
ρ	density [g/cm³]

INTRODUCTION

With the demand of vehicles, both for space travel or commercial aviation, travelling at high supersonic or hypersonic speed, a lot of problems arise. For leading edges and engine components, severe thermal loads occur. The project 'Aerodynamic and Thermal Load Interactions with Lightweight Advanced Materials for High Speed Flight' (ATLLAS) deals with several aspects of high speed flight [1]. Within this framework detailed investigations on cooling techniques as well as thermal, mechanical and chemical compatibility of both metal-based and ceramic materials are conducted.

One aim of future combustion chamber applications is to increase the combustion chamber temperature and hence the thermal efficiency and overall engine performance. Thereby, resulting wall temperatures can easily exceed the capabilities of conventional materials as well as usual cooling techniques. For such applications, lightweight and high-temperature ceramics like lightweight ceramic matrix composites (CMCs) have to be applied alongside with advanced cooling techniques. Hereby, a promising candidate seems to be transpiration cooling [2], which consists of two main effects: First of all, a homogeneous coolant film forms over the surface thickening the boundary layer and reducing the wall heat fluxes. Additionally, the coolant removes an amount of heat from the solid, porous CMCs by internal convective heat transfer.
In ATLLAS, different transpiration cooled non-oxide and oxidic CMCs provided by DLR are tested at Technische Universität München (TUM) under GOX/kerosene combustion environment.

Within the development works at DLR on cryogenically operated ceramic rocket combustion chambers, transpiration cooled inner CMC liners are in the focus of interest [3]. For more than one decade, basic physics have been investigated accurately using carbon/carbon (C/C) as a perfect reference liner material. Because of its thermal and mechanical properties and suitable coolant diffusion behaviour, this material demonstrates a well running process-related functionality. Unfortunately, C/C material offers one disadvantage: At hot gas temperatures significantly higher than 400°C, temporary stochastic existence of free residual oxygen has to be considered; at the same time, carbon based materials show a high sensitivity against oxidation and can degenerate partially. Nevertheless, the porous wall structure with its homogeneous blowing seems to be well suited for transpiration cooling operation. Hence, the material's chemical resistance has to be improved in future. Preliminary tests without cooling have been conducted, which act as a worst case scenario. First results for non-oxide and oxidic CMCs, provided by DLR and WPS, are shown. In addition to CMCs used for transpiration cooled parts, also C/C-SiC was investigated which allows no cooling as it is not permeable.

MATERIALS

Within the scope of this work, several non-oxide (C/C, C/C-SiC) and oxidic CMCs (AvA, OXIPOL, WHIPOX) were investigated.

C/C is an intermediate of the C/C-SiC manufacturing process [4]. This process is characterised as follows: Using commercially available 0|90° carbon fabrics and one-part thermoset resins, a green body of carbon fibre reinforced plastic (CFRP) is formed in the first step by autoclave technique or resin transfer moulding (RTM). After curing, the green body is being pyrolysed at 900°C in nitrogen atmosphere resulting in a C/C body containing patterns of micro cracks. C/C material offers low expansion and specific weight as well as high temperature stability at protecting atmospheres. It can be reproduced very well and has already been tested widely in combustion chamber application. The design and testing of the porous C/C samples has therefore been the main focus for the tests at both DLR and TUM.
In order to obtain C/C-SiC [4], the C/C material has to undergo a second pyrolysis process in vacuum at 1650°C. In the last step, the material has to be siliconised by means of liquid silicon infiltration (LSI) in vacuum at 1650°C.

AvA is an industrial standard CMC material of WPS [5] [6]. It is based on Nextel 0|90° fabrics and oxide matrices and a hand-laminated material with similar fibre lay-up as C/C. Within the sensitivity tests until now only raw AvA has been investigated. For real combustion chamber applications DLR will finish this raw material to a suitable liner structure under the use of further DLR processing steps.

