Lightweight Hot Structures from Oxide Ceramic Matrix Composites

J. Schmidt, Ch. Eckardt, A. Rüdinger

Oxide CMC (O-CMC) are currently used mainly in various industries due to their low weight, high ductility and high strength up to high temperatures of around 1200 °C. The most common manufacturing techniques already used in industry are filament winding and lamination. The increasingly used textile fibre preforms with aligned fibres and joints allow the manufacturing of more complex designs and enable a variety of novel geometries. The Fraunhofer Center for High-Temperature Materials and Design (HTL) develops new CMC technologies and products in joint projects with industrial partners.

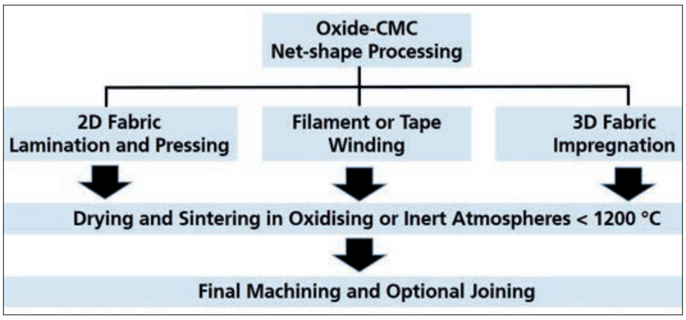

Fig. 1 Flow chart of the state-of-art manufacturing processes for the production of oxide CMC parts with different net-shape geometries

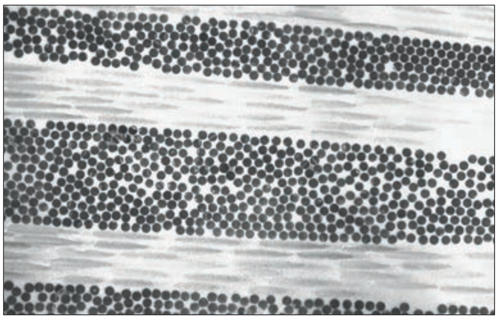

Fig. 2 Typical microstructure of O-CMC with completely matrix impregnated oxide fibre bundles

State-of-the-art manufacturing

O-CMC are used worldwide for furnace equipment, burners as well as aerospace applications [1]. The companies familiar with this technology use hand-made or semi-automatic processes for small-scale production. The three basic manufacturing processes are shown in Fig. 1. Simple geometries such as plates and tubes are produced by laminating 2D fabrics in combination with axial pressing or filament winding. Complex geometries can be made by joining of pre-machined parts and require an additional sintering step [2].

A typical composite microstructure can be derived from Fig. 2. The oxide fibres are embedded and uniformly distributed in a porous oxide matrix. The net-shape production of 2D- and 3D structures is focusing more and more on aligning the load bearing fibres and saving fibre costs [3]. However, the infiltration of powder-based slurries into dry textile preforms still implies potential for optimization. Impregnated preforms produced by Liquid Polymer Infiltration (LPI) or Resin Transfer Moulding (RTM) have a high mass loss during their pyrolysis requiring many reinfiltration steps to obtain dense matrices [4].

3M™ oxide fibre grades, Nextel™ 610 and Nextel™ 720, have excellent tensile strength of up to 3000 MPa and are therefore used in most of today's O-CMC applications [5]. Fraunhofer Center HTL developed a material family called CerOx from these fibres. Typical properties from Type AZ-N6-F at room temperature are presented in Tab. 1.

Tab. 1 Properties of CerOx type AZ-N6-F developed at the HTL

3-point bending strength	320 MPa
Young's modulus	86 GPa
Strain to failure	0,36 %
Interlaminar shear strength	17 MPa
Open porosity	29 vol.-%
Density	3,02 g/cm^3
Thermal conductivity	6,3 W/(m·K)
Coefficient of thermal expansion	$5{,}5 \cdot 10^{-6}$ 1/K
Specific heat	0,7 J/(g·K)
Maximum operating temperature	1150 °C

Jens Schmidt, Christian Eckardt,
Arne Rüdinger
Fraunhofer-Institute for Silicate Research – ISC, Center for High-Temperature Materials and Design – HTL
95448 Bayreuth
Germany

