Comparison of Oxide/Oxide Ceramic Matrix Composites based on either woven fabric or bi-directional laminates

Walter Pritzkow (*), Thomas Krause (**), Kamen Tushtev (**), Dietmar Koch (**)

(*) Walter E.C. Pritzkow Spezialkeramik, Stuttgart
(**) Advanced Ceramics Group, University of Bremen, Bremen

1. Introduction

Most Oxide/Oxide Ceramic Matrix Composites (Ox/Ox-CMC) are based on bidirectional (2D) oriented alumina or mullite fibres and matrices in the system of alumina, silica, zirconia. The two main manufacturing technologies are either winding of matrix infiltrated rovings or knife blade slurry impregnation of woven fabric layers and subsequent stacking. While these structures show fibre bundle intersections and undulations, the layup of unidirectional (UD) layers in 2D orientation would lead to even fibre bundle alignment which could improve the mechanical performance of these composites. A newly developed method to produce such UD layers used for production of a multiply 2D layup is presented. The mechanical properties of two 0°/90° reinforced composites manufactured from layup of 2D fabrics and UD plies, respectively, are compared. The composites manufactured by Pritzkow Spezialkeramik consist of 3M™ Nextel™ Roving 610/1500 denier and Al_2O_3-8YSZ matrix. While the 3M™ Nextel™ woven fabric type DF11 has a fixed fibre volume of 11 roving per cm in the direction of 0° and 90°, the number of rovings per cm in the UD plies are varied with 9, 10 and 11. For the layup of the UD plies the same fibre volume in the direction of 0° and 90° are used. Shear properties were evaluated with tensile tests on the Ox/Ox-CMC and tensile tests on the fabrics and rovings. Short beam bending tests were used for investigation for interlaminar fracture behaviour. The influence of fibre intersections, undulations and matrix properties in these regions are studied. Optical methods are used to evaluate the fracture behaviour dependent on these different composite layups.

2. Experimentals

2.1 Material

For this investigation an Ox/Ox-CMC is used based on watersized rovings of alumina fibre type 3M™ Nextel™ 610/1500 denier. The matrix consists of Al_2O_3 powders, Al_2O_3-binder sol and an 8YSZ-infiltration sol, developed by Fraunhofer-ISC [1-3]. As this composite has a porous and weak matrix with low strength and stiffness it may be attributed as a Weak Matrix Composite (WMC) where the properties of the fibre-matrix interface are of minor importance for providing enhanced fracture behaviour [4,5]. The microstructural design is comparable to the design of Levi et al. [6] with a matrix based on Al_2O_3 powder and surrounding Al_2O_3-8YSZ binder around the fibres.
The Ox/Ox-CMC designed and manufactured by Pritzkow Spezialkeramik uses either fabric type DF11 woven as eight-harness satin fabric or UD-plies. The textile structures were impregnated with the paste-like Al_2O_3 slurry by knife blade coating. Layers were stacked together (fig. 1), pressed and dried whilst the subsequent sintering process was followed by an infiltration with an 8YSZ-sol and a second sintering process for 5 hours at a temperature of 1200℃.

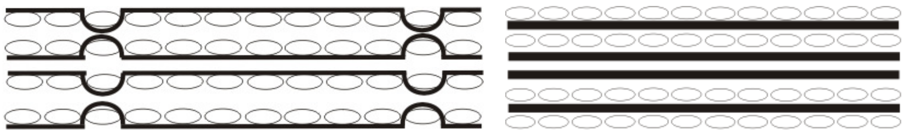

Figure 1. 4-layer fabric layup (left) and 8-layer UD-ply layup (right) both presenting a 0°/90° fibre orientation.

The composite GW11 is produced by use of four layers of fabric DF11 which has 11 rovings per cm in the direction of both warp and weft. The UD-ply layup was varied using 11 rovings per cm (UD11) and additionally also layups of 10 and 9 rovings per cm (UD10 and UD09, resp.). The UD-plies are produced by winding the roving on a square tube in 0° oriented stacks. Each layer was infiltrated individually with the same matrix as the fabrics. The resulting components had a dimension of about 200 x 200 mm² and thicknesses between 1 and 1.3 mm depending on number of rovings per cm.
Microstructural investigations show that in the fabric based Ox/Ox-CMC GW11 the fibre bundles are not perfectly impregnated and larger pores can be found between the layers. This is mainly due to the undulation of the fibre bundles in this type of fabrics which impedes a homogeneous impregnation

with a paste-like matrix. The UD11 layup showed poor impregnation of fibre bundles and large delaminations between the unidirectional layers as the matrix could not be placed within and between the close packed fibre bundles. When the fibre bundles are less compacted as it is the case in UD10 and UD09 the matrix could be well distributed in and around the fibre bundles. While the UD10 layup shows a homogeneous matrix distribution the UD09 allows a higher concentration of pure matrix regions due to the larger distance between the fibre bundles.