OXIPOL [7] is based on 0|90° fabrics of Nitivy 2626P and a mixture of polysiloxane precursors, cured by a polycondensation reaction. Thereby, the manufacturing process consists of four steps: fibre coating, green body manufacturing via resin transfer moulding (RTM), pyrolysis and oxidation of the fibre coating. The

material has to be pyrolysed several times at 1100°C in nitrogen atmosphere in order to densify the material. After a final oxidisation process, the result is a SiOC matrix with alumina silicate fibres.

WHIPOX [8] is based on N610 fibres and produced by a continuously working winding technique. The roving is infiltrated with a water-based matrix slurry and pre-dried in a microwave. The infiltrated yarns are wound onto a plastic mandrel with a winding angle of ± 45°. In the final step, the green bodies are sintered in air at 1300°C. This results in an alumina oxide matrix with alumina oxide fibres.

EXPERIMENTAL SETUP

Tests at TUM

Within the scope of ATLLAS, the high pressure combustion facility located at the Institute of Flight Propulsion of TUM has been chosen as test bed for transpiration cooling [9] [10] [11]. The facility features a water-cooled single element subscale rocket combustion chamber with an inner diameter of 37 mm, which is operated with kerosene and GOX at pressure levels up to 10 MPa and hot gas temperatures exceeding 3500 K. For the experiments conducted with CMCs, the 3rd and 4th segment of the modular baseline chamber are replaced by the CMC test sample (Fig. 1); at this position, a completed combustion process and a fully developed flow can be ensured which effects in reliable and well-defined test conditions for the investigations on the heat transfer and cooling effectiveness of the CMC test samples. Up to now, only gaseous nitrogen was tested as coolant, as nitrogen serves as an inert model fluid and substitute for air.

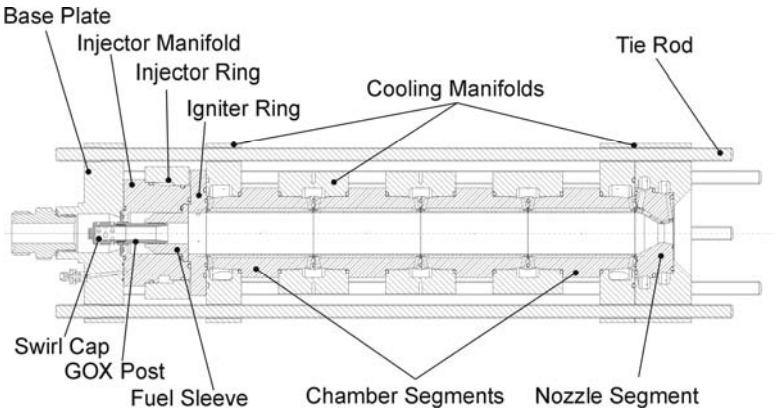

Fig. 1: Sketch of TUM combustion chamber

The setup of the test sample consists of the CMC liner itself and a metallic jacket (see Fig. 2). The coolant is fed through two inlets in the manifold between CMC and metallic jacket. In order to limit the maximum differential pressure, to which the CMC is subjected and to drain the manifold after test, a discharge orifice is provided. Alongside with pressure sensors and thermocouples integrated in the feed and discharge line, a number of thermocouples has been mounted in the CMC liner with a distance of 1 mm to the hot chamber wall in order to estimate the maximum wall temperature.

The tests include cold flow characterization as well as hot fire tests. Cold flow tests prove the sufficient mechanical strength, leak-tightness against ambient and a proper interface design. To estimate coolant flow characteristics and corresponding pressure drops, the CMCs permeability is determined by means of a static approach with constant coolant mass flow steps. The Darcy and Forchheimer coefficients, k_d and k_f, are used to describe the pressure drop dp of the viscous fluid's compressible flow, described by the Forchheimer equation as $dp/dx = \eta/k_d \cdot v + \rho \cdot k_f \cdot v^2$. The permeability measurements before and after each test indicate changes in flow through characteristics and hence material changes, which is a measure of erosion and - if hydrocarbon-based fuels will be used (especially as coolant) - possible coking effects due

to decomposition. Altogether, special attention is paid on the material compatibility and all changes of the material properties under the influence of varying coolant or hot gas flow.