Corresponding author: J. Schmidt
E-mail: jens.schmidt@isc.fraunhofer.de

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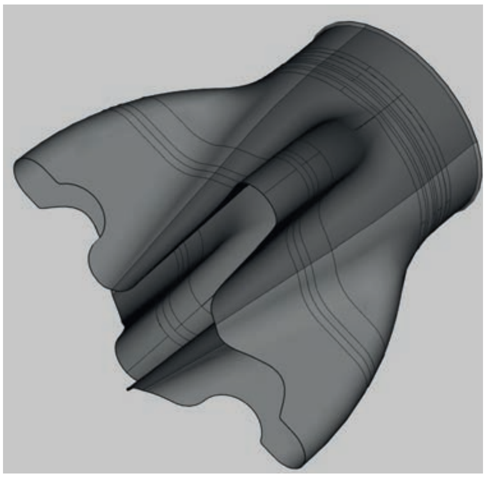

Fig. 3 a Exhaust mixer design study

Fig. 3 b Plastic positive mould for laminating with pre-cut prepregs

Fig. 3 c Laminated thin-walled oxide CMC mixer prototype

Exhaust mixer structures for aircrafts

The use of CMC in the hot gas area of aero-engines enables a significant increase in the permissible working temperature compared to metallic components. At the same time, the weight of the turbine is reduced due to the lighter CMC material. Both factors contribute directly to environmentally friendly aviation. The exhaust mixer is a feature of many turbofan engines, where the bypass of cold and slow air is mixed with the hot and fast exhaust gases from the core, before it is discharged into the atmosphere (Fig. 3 a–c). The mixer reduces the velocity of the air present in the core and thus also the noise. For the manufacturing of prototypes of a thin-walled exhaust mixer, the well-known manufacturing technology of laminating 2D oxide fibre fabrics with aqueous powder suspensions can be used.

In order to obtain a material with high strength, the Nextel™ 610 fibres with matrices based on Al_2O_3–SiO_2 or Al_2O_3–ZrO_2 and sintering temperatures <1200 °C were investigated. The mechanical properties of specimens derived from flat plates were determined according to international aviation certified standards such as ASTM C1292 for Iosipescu shear strength, ASTM C1358 for compression and ASTM C1359 for tensile strength. Mean strength values of 77 MPa for Iosipescu shear, 388 MPa for compression and 207 MPa for tensile were obtained. They are in the accepted range for the manufacturing of prototype parts.

The work at Fraunhofer-Center HTL to realise the complex mixer design is part of the project CMC-TurbAn [6]. First, male and female moulds for axial pressing were designed based on CAD data sets. These moulds were made from large plastic parts with a 5-axis machining centre. The required prepregs were prepared from dry 2D fabrics and matrix slurries. A roll-to-roll machine was used for continuous impregnation of the fabric with the slurry using the foulard technique (Fig. 4). To achieve high homogeneity, the rheological properties of the suspensions, the particle sizes and the drying behaviour of the suspensions were adjusted in such a way that the suspensions could be homogeneously introduced into the fibre bundles without damaging the fabric.

The prepreg roll was cut into prepreg plys with defined geometries using a programmable 2D cutter (Fig. 5). For manual lamination of the predefined prepreg plys onto the mould surface, the lay-up pattern was defined. The fibre volume content was set by the adjustment of both moulds during the pressing process. In order to save material costs during the development phase, low-temperature sintering ceramic matrices and silica fibre fabrics were chosen to produce prototypical CMC mixer segments.

Fig. 4 Continuously operating roll-to-roll machine for production of prepregs by impregnation of oxide slurries
(Copyright: Mathis AG)

Fig. 5 Automatic cutting table for pre-cutting of individual coupons from the prepreg roll

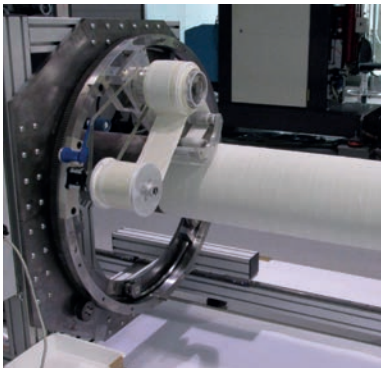

Fig. 6 Manufacturing study: automated machine for endless lay-up of wet prepreg tapes on straight metal pipes

