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# Fatigue behavior and residual strength evolution of 2.5D C/C-SiC composites



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Yang Li<sup>a,b</sup>, Peng Xiao<sup>a,\*</sup>, Heng Luo<sup>a</sup>, Renato S.M. Almeida<sup>c</sup>, Zhuan Li<sup>a,\*</sup>, Wei Zhou<sup>a</sup>, Alexander Brückner<sup>d</sup>, Florian Reichert<sup>b</sup>, Nico Langhof<sup>b</sup>, Walter Krenkel<sup>b</sup>

<sup>a</sup> State Key Laboratory of Powder Metallurgy, Central South University, Changsha 410083, PR China

<sup>b</sup> Ceramic Materials Engineering, University of Bayreuth, Bayreuth, 95447, Germany

<sup>c</sup> Advanced Ceramics, University of Bremen, Bremen, 28359, Germany

<sup>d</sup> Polymer Engineering, University of Bayreuth, Bayreuth, 95447, Germany

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#### ABSTRACT

The residual tensile strength (RTS) evolution of a chemical vapor infiltration and liquid silicon infiltration based 2.5 dimensional reinforced C/C-SiC (2.5D C/C-SiC) composites after fatigue loadings has been investigated. The results show that the fatigue limit (10<sup>6</sup> cycles) of the 2.5D C/C-SiC composites reaches 58.2 MPa, which corresponds to 75% of the virgin static tensile strength (77.7 MPa). Moreover, an ultimate strength enhancement is observed after fatigue loading. The most pronounced RTS increases to 92.5 MPa when specimens are subjected to fatigue stress of 69.3 MPa for 10<sup>5</sup> cycles. The microstructural analysis indicates that RTS after cyclic loading is affected by the formation and propagation of cracks and interfacial degradation. Furthermore, a model proposed in this work can well evaluate the RTS of the composites in relation to the number of the applied fatigue cycles.

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#### 1. Introduction

Ceramic matrix composites (CMCs) show high strength with considerable fracture toughness, and are initially developed for aeronautical purposes [1,2]. Nowadays, the related processing methods have reached a high level of reproduction, and since the 1990s, the liquid silicon infiltration (LSI) method has allowed the production of C/C-SiC composites with relatively low costs [3,4]. Therefore, their areas of application have been widened to industrial applications like ceramic brake discs for sports cars and luxury sedans [5]. In this context, the needled carbon fabric preforms are promising candidates for the production of 2.5 dimensional C/C-SiC composites (2.5D C/C-SiC). These composites show excellent mechanical properties, good coefficient of friction, relatively low manufacture cost and excellent humidity tolerance in tribological applications [6]. Hence, the 2.5D C/C-SiC composites present enormous potential applications in high-speed and heavy-duty brake systems [6-8]. Currently, most works regarding the 2.5D C/C-SiC materials are focused on the topics of cost reduction, mechanical optimization, friction behaviors and even industrial applications for service and emergency brake in the trains or aircrafts [9–14]. However, there are still few investigations concentrating on the influence of dynamic loadings on the long-term mechanical performances of these 2.5D C/C-SiC composites. It is generally believed that the cyclic stresses during practical service can lead to the degradation of strength and even catastrophic failures in CMCs [15–18]. Thus, it is of great significance to get a comprehensive investigation on the mechanical behaviors of the 2.5D C/C-SiC composites under dynamic loads. Therefore, this work aims to study the fatigue behavior and the evolution of residual tensile strength (RTS) of the 2.5D C/C-SiC composites subjected to different fatigue loadings. Experimentally, the fatigue behavior including the stress versus cycles to failure (S-N curves), RTS after preselected cyclic loadings were investigated. Moreover, the microstructures and fractured surfaces were characterized by optical microscope (OM) and scanning electron microscope (SEM) to study the fatigue damage behavior. Finally, a mechanical model was introduced, based on the experimental results, to describe the RTS evolution of the 2.5D C/C-SiC composites in relation to the number of fatigue cycles.

\* Corresponding authors. *E-mail addresses:* xiaopeng@csu.edu.cn (P. Xiao), lizhuan@csu.edu.cn (Z. Li).

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#### 2. Materials and experiments

#### 2.1. Fabrication of the composites

The fabrication process of 2.5D C/C-SiC composites consisted of the following three steps as shown in Fig. 1. The commercially available polyacrylonitrile (PAN) based carbon fiber (Toray, Japan, T700, 12k) were applied as raw material for the 2.5D fiber preforms. Firstly, the short fiber web and the unidirectional fiber cloth were repeatedly stacked and afterwards needle-punched. The needle punching density was 15–35 pin cm<sup>-2</sup> and the bulk density of the 2.5D fiber preform was approximately  $0.65 \text{ g cm}^{-3}$ . Afterwards, the chemical vapor infiltration (CVI) process at 1000 °C for 100-120 h in an argon atmosphere with the absolute pressure of 0.1 MPa was performed. The C<sub>3</sub>H<sub>6</sub> was applied as a precursor and H<sub>2</sub> as a carrier and diluting gas  $(C_3H_6/H_2 = 10 \text{ ml} \text{min}^{-1}$ :  $H_2 = 20 \text{ ml} \text{min}^{-1}$ ). The final step was the preparation of the C/C-SiC by an infiltration of liquid silicon (LSI) into the porous C/C material. The silicon powders (Da Zelin-silicon Co., LTD, Beijing, China) with the particle size of approximately 50 µm and a purity of 99.0% were applied for LSI, and the process was carried out at 1650 °C with 0.5 h dwell time under vacuum conditions (absolute pressure <1 Pa). More details about the fabrication process of the 2.5D C/C-SiC was reported previously [7].