2.2 Test procedures

The fabrics and the fibre rovings were tested in order to compare the properties of the untreated state with the in-situ mechanical strength of the fibres in the composites after heat treatment of desizing and sintering.
Tensile tests on the fabrics were done by the ITCF-Denkendorf based upon ASTM D-5035. The fabric samples had a size of 38 x 150 mm with the edges unraveled to 25 mm. The test device was set up with a gauge length of 75 mm and a crosshead speed of 12 mm/min.
Tensile tests on the fibre rovings were carried out using a Zwick 1465 50 kN device at a speed of 1 mm/min. Dogbone like specimens with width of 20 mm were prepared with a gauge section of 10 mm. Strain was recorded via laser extensometry.
For the interlaminar shear strength, short beam four point bending was applied on a Zwick Z005 tensile machine at 1 mm/min. Distances of the upper and lower rolls were 4.1 and 10 mm with a roll diameter of 1.5 mm. These values are chosen in order to achieve a span to thickness ratio around 5 to provoke interlaminar failure in the thin specimens.

3. Results and Discussion

3.1 Properties of Fabrics and Rovings after Heat Treatment

To understand the results of the mechanical properties of the Ox/Ox-CMC the mechanical properties of the 3M™ Nextel™ 610 textiles and rovings are tested individually. To compare the characteristics presented by 3M™ [7] with the used fabrics and roving after simulated processing treatment the textiles were heat treated at 800°C for 1 hour according to the desizing process, and then were heated at 1200°C for 5 hours which correspond to the sintering process. The failure load of the fabric was calculated based on ASTM D-5035.

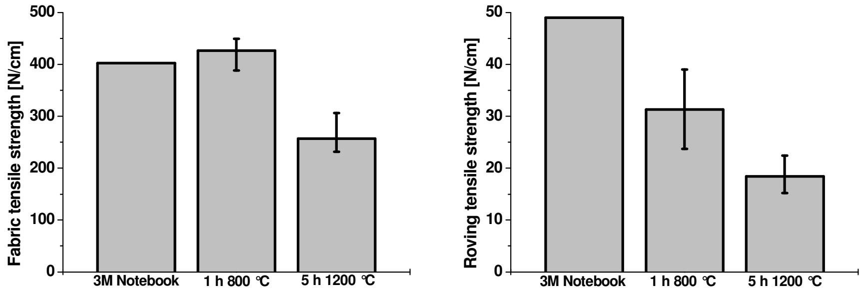

Figure 2. Failure load per cm of the fabric (left) and roving (right) after heat treatment in comparison to manufacturer data sheet values [7].

In figure 2 it is shown that the desizing temperature does not reduce mechanical properties of the fabric significantly while the sintering temperature treatment results in a drop of mechanical strength of the fabric from about 400 N/cm to about 260 N/cm. The reduction of measured strength values from treatment at 800°C to treatment at 1200°C was calculated to 60%.
Testing of rovings showed much lower values compared to manufacturer data sheet values [7] as the sizing had already been removed before testing, resulting in a fan out of the roving. Therefore it was not possible to load all filaments simultaneously during the tensile test. However a comparison of the values after 800°C and 1200°C, resp. show a strength reduction of 59% which is equal to the reduction of strength of the fabrics.

3.2 Tensile Tests on Composites

The results of tensile tests on the varied Ox/Ox-CMC are shown in figure 3. The failure load values are calculated from the strength values referring to the gauge width. In comparison expected failure loads are calculated from the number of rovings in loading direction using the results from fabric and roving tests.
The UD09 specimens show high scattering of mechanical strength due to inhomogeneity during manufacturing. As the rovings are placed pretty loose the orientation in the composite was not as accurate as in case of the other types UD10 and UD11. UD10 specimens show a higher failure load than expected from roving data which indicates that due to the successful impregnation of the layers with matrix additional load can be carried by the matrix resulting in higher strength. In case of UD11 the results of the composite tests

well coincide with the values calculated from roving tests. This shows that all fibres oriented in loading direction are able to carry the load. The same observations can be made for the GW11 specimens. The fabric composites present lower failure loads compared to the UD-composites, however these loads are to be expected from the fabric tests (figure 2).

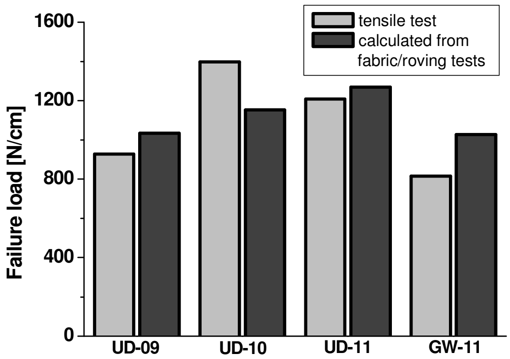

Figure 3. Tensile failure load of 0°/90° UD-ply and fabric based Ox/Ox-CMC and comparison with failure loads calculated from results of roving and fabric testing.

