Development, Manufacture and Characterization of C/C-SiC Components Based on Filament Winding

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Abstract

In this contribution development, manufacture and characterization of C/C-SiC tubes based on different winding angles is described. Therefore, CFRP tubes were made by wet-winding of C-filaments using thermoset resins with high char yield and, in a second step were converted to C/C tubes via pyrolysis. Then, the porous tubes were infiltrated by liquid silicon providing C/C-SiC tubes which were characterized by their microstructure and mechanical properties. Mechanical testing was performed under tensile loading in axial and circumferential direction (burst test). In addition, a correlation of mechanical properties to the winding angle will be presented.

1. Introduction

Ceramic matrix composites (CMC) are ideal candidates for applications in hot gas environment in aerospace (e.g. rocket propulsion) due to their superior properties such as high hardness, abrasion, heat and thermal shock resistance in combination with high damage tolerance in case of excessive loading [1-7].

In short term application C-fibre based composites, such as carbon-silicon carbide composites (C/C-SiC) developed by DLR using the well-known LSI-process are superior to other CMC materials due to their ease of manufacture, variability of raw materials and cost. Thus, there is a huge potential for future rocket propulsion applications [8-9]. State of the art is the use of commercially available fabrics to manufacture complex shaped CMC components with low porosity in one cycle, providing moderate strength materials, or even lower performance materials based on short fibres applied on cost-efficient components where low strength can be accepted [10]. However, for applications requiring high specific strength components of tubular shape, a new approach combining wet filament winding technique with the LSI-route to provide a cost and time efficient process is obvious, but still is a real challenge for material scientists and engineers.

2. Experimental procedure

C/C-SiC composite tubes as well as plates were manufactured by a three-step process: 1) CFRP green body shaping by wet filament winding and pressure-less curing on the mandrel, 2) pyrolysis to a porous C/C preform and 3) densification and build-up of SiC matrix by melt infiltration.
1) The manufacture of structures with rotational symmetry requiring high specific strength in main load direction, such as combustion chambers, affords the use of winding machines and fibres of high specific strength. Therefore, a wet filament cross winding process was developed using continuous T800 12k carbon fibres, which were impregnated prior to winding with a phenolic resin (JK60) and wound onto a rotating mandrel (ø = 120 mm and 160 mm with a length of about 1000 mm) in a predetermined pattern to form an interweaved regular laminate using winding angles of ±15°, ±30, ±45°, ±60° and ±75°, respectively. Since mechanical characterization is much easier to achieve on plate than on tubular samples, a part of the uncured winding form was cut along the rotation-axis and then unrolled to a plate shape. The uncured composite preforms (tubes and plates) were then cured at ambient pressure to achieve a predetermined fibre volume content of about 60 %.
2) The polymer based CFRP green body tubes as well as plates were pyrolysed in an inert gas atmosphere at temperatures up to 1650°C leading to a porous C/C preform. In case of tubes graphite mandrels with shrinkage adapted diameters were used up to 900°C.
3) Finally, the porous, fibre reinforced preform (C/C) was infiltrated with molten silicon in vacuum at up to 1650°C. The molten silicon reacted with the carbon and formed the ceramic SiC matrix.

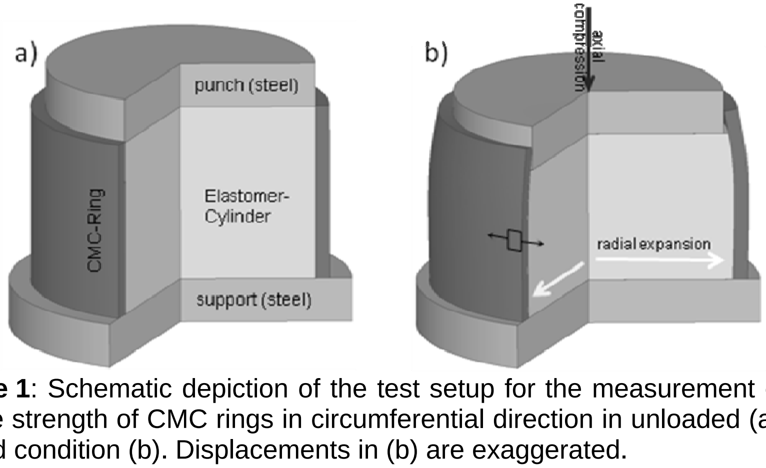

Figure 1: Schematic depiction of the test setup for the measurement of the tensile strength of CMC rings in circumferential direction in unloaded (a) and loaded condition (b). Displacements in (b) are exaggerated.