Fig. 3 shows the tested CMC liner materials (via wound technique), which are C/C, OXIPOL and WHIPOX.

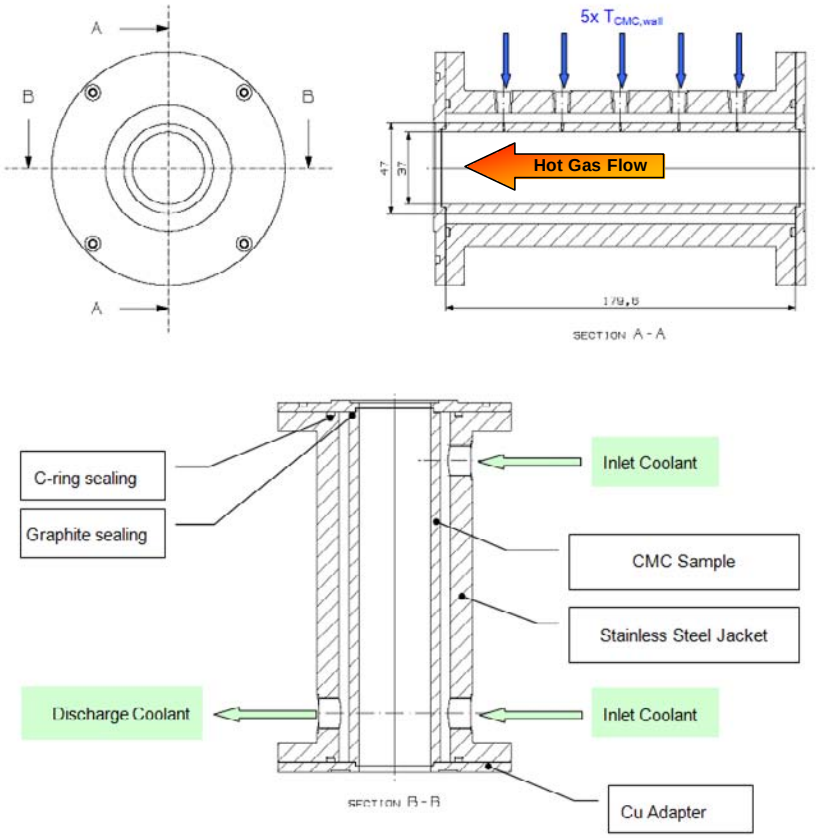

Fig. 2: Transpiration cooled CMC setup at TUM

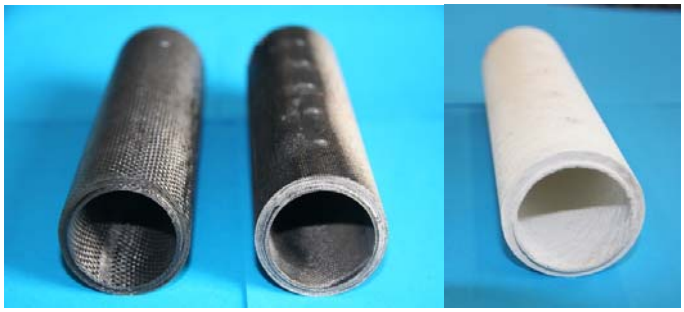

Fig. 3: CMC samples for TUM tests, from left: C/C, OXIPOL, WHIPOX

Tests at DLR

For the hot gas material sensitivity, intensive investigations at the M3 test bench at DLR Lampoldshausen have been performed [12] [13]. A micro-combustor with an inner diameter of 30 mm serves as a sample platform for the sensitivity tests as can be seen in Fig. 4. The test facility is operated with GH2 and GOX at pressure levels up to 2 MPa for the conditions applied. The cylindrical samples with 8 mm diameter are centrically inserted facing directly the hot gas flow without any cooling (Fig. 4, Fig. 5); this equals worst case conditions.