3.3 Results of Interlaminar Shear Tests

The short four point bending test allows a confident interpretation of the matrix properties between the layers if the specimens fail in shear mode. In all specimens valid failure behaviour was observed. The composites UD09 and GW11 show moderate interlaminar shear strength indicating that after matrix impregnation some pores between the layers remained (figure 4). As already observed in microstructural analysis the matrix incorporation in type UD11 was very difficult thus interlaminar shear strength is very low. The best interlaminar properties are measured in case of UD10 composites where the matrix could be impregnated in good quality. For UD10 shear strength values beyond 10MPa could be achieved.

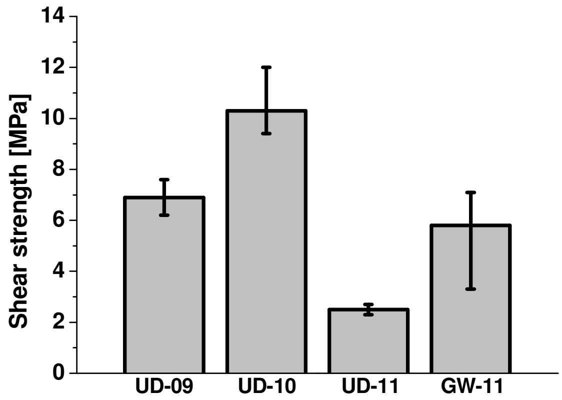

Figure 4. Interlaminar shear strength of 0/90 UD-ply and fabric based Ox/OxCMC.

4. Discussion and Conclusions

Ox/Ox-CMC with different fibre volume content had been produced and the effect of fabric compared to unidirectional layers laminated in 0°/90° had been investigated. For reasonable comparison of the tensile tests with different fibre-volume contents, the strength results were converted to failure load per width of the specimen considering the number of rovings per cm and the number of layers in loading direction (four layers).

If it is assumed that during tensile loading all mechanical load is carried by the 0° fibres only, then a comparison of composite properties with fibre properties can be drawn. In order to do this the fabric as well as the rovings were tested after thermal treatment which is comparable to the sintering treatment during manufacturing of the oxide composites. The results show that the failure loads expected from fabric and roving tests are the same as the measured tensile failure loads of all types of composites except UD10. For these composites it can be concluded that the matrix is not able to carry significant load as the matrix impregnation during processing was not homogeneous enough. While in case of composites GW11 and UD11 the matrix could not be filled in a sufficient manner between the woven cloth and the densely packed rovings, respectively, in case of UD09 a large content of voids and porosity remained after matrix impregnation due to the loose assembly of the rovings. In case of UD10, however, it becomes obvious that the strength of the composite is higher than calculated from the roving tests. The reason for

that is that the matrix is distributed nicely in the composite allowing stress transfer from fibres to matrix and vice versa.
The interlaminar shear strength tests can be interpreted in good agreement with the tensile test results. The UD10 specimens show good interlaminar properties while the other composite variations which are characterised by imperfect matrix impregnation show minor interlaminar properties. The results show that the interlaminar properties are defined by the matrix properties while a possible influence of woven fabric compared to unidirectional reinforcement could not be demonstrated.
It can be concluded that the processing of oxide composites with a paste like matrix needs a very specified free volume between fibres for good impregnation. If this is assured excellent mechanical properties in tensile as well as in interlaminar loading can be achieved and stress transfer between fibre and matrix can be realised which is mandatory for damage tolerant behaviour.

5. Acknowledgement

The authors are grateful to the Institut für Textilchemie und Chemiefasern (ITCF) Denkendorf for carrying out the tensile tests on the fabrics.

6. Literature

1. A. Rüdinger, W. Glaubitt, W. Pritzkow, *Verbundwerkstoffe und Werkstoffverbunde* (Ed.: M Schlimmer) Wiley-VCH. Weinheim, **2005**, p. 261-264
2. A. Rüdinger, W. Glaubitt, *cfi/Ber. DKG 82 No. 13*, **2005**, 51-54
3. W. Glaubitt, A. Rüdinger, DE 10 2006 011 224 A1, **2007**
4. F. W. Zok and C. G. Levi, *Adv. Eng. Mater., 3[1-2],* **2001**, p. 15-23
5. D. Koch, K. Tushtev and G. Grathwohl, *Compos. Sci. Tech. 68*, **2008**, p. 1165-1172.
6. Levi C.G., Yang J.Y., Dalgleish B.J., Zok F.W. and Evans, A.G., *J. Am. Ceram. Soc., 81 (8)*, **1998**, p. 2077-2086
7. 3M™ Nextel™ Ceramic Textiles Technical Notebook S. 28