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### Inspection of geometry influence and fiber orientation to characteristic value for short fiber reinforced ceramic matrix composite under bending load

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### ABSTRACT

For use of short fiber reinforced ceramics the knowledge of the influence of coupon geometry on the failure mode and the determined resistance in bending tests is necessary. In contrast to the continuous fiber reinforced ceramic matrix composites (CMC), for short fiber reinforced CMC there are only few studies and no standard on consideration of size effects. In the present work, the influence of coupon geometry and test conditions on the average value and distribution of flexural strength has been investigated. Two short fiber CMCs with different fiber length were examined under four point bending load. Moreover, the relationship between fiber orientation in the loaded area, failure location and measured flexural strength was investigated through high resolution X-ray computer tomography ( $\mu$ CT) and SEM. The presented outputs will be proposed to a future standard for bending test of short fiber reinforced CMC materials with different fiber length.

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### 1. Introduction and objective

Due to its non-brittle mechanical behavior and its resistance against severe environments (temperature, corrosive atmospheres, etc.), CMCs have a high potential for applications in different high tech fields [1]. However, the lack of standards and inadequate databases limit the use of these newly developed materials [2].

For long fiber reinforced carbon/carbon (C/C) and carbon reinforced silicon carbide (C/C-SiC) material with  $0^{\circ}/90^{\circ}$  fiber orientation, the ratio between loading span and coupon thickness shows minor influence on three-point bending strength [3,4]. For short fiber reinforced ceramics, however, only few studies of characterization of mechanical properties are published. It has been observed that under shear load, different coupon sizes of short fiber reinforced C/SiC composite lead to different failure mechanisms between losipescu- and asymmetric four point bend shear tests [5]. For tensile strength measurements of C/C-SiC a dependency on cross section has been reported in [6] and the notch sensitivity of same material under tensile loading is extremely low [7]. High resistance to cyclic fatigue under four point bending loads of these materials have been investigated in [8]. The oxidation kinetics

http://dx.doi.org/10.1016/j.jeurceramsoc.2016.11.042 0955-2219/© 2016 Elsevier Ltd. All rights reserved. and its impact on the bending strength of short C/C-SiC ceramics showed a strong dependency on fiber orientation and length [9]. Significant strength decrease after exposure to high temperature was found in material with long cut fiber and orientation in parallel to the beam axis [9]. In three-point (3-P) and four-point (4-P) bending modes, no clear tendency of flexural strength as a function of coupon size had been observed [10,11]. Due to these different properties and failure behavior, neither the testing standard for bending strength of ceramic composites with continuous fibers [12,13] nor the testing standard for monolithic ceramics [14,15] are suitable for determination of flexural strength of the short fiber reinforced CMCs. Nevertheless, up to now there is no valid international standard for the mechanical testing of these materials.

The determination of material properties of random distributed short fiber reinforced composites has always been challenging due to the large scatter of material properties. Although the random orientation of the short fibers results in isotropic material behavior in large volume structures, the typically small volume of extracted testing coupons usually results in a non-isotropic fiber orientation and fiber content within the coupon [16–18]. Therefore, it is essential to include the knowledge of the actual local fiber distribution within the coupon volume in order to ensure a valid interpretation of the obtained mechanical properties. A reliable and practical method for determining the orientation and distribution of fiber bundles can be performed using volumetric



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### Table 1 Composite parameters.

	Fiber length [mm]	Fiber volume content [%]	Open porosity [%]	Density [g/cm <sup>3</sup> ]	Fiber diameter [µm]	Filaments per bundle [-]
SFC	3	50	2.6	2.3	7	3k, 6k, 12k
LFC	10	40	3.6	2.3	7	3k

#### Table 2

Coupon geometry and experimental parameters of composite SFC with fiber length of 3 mm.