Considering both the high geometrical L/d - ratio of the chamber and the fuel-rich conditions applied, significant amounts of free oxygen can be excluded. It is supposed, that the non-cooled sample is exposed to a fully developed hot gas stream at this position. All CMC sensitivity tests have been performed at a constant combustion chamber pressure of 1 MPa.

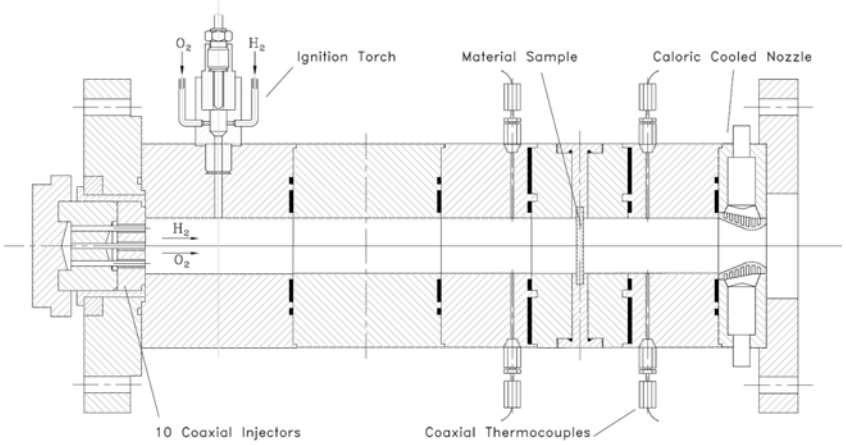

Fig. 4: M3 micro-combustor as a platform for material sensitivity tests

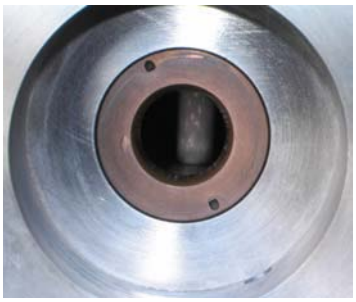

Fig. 5: Inserted sample

Experiences made during the last years confirmed that the basic transpiration cooling liner setup offers good thermo-mechanical and flow properties. On the other side, the chemo-physical properties should be improved without an impact on the basic design principles. So, recently several tests have been conducted in order to investigate the hot gas resistance of CMC alternatives. At this following materials were investigated: C/C, C/C-SiC, AvA, OXIPOL and WHIPOX.

FIRST EXPERIMENTAL RESULTS

Tests at TUM

So far, cold flow experiments as well as first hot fire tests have been conducted with GN2 as transpiration coolant. In Table 1 the measured permeability coefficients are listed, which have been determined by a least square regression analysis.

Table 1: Permeability coefficients at TUM tests

Material	K_d [m²]	K_f [1/m]
C/C, before test	1.122E-12	9.040E+06
C/C, after test	1.121E-12	9.175E+06
WHIPOX	4.225E-13	5.862E+07
OXIPOL	1.267E-14	8.175E+08