Density and open porosity of the C/C-SiC composite plates were determined in all stages by Archimedes method. Tensile strength (at room temperature) of C/C-SiC specimens made of pipe and plate material was determined by using a (Frank-Zwick-81120) universal testing machine. Extensometer measurements of the specimen elongation were used to determine the tensile modulus and the elongation at fracture. Typical dimensions of the tensile test specimens were 200x10x5mm. If not otherwise stated reference is the winding angle or due to practical reasons the loading direction.

Figure 2: Waisted specimens for the measurement of tensile strength in axial direction with typical dimensions indicated were extracted from CMC pipes by water jet cutting. Similar specimens were extracted from plate material with orientations parallel and transverse to the original axial direction.

In order to measure the tensile strength of CMC ring-specimens in circumferential direction, a test setup similar to the compressed elastomer method delineated in [11] was used. Compared to testing methods using fluids, the compressed elastomer method (schematically depicted in Fig. 1) enables a simplified internal pressure loading of ring specimens because no seals are required. The CMC ring is filled with an elastomer cylinder, which is compressed in axial direction by a servo-hydraulic testing machine. If the elastomer is assumed to have negligible resistance against shear deformation and high resistance against volume change (incompressible behaviour) the inner surface of the CMC ring is loaded with a radial pressure which is equal to the contact pressure between steel punch and elastomer [11]. The accuracy of this test method is limited e.g. due to the following reasons:

- stress-strain gradient in thickness direction of the ring,
- the shear modulus of the elastomer is not completely negligible,
- shear stress transfer between elastomer and CMC ring by friction,
- constraint effects of steel punch / support on elastomer due to friction,
- inhomogeneous stress distribution in axial direction close to the load-free upper edge of the CMC ring, where the steel punch enters into the ring.

For the specimen geometries tested, the influences of these effects were investigated by finite-element simulations. It was found that inaccuracies are below 15%. Thus, the accuracy of compressed elastomer method is lower than the accuracy of tensile tests but it is still a useful screening test.

The microstructure of the C/C-SiC composites was investigated by means of scanning electron microscopy (SEM, Zeiss Ultra 55) for each fibre orientation as mentioned above. Furthermore, selected samples were tested in two directions (axial (ax) and tangential (tan)) in respect to fibre orientation.

3. Results and discussion

Table 1 provides typical ranges of densities and open porosities of the filament wound composites in all stages. Increased open porosity of CFRP (and C/C) is due to pressure-less curing of phenolic resin based on condensation.

Table 1. Density and open porosity of C/C-SiC composite tubes.

Material	CFRP	C/C	C/C-SiC
Density [g/cm³]	1.36 - 1.54	1.26 - 1.37	1.98 - 2.07
Open porosity [vol.-%]	6.55 - 15.6	16.7 - 25.8	1.23 - 2.74

Physical as well as preliminary mechanical properties of C/C-SiC tubes, derived from tensile tests, are shown in Tab. 2. Typical strength-strain values of each tested fibre orientation are shown in Fig. 3. As expected from CFRP, tensile strength and modulus increased with winding angle as well as decreasing fibre orientations with respect to load direction. These findings can be used for a first design of real components.

Table 2. Mechanical properties, density and open porosity of C/C-SiC composite tubes and plates at various winding angles.

Winding angle [°]	15	30	45	60	75
Tensile strength (ax) [MPa]	161	147	64	27	23
Tensile modulus (ax) [GPa]	123	73	25	20	35
Elongation at fracture (ax) [%]	0.16	0.30	0.59	0.41	0.08
Tensile strength (tan) [MPa]	13	43	105	149	155
Tensile modulus (tan) [GPa]	23	24	15	54	128
Elongation at fracture (tan) [%]	0.10	0.80	0.70	0.28	0.15
Density [g/cm³]	1.99	2.07	2.02	1.99	1.98
Open porosity [vol.-%]	1.34	1.23	2.74	1.34	1.91

Typical microstructures of C/C-SiC composite tubes are shown in Fig. 4 and 5 revealing the distribution of carbon fibres, C (dark), SiC and Si (bright) in dependence of the winding angle. As expected from the manufacture of fabrics intended segmentation of C/C-bundles was observed. However, C/C segments reveal some SiC formation inside, which is probably due to increased porosity in CFRP. Nevertheless, the winding angle does not influence microstructure and ceramic content of C/C-SiC significantly, which is confirmed by mechanical testing.