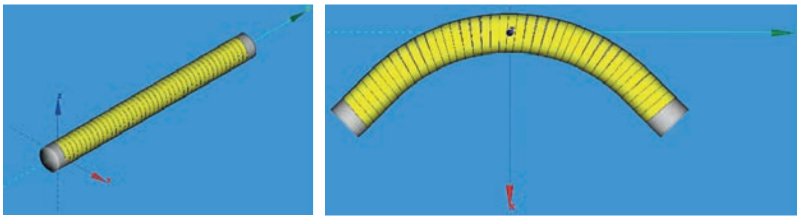

Fig. 7 Simulation of the fibre tape lay-up on straight and curved metal cores using the winding program CADWIND

Fig. 8 Hybrid pipe elbow (Ø~320 mm) with an O-CMC ceramic fibre jacket (t= 20 mm) with a total length ~4 m installed in a coal fired power plant

Currently, a complete mixer consisting of several segments is built up and application-related tests are planned to validate the current design.

Metal tubes with ceramic fibre jacket for power plants

New concepts for steam power plants still require high temperature and creep resistant materials for their heat transfer system. These plants could work more efficiently, if the superheated steam temperature and internal pressure could be increased. As an enveloping support structure, creep-resistant ceramic composites have the potential to effectively impede the creep deformations of steel pipelines, when hot gas temperature and pressure are increased. The concept of a hybrid metal-CMC system was successfully demonstrated.

Within the project Compound Tubes [7] it was shown in lab-scale experiments that the creep of hot gas steam tubes made of steel could be significantly reduced by using the CMC jacket [8]. However, the use in a plant environment places further demanding requirements on the design of ceramic jackets, such as the pipe curvature, changing pipe diameters as well as pipe exits. In the follow-up project, it was possible to solve many of these technical difficulties [9]. In this project, a pipe elbow was constructed as a test carrier with a diameter of 320 mm and a total length of approx 4 m and manufactured with a ceramic jacket [10]. In the first step, wet-prepregs based on almost unidirectional tapes of Nextel™ fibres were produced in the prepreg process and wound with a semi-automated laminating machine to simulate the lay-up (Fig. 6). Polysiloxan resin with boron nitride particles as fillers was used as the matrix. The prepreg-tapes were placed on the pipe elbow with an overlap of the tapes in the inner radius to obtain a face-to face lay-up in the outer radius (Fig. 7). This was the only way to avoid gaps between the individual tape layers. In addition, a ceramic fibre fleece was applied between the inner metal pipe and the outer CMC jacket as an interface to reduce thermal stresses due to the mismatch of thermal expansion. The laminated pipe was subsequently pyrolysed to convert the polymer matrix into a stable SiOC ceramic. The ceramic conversion was performed at low temperature <800 °C. The hybrid pipe with multiple sensors was installed in a coal fired power plant (Grosskraftwerk Mannheim/DE) and was tested successfully for more than three years (Fig. 8).

Net-shape airfoils based on 3D textiles

Lightweight and robust components are required to further decrease energy consumption in hot gas engines or high temperature furnace processes. By using CMC, the density can be reduced up to one third compared to conventional nickel alloys or steel. Oxidation and corrosion problems in the combustion environment can be overcome. In addition, with its high temperature resistance, CMC is a promising candidate for more efficient combustion processes. Airfoils are key components for fan wheels in turbine engines. The shape of such components is very complex, and thin-walled parts are needed. Moreover, the ceramic fibres have to be aligned in the load-bearing direction, so that the part can withstand the high thermomechanical loads.

Within the project AirfOx [11], textile preforms for airfoils are produced in a single step. Highly sensitive oxide fibres are manufactured to woven net-shaped 3D preforms by using a double rapier weaving machine. For flexible structure design, the plant is equipped with a Texmer creel for a balanced yarn tension, a warp beam harness for the draw warp and an Unival jacquard device for individual control of the more than 4000 warp threads. It also has special rapiers for processing the brittle ceramic fibres and a