As can be seen, the tested C/C sample has been quite permeable leading to very low pressure losses. This is usually not wanted, as a specified pressure difference between transpiration coolant feed line and combustion chamber is demanded in order to separate the feed system from the combustion chamber and to prevent hot gas from penetrating the porous combustion chamber wall. Compared to C/C, the OXIPOL sample has been found to be quite dense resulting in high pressure losses. This may lead to high mechanical loads especially during start-up and shut-down sequence, therefore much care had been taken in designing a suitable test sequence for OXIPOL avoiding overload.
With C/C, four tests with a total test time of 80 s have been conducted so far. All these tests were performed at a mixture ratio of 1.8, a combustion pressure of 2 MPa and a coolant mass flow of approximately 50 g/s nitrogen. The test time has been increased from 5 s up to 30 s in order to determine

the time required to reach steady-state conditions in the CMC. The behaviour of the pressure loss over time of the C/C sample is shown for all four tests in Fig. 6. Obviously, the pressure loss is increasing from roughly 300 mbar up to 750 mbar within a test time of 30 s. Although within limits of measurement accuracy, the slight gradient at the end of the test indicates that a test interval longer than 30 s is required to reach steady-state conditions. An explanation for the increasing pressure loss could be a change of the material and hence permeability. This assumption has not been confirmed as the measured permeability before and after the GN2 tests (no decomposition) did not change, as can be seen in Table 1. Another explanation could be that the CMC material is still heating up as it did not reach steady state conditions yet. As the coolant mass flow rate is constant, the transpired coolant would thereby be convectively heated up more causing an increase of coolant pressure and hence pressure drop, which has to be verified in future. Furthermore, the change of the pressure loss within test time seems to be quite repeatable and the reproducibility of the test data is satisfying.

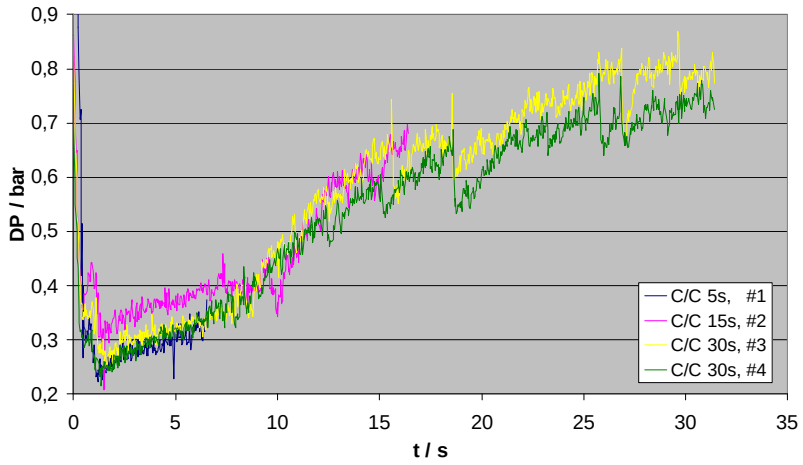

Fig. 6: Pressure loss for C/C liners

Fig. 7 depicts the measured data from the thermocouples integrated in the CMC liner for the third C/C test alongside with the coolant mass flow determined by a Coriolis mass flow meter. The mass flow rate is virtually constant. In contrast, the thermocouples show an inconsistent behaviour. In all tests conducted so far, it has been found that the temperature measurements in the CMC chamber wall are very sensitive to the actual mounting position and test history. The experiments showed evidence that the thermocouples, which are glued into the CMC liner, tend to detach. Additional tests on this issue are planned.