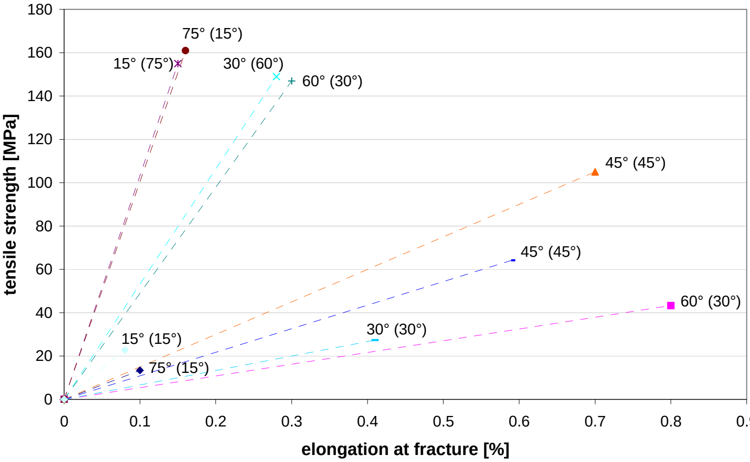

Figure 3. Typical strength-strain values of C/C-SiC samples with different fibre orientations: given angle denotes fibre orientation in loading direction, winding angle in brackets, (dotted lines serve clear arrangement, only).

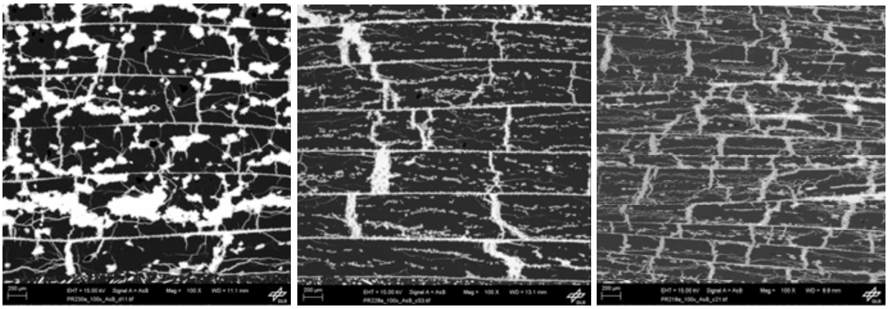

Figure 4. SEM micrographs of polished surfaces of C/C-SiC wound at angles: ±15° (l.), ±30° (m.) and ±45° (r.), view in axial direction (magn. 100x).

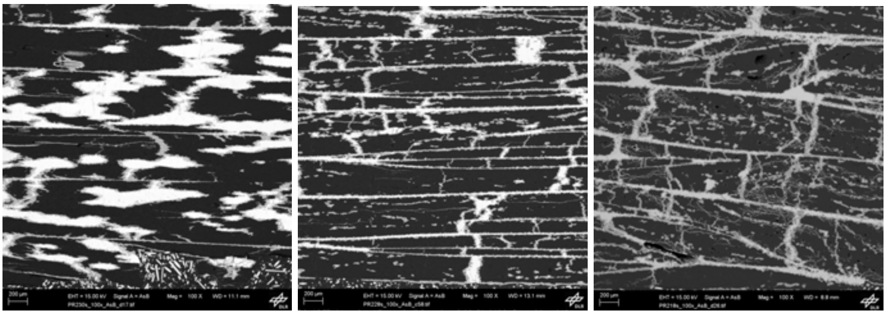

Figure 5. SEM micrographs of polished surfaces of C/C-SiC wound at angles: ±15° (l.), ±30° (m.) and ±45° (r.), view in tan direction (magn. 100x).

4. Conclusion

C/C-SiC composite tubes (ø=120mm) based on filament winding of T800 fibres and LSI route were successfully manufactured and mechanically tested for the first time. Preliminary results show how fibre orientation influences mechanical properties and microstructure of C/C-SiC. Although on a lower level, results of mechanical screening tests are in accordance with well-known CFRP knowledge and will allow a first cautious design of high performance future rocket motor components, for e.g. combustion chambers, of tubular shape. Furthermore, the results of this work will be continued to obtain reliable reference data for increasing desire of customers on components in this upcoming application field.

Acknowledgement

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