Fig. 9 Airfoil shape made from load-bearing 3D fibre textile preform

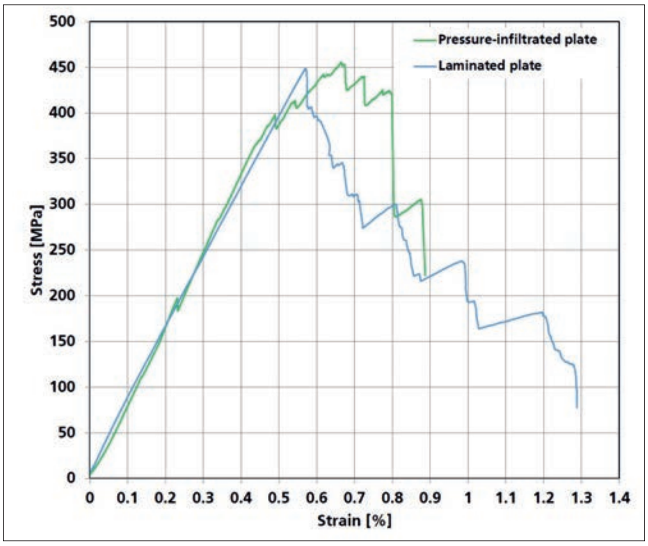

Fig. 10 Characteristic stress-strain curves of the pressure-infiltrated and the laminated O-CMC plate in comparison

linear take-off for pressure-sensitive multilayer structures [3]. In the project, a load-adapted near-net shape airfoil preform with a multilayer structure is being developed to achieve the required wall thickness of <2 mm.

Computer simulation of the bond structure as well as the final loads in application support the development of an optimized load-bearing fibre preform (Fig. 9). The project is currently developing a multi-stage pressure-assisted infiltration process for complex 3D fibre preforms. The aim is to effectively stabilise the contour of the flexible textile preform and to perform a complete and homogeneous matrix infiltration. Initial tests with the pressure assisted infiltration process were carried out with flat fibre preforms produced by textile technology. An oxide ceramic slip based on ZrO_2/Al_2O_3 was used for the matrix infiltration. The plate-shaped composite bodies obtained in this way were subsequently dried and sintered. Mechanical characterisation of these plates was carried out via three-point bending tests according to DIN EN 658-3.

The resulting stress-strain curves are shown in Fig. 10 in comparison with a conventional O-CMC laminate based on matrix-infiltrated single fabric layers. It can be seen from the plot that the pressure-infiltrated O-CMC plate has a similar strength level to the conventionally laminated plate in the range of 450 MPa, which can be taken as evidence of very good matrix infiltration.

Joint O-CMC-alumina hybrid lances

Specific sensors can help to obtain as much information as possible from high temperature processes to make these processes more efficient. A gas measuring lance was developed for use in high-temperature furnaces that can determine temperature, gas composition and gas flow.

Firstly, the functional test was carried out in a roller furnace, followed by a field test in an incineration power plant in Sweden

Fig. 11 Use of the ceramic lance for measuring the gas flow and gas composition in a waste incineration plant at MälarEnergi in Västerås/SE

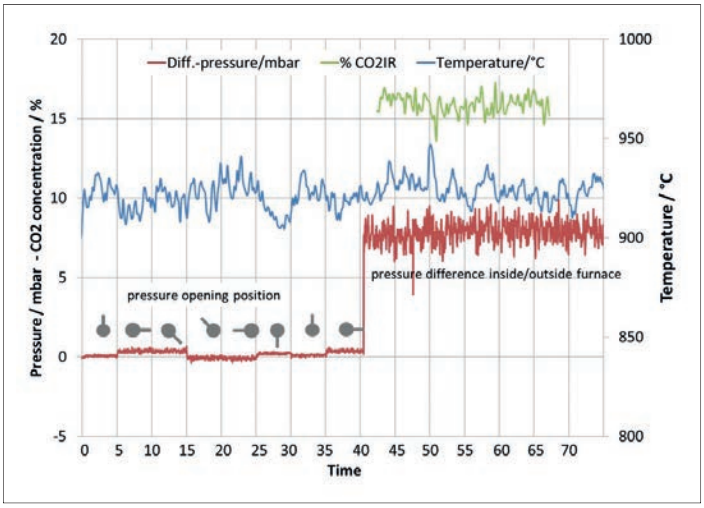

Fig. 12 Measurement of pressure, temperature and CO_2 concentration with the hybrid lance inside the furnace

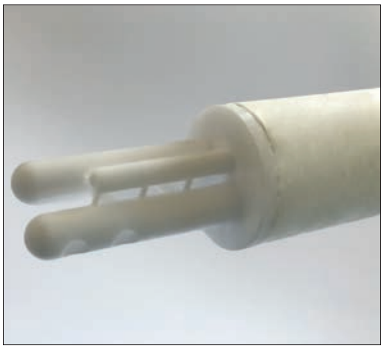

Fig. 13 **Measuring tips for temperatures and gases made of alumina by 3D printing joined to the head of O-CMC lances**