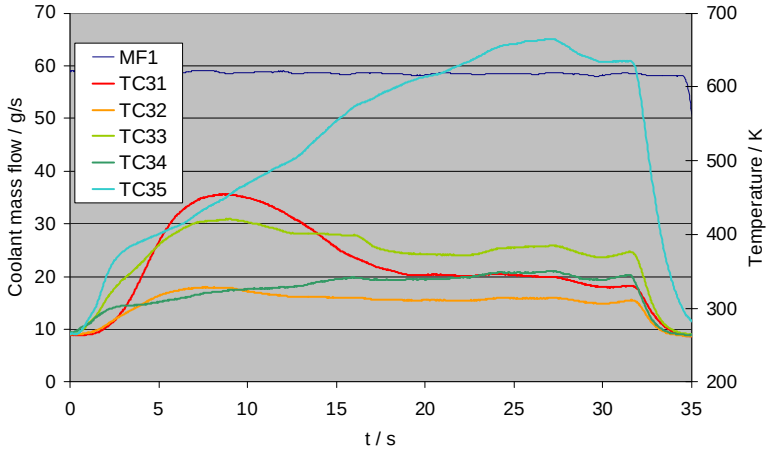

Fig. 7: Thermocouple temperatures for C/C

WHIPOX and OXIPOL have both been tested in a short time hot fire test at a mixture ratio of 1.8, a combustion pressure of 2 MPa and a coolant mass flow of approximately 50 g/s nitrogen. The results of these experiments are currently under investigation.

Tests at DLR

A representative extraction of the wide test campaign is given in Table 2. The maximum test time has been 15 s, due to restrictions of the capacitive cooled combustion chamber; the mixture ratio was varied between 1.0 and 2.4. Hereby, the corresponding hot gas temperatures have been calculated using the well-known approach of Gordon and McBride [14] assuming equilibrium conditions.

Table 2: M3 test data

Material	Sample no.	Sample orientation	p / MPa	T / K	t / s
C/C	CC-1	⊥	1.0	2200	9
C/C-SiC	CS-1	II	1.0	2200	10
C/C-SiC	CS-2	II	1.0	2700	10
OXIPOL	O-1	⊥	1.0	1400	15
OXIPOL	O-2	⊥	1.0	1600	15
OXIPOL	O-3	⊥	1.0	1800	15
WHIPOX	W-1	⊥	1.0	1800	10
WHIPOX	W-2	II	1.0	1800	10
AvA-Z	AZ-1	⊥	1.0	1800	10
AvA-Z	AZ-2	⊥	1.0	2000	10
AvA-A	AA-1	II	1.0	2000	10

The samples were generally mounted in two different orientations: Either perpendicular (⊥), where the hot gas flow collides normal to the fibre layers, or parallel (II), where the hot gas flows alongside the fibre layers. Displayed samples without marks are perpendicular ones, samples marked with 'II' are parallel ones. First phenomenological effects will be described by means of a preliminary visual inspection:

C/C degenerates without any melting phases, as can be seen in Fig. 8. Abrasive gas stream attacks combined with carbon gasification are probable, leading to material erosion.

OXIPOL contains silicon dioxide which is extracted under the applied temperatures. Fig. 9 shows significant growing SiO_2 traces and finally extensive erosion accompanied by molten SiO_2.

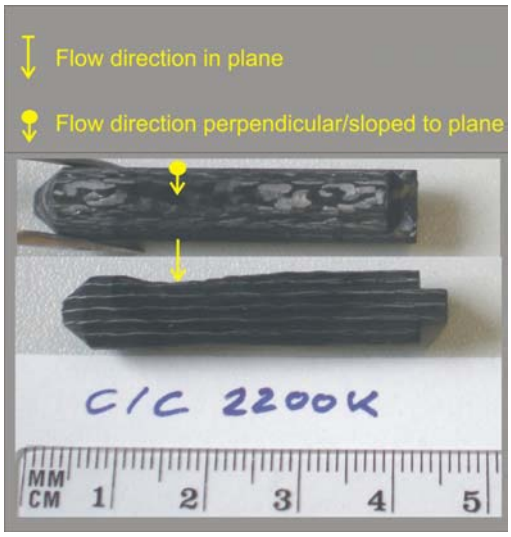

Fig. 8: C/C sample (⊥) after two tests

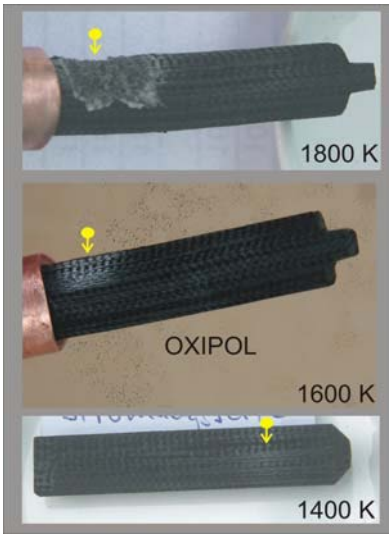

Fig. 9: OXIPOL (⊥) samples after tests

In principle, C/C-SiC resists high temperatures. First indications of changes inside the material are observable by separation of little pure silicon drops at the side edges and rear edge of the sample. Fig. 10