Coupon numbering	Coupon geo	Coupon geometry [mm]		$(L_o - L_i)/t$ [-]	Number of coupons [-]
	t	w	1		
3-3-50	3	3	50	6.7	5
3-6-50	3	6			5
3-9-50	3	9			10
3-12-50	3	12			5
3-15-50	3	15			5
6-6-50	6	6	50	3.3	4
6-9-50	6	9			5
6-12-50	6	12			5
6-15-50	6	15			5
6-18-50	6	18			5
3-3-90	3	3	90	20.0	4
3-6-90	3	6			5
3-9-90	3	9			10
3-12-90	3	12			5
3-15-90	3	15			5
6-6-90	6	6	90	10.0	5
6-9-90	6	9			5
6-12-90	6	12			5
6-15-90	6	15			5
6-18-90	6	18			5

micro-computed tomography analysis [19–21]. Computed tomography data provides the complete volumetric data of the coupon and can discern between fibers and matrix components [19–22]. Using volumetric algorithms such as the structural tensor analysis, an analysis of the quantitative determination of fiber and fiber bundle orientation is possible [23,24]. This can determine the location and orientation of all fiber bundles and thereby permits a correlation between local fiber orientation and strength [18].

Therefore, the main objective of this paper is to determine the influence of coupon-geometry and fiber orientation on characteristic mechanical properties of short fiber reinforced CMCs. Extensive investigations with varied width, thickness and length of coupon are carried out under bending load. The strength and the standard deviation of strength as well as the failure modes are investigated.

### 2. Material and experiment

### 2.1. Material description and test coupon

Two short fiber reinforced C/SiC composites with different fiber lengths were selected, short fiber composite (SFC) with 3 mm fiber length and a fiber composite with longer fiber size 10 mm (LFC), which were produced by a three-stage process. First, the short carbon fibers were mixed together in dry state with phenolic resin and a small amount of solvent until they formed a homogeneous mixture. After molding this mixture was pressed into a carbon fiber reinforced polymer at around 200 °C. In a subsequent carbonization stage, the so-called green body was pyrolyzed at about 900 °C in inert atmosphere and the phenolic resin matrix was converted into carbon. Finally, liquid silicon was infiltrated into the porous C/C body at temperature above 1400 °C to form silicon carbide after reaction. The coupons were cut from the composite plates (approx. 230 mm \* 230 mm with different thickness) using waterjet cutting method. According to the report about investigation of cuttinginduced damage in CMC bend bars [25], waterjet cutting had no significant effect on the mechanical properties. The open porosity and density of the materials were determined with the help of the Archimedes principle [26]. The material parameters are summarized in Table 1. In SFC filament bundles with three different sizes (3k, 6k and 12k) were mixed together while in LFC only 3k filaments were used.

The geometry of SFC bending coupons is listed in Table 2 The thickness (t) is either 3 mm (equal to fiber length) or 6 mm (double as fiber length) and the width (w) of coupon varies from 3 mm (equal to fiber length) to 18 mm (six times to fiber length). The coupons were planned for two different testing parameters. A coupon length (1) of 50 mm was chosen for 4-P bending test with  $L_0 = 40 \text{ mm}(L_0 \text{ is distance between supporting rollers in Fig. 1})$  while coupons with length of 90 mm were used for 4-P-bending test with  $L_0 = 80$  mm. The two different lengths of coupon lead to different span-to-thickness ratio  $(L_0 - L_i)/t$  from 3.3 to 20  $(L_i$  for distance between loading rollers in Fig. 1). The numbering of coupon in the first column followed the rule of thickness-width-length(t - w - l). As mentioned above, the coupons were cut from several composite plates. Due to the varied coupon geometries, different numbers of coupons per series were prepared and tested. The number of SFC-coupons for different geometry is summarized in Table 2. The influence of different numbers of SFC-coupons on the mechanical properties is discussed in Section 3.1.3.

To test the influence of fiber length, a second set of coupons was manufactured using 10 mm short fibers. Table 3 contains the coupon geometry of composite LFC with fiber length of 10 mm. The thickness (*t*) is either 5 mm (half the fiber length) or 10 mm (equal to fiber length) and the width (*w*) of coupon is from 5 mm (half the fiber length) to 40 mm (four times the fiber length). The span-to-thickness ratio  $(L_o - L_i)/t$ varies from 2 to 12. Similar to SFC, t - w - l was used to numbering of the coupon and different numbers of coupons per series were prepared and tested. The number of LFC-coupons for different geometry is summarized in Table 3 and the influence of different numbers of LFC-coupons on the mechanical properties is discussed in section 3.2.3.

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