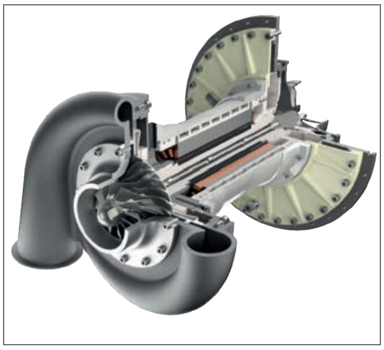

Fig. 14 **Schematic drawing of a micro gas turbine including the demonstrator "stator" on the front side (Copyright Euro-K)**

Fig. 15 **Laminating study of a stator for micro gas turbines made of woven silica fabric and Al_2O_3–SiO_2 matrix**

(Fig. 11). As part of the European project FuDiPO, on site measurements of pressure, temperature and CO_2 concentration were carried out with the ceramic lance [12, 13] (Fig. 12).

Moreover, gases can be sampled and measured with an external mobile test stand. The structure of the ceramic lance is a material hybrid which combines an O-CMC tube, various alumina measuring tips and a metal grip. The O-CMC tubes were manufactured using filament winding, whereas the ceramic tips were made by 3D printing (Fig. 13). The integrated inner tubes, which are also made of alumina, contain the thermocouples.

All materials were precisely machined before joining. To achieve the complex ceramic geometry in combination with the required length of about 1,5 m, joinings were made. These joints with high durability were made by form-fit and material bonding. Specific glass solders with the composition SiO_2–MgO–CaO–Al_2O_3 were used for the material bonding and surface sealing, as these crystallise at high temperatures and form high temperature stable ceramic phases such as spinel [14].

Stators for micro gas turbines

The development of the stator for micro gas turbines is based on several interconnected as well as consecutive elements along the value chain (Fig. 14). For the final purpose of creating a properly designed O-CMC stator, several technological problems need to be solved.

The challenge of the project doMiGat was to design a stator made from O-CMC, which is highly dense and which exhibits a high strength up to 1250 °C [15]. The material must work in burning gas atmospheres with a certain water vapour content. Until now, O-CMCs have a matrix porosity which is around 30 vol.-%. The large porosity contributes to a weak fibre-matrix interface and enables a quasi-ductile fracture behaviour.

On the other hand, the porosity might cause the mechanical instability of the component at high temperatures and increase the risk of penetration of hot corrosive gases into the component.

To overcome these problems, material improvements are required. A wet-chemical fibre coating technology was used to protect the fibres against degradation [16]. Since the weak-interface concept needs a low-friction coating on the fibres' surface if the matrix is dense with low porosity, a thin coating made of aluminium phosphate was applied on the Nextel™ 720 fibres.

In addition to sealing the open porosity – lower than 10 vol.-% – of the matrix by infiltration and pyrolysis of silicone resins, moreover, a surface coating based on a crystallizing ceramic-glass solder was applied to close the open porosity on the outer surface. Though the quasi-ductile fracture behaviour remained intact, it could be shown in mechanical tests that both procedures – densification by PIP together with fibre coating or sealing the surface – did not reduce either the strength nor the strain-to-failure.

To show the viability of the manufacturing concept, silica fabric prepregs were used to obtain a prototype (Fig. 15). The material will be tested in turbine environments in a next step.

Conclusions

Technical progress in the manufacturing of oxide CMC has been achieved in recent years due to the transformation from manual to semi-automated processes. Meanwhile, improved textile techniques can process brittle oxide ceramic fibres to produce 2D- and 3D preforms. 2D preforms from these processes can either be impregnated with oxide slurries by a foulard process to obtain prepregs, followed by a lamination when large parts are required. Automatic cutting of prepregs to the desired size and shape facilitates the homogeneity and quality of the process and the product. 3D preforms can be infiltrated directly with pressure support. Both techniques allow the complete impregnation of matrices into the fibre bundles of dry textile preforms and enable the production of near-net-shape components with high reproducibility. Joining technologies are available when very long or geometrical complex parts are required. More recently, low-cost CMC have been developed. They are based on cheaper raw materials such as silica or basalt fibres and a sintering process below 1000 °C [17]. Considering use of automated production processes, oxide CMCs are becoming increasingly competitive with metal structures.

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