and Fig. 11 show a series of two different operation temperatures, 2200 K and 2700 K, respectively. At lower temperature levels (2200 K) only brown coloured traces are recognizable as well as the discharging of silicon drops, an effect of further de-siliconisation. The geometry is still contour stable. On higher temperature levels significant traces of molten SiO_2 are visible and erosion begins.

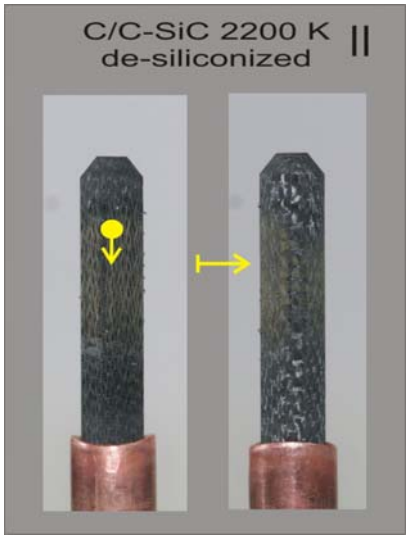

Fig. 10: C/C-SiC sample (II) after tests (2200 K)

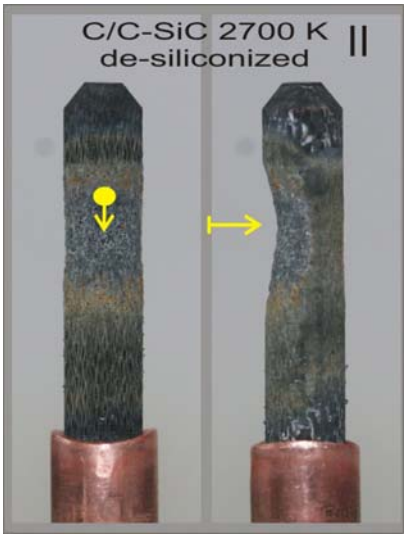

Fig. 11: C/C-SiC sample (II) after tests (2700 K)

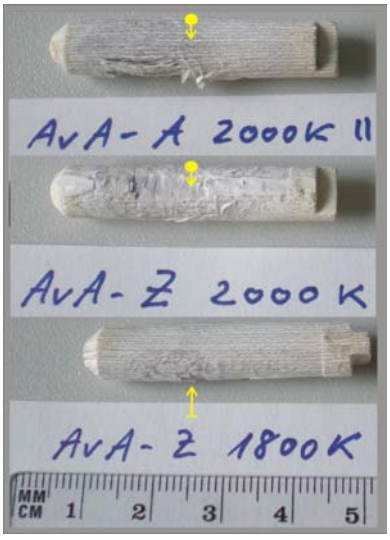

Fig. 12: AvA samples (II and ⊥) after tests

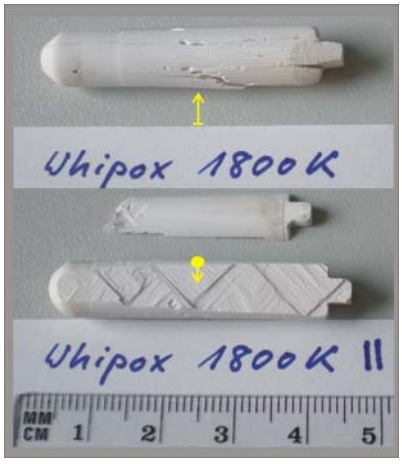

Fig. 13: WHIPOX (II and ⊥) samples after tests

The effect on AvA materials (AvA-A and AvA-Z) can be seen in Fig. 12. It can be explained by a thermo-mechanically point of view: The fibre bundles expand rapidly (high CTE) and lift off from the surface due to low intra- and interlaminar strength. This results in compressive failure of the material.