#### 2.2. Initial characterization

The initial characterizations were done concerning the porosity, bulk density and virgin tensile strength. Therefore, all specimens were machined to obtain a grinded and polished surface. The open porosity and the bulk density of the as-processed 2.5D C/C-SiC composites were measured by the Archimedes method with distilled water, following the standard DIN EN 1389. The dog-bone shaped specimens for the tensile tests were prepared by wire eroding with the general dimensions, which can be seen in Fig. 2. In order to avoid grip slipping during the tensile loading, the clamped portions of the specimens were coated by a cured adhesive (allcon10 original, Beko, Germany) with a thickness of about 0.75 mm. Quasi-static tensile tests were performed on a Zwick/Roell test machine (Z1485, Makro, 250 kN) with a crosshead speed of 1 mm/min, following the standard DIN EN 658-1.

#### 2.3. Tensile fatigue tests

According to the standard ASTM C1360, the tensile-tensile fatigue experiments were performed following a sinusoidal wave with the frequency of 10 Hz and the fatigue stress ratio of 0.1 on a servo hydraulic testing machine (IST Hydro pulse MHF). In this work, three sets of cyclic loadings were performed on the specimens to study the fatigue behaviors. Initially, within the "Set 1", the specimens were tested with five different stresses (75%, 80%, 85%, 90% and 95% of the initial tensile strength, corresponding to about 58.2, 62.1, 66, 69.3 and 73.8 MPa, respectively) to define the fatigue limit. The specimens were cyclically loaded until total failure or until run-out, defined here as 10<sup>6</sup> cycles, was reached. During the tests, a period of approximately 1000 cycles was required to achieve the desired maximum stress.

Afterwards, the fatigue tests were performed to analyze the stress dependence of RTS within the "Set 2". To ensure that the samples would not fail during the fatigue tests, the run-out was reduced to 10<sup>5</sup> cycles. For these tests, four stress levels ranging from 75% to 90% were applied. In addition, within "Set 3", fatigue cycles with different numbers of pre-selected cycles (10<sup>4</sup>, 10<sup>5</sup> and 10<sup>6</sup>, respectively) were performed at the stress level of 75% (58.2 MPa). Three sets of tests were performed in a strain-control mode with the parameters shown in Table 1. Quasi-static tensile tests were

then carried out, following the aforementioned procedure, in order to measure the RTS after fatigue loadings.

Finally, the microstructure, the fractured surfaces and the type of fracture were studied by an optical microscope (Axiotech HAL100, Zeiss) and a scanning electron microscope (SEM, FEI Nova Nano SEM-230).

#### 3. Results and discussion

### 3.1. Fatigue life time of the 2.5D C/C-SiC composites at room temperature

The general properties of the as-processed 2.5D C/C-SiC composites before fatigue test are summarized in Table 2. The 2.5D C/C-SiC composites are dense and show a mean tensile strength of 77.7 MPa after the quasi-static tensile tests.

Fig. 3 presents the diagram of stress versus cycles to failure (S-N curve) for the 2.5D C/C-SiC composites at room temperature. The failure of the cyclic loaded specimens depends on the applied stress level. As shown in Fig. 3, all specimens subjected to the stresses which are higher than 80% of the static tensile strength failed before finishing the test. When the stress level is 80%, only one-third of the tested specimens failed before achieving the desired 10<sup>6</sup> cycles. However, when the stress level decreases to 75%, all of the three specimens could undertake the fatigue loading for 10<sup>6</sup> cycles. Hence, the 2.5D C/C-SiC composites exhibit a high resistance to the fatigue stress and the fatigue limit (10<sup>6</sup> cycles) is probably around 58.2 MPa, corresponding to 75% of the virgin tensile strength.

#### 3.2. Stress-dependence of the residual tensile strength

The dynamic modulus (DM) versus fatigue cycles with the fatigue stress levels of 75% and 90% is shown in Fig. 4(a). During the cyclic loading, an initial decrease of the DM was observed. This decrease can be related to the rapid initiation and the growth of matrix cracks. The effect of matrix cracks deflecting around the fibers should also be taken into account as it leads to relative movements between fiber/matrix. Subsequently, the DM are roughly stabilized, which indicates that almost no new formation of cracks occured (near saturation of cracks). Moreover, it is found that the higher applied fatigue stress leads to more fatigue damages and results in a relatively lower DM. In addition, the DM fluctuates slightly due to the opening-closing effects of cracks under cyclic loading.

Fig. 4(b) shows the relationship of the RTS/elastic modulus versus four different stress levels. For the same reason discussed above, the fatigue loadings lead to reductions for the measured elastic modulus. The similar phenomena were also observed in other CMCs [19,20]. However, no significant difference is observed between different fatigue stress levels. This can be explained by the "self-healing" effect of the composite. During unloading, the formed cracks are partially closed, and the matrix cracks/fibers tend to move to their original places. This can also explain why the DM behaves differently than the resultant E-modulus. In contrast to the decreasing modulus, the samples which were previously loaded with different stresses for 10<sup>5</sup> cycles consistently show an increase of residual tensile strength (RTS). The RTS incline to increase with the higher stress levels. Compared to the virgin samples (about 77.7 MPa), the RTS after the fatigue loadings with the stress levels of 75%, 80%, 85% and 90% for 10<sup>5</sup> cycles increase to  $85.8 \pm 16.3$ ,  $84.3 \pm 17.3$ ,  $86.6 \pm 5.8$  and  $92.5 \pm 8.3$  MPa, respectively. This enhancement of the composite strength can be mainly explained in matters of reduction of internal stresses due to matrix cracking and interfacial degradation, which are also consistent with the other composites as reported in previous studies [21,22].

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