For WHIPOX typically the same effect as for AvA can be observed (Fig. 13) as the CMCs are very similar.

Altogether it can be summarised, that apart from different temperature levels applied the cardinal damage difference between non-oxide and oxidic CMC material does not consist in an erosion mechanism, but in a thermo-mechanical failure mechanism.
The evaluation of all oxidic samples shows, that for parallel inflows bigger delaminations can be observed than for the perpendicular inflows. Thereby, WHIPOX seems to be more sensitive than AvA, as bigger

fragments seemed to be broken apart. In principle, it is supposed that different heat distributions caused by orthotropic material properties lead to different fracture patterns, especially for the oxidic CMCs.
At 2000 K, for AvA-A smaller fragments broke apart than for AvA-Z. Comparing AvA-Z at 1800 K and 2000 K, the attacked sample surface at 2000 K is significantly larger than expected. Up to now, it could not be reproduced or verified, whether melting phases appeared at the surface at this temperature level.
A general difference between AvA and WHIPOX seems to be the black colouration of AvA in reducing atmosphere, which WHIPOX does not show at 1800 K. A possible explanation is that the components of the AvA-matrices seem to be sensitive against a $H_2O/H_2/OH$ atmosphere at 1800 K. This is not considered to be critical for short-term applications, but this effect could be very dangerous for long-term applications.

As a matter of course, lower temperature levels can be applied on the oxidic CMCs compared to the non-oxide ones. But these temperatures are still higher than the maximum operation temperatures of typical metals used in current propulsion systems. Additional advantages as for instance good diffusion properties, sweet-tempered mechanical properties as well as favourable oxidation-resistance are the base for an introduction of such a material class into hypersonic and space propulsion research.

UPCOMING EXPERIMENTAL ACTIVITIES

The test activities just have started at both facilities. Within the ATLLAS programme, further TUM test campaigns will include transpiration cooling testing with redesigned CMC liners based on a new, stratified design. In addition to gaseous nitrogen, kerosene will be applied as coolant in order to detect possible coking effects due to decomposition. For the tests with kerosene cooling, thermal and mechanical loads will be increased whereas the coolant mass flow is decreased by a stepwise approach.
At DLR, further tests on the sensitivity and thermo-chemical resistance, respectively, of CMCs will be performed as well.

SUMMARY

Different non-oxide and oxidic material was designed and manufactured by DLR and WPS, respectively.

Within the multi-national EU-project ATLLAS, dealing with several aspects of high speed flight, experimental as well as numerical investigations on novel cooling techniques and advanced materials are carried out. The investigations of newly developed transpiration cooled CMCs are summarised here. Such CMCs are candidate materials for future ramjet and scramjet, as well as rocket combustion chamber applications. Several tests are conducted at the high pressure combustion facility of TUM under kerosene/GOX environment. The experimental activities as well as cross-checks with the numerical tools have started but are still on-going.

At DLR's facility M3, dedicated uncooled CMC samples have been exposed to main hot gases $H_2O/H_2/OH$ in terms of a worst case condition (resulting from GH2/GOX combustion). Preliminary results show, that non-oxide CMCs tend to degenerate by means of erosion effects, whereas oxidic CMCs are weak with regard to thermo-mechanical stability, which has to be considered in future designs.

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Framework Programme Priority 1.4, Aeronautic and Space, contract no.: AST5-CT-2006-030729. Further info on ATLLAS can be found on: www.esa.int/techresources/atllas.

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