



# Effects of porosity on in-plane and interlaminar shear strengths of two-dimensional carbon fiber reinforced silicon carbide composites

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## ABSTRACT

Porosity effects on shear properties of 2D C/SiC were investigated based on the corresponding shear damage mechanisms. The results show that as the total porosity increases from 12.2% to 26.1%, the interlaminar shear strength decreases from 44.58 MPa to 17.80 MPa according to a power law, while the in-plane shear strength decreases linearly from 143.25 MPa to 74.38 MPa. Under interlaminar shear stress, delamination is resulted from matrix cracking and interface debonding/sliding mechanisms. Effects of porosity on interlaminar shear strength is controlled by the volume fraction of the delaminated matrix. Under in-plane shear stress, echelon matrix shear cracking and large scale fiber bridging mechanisms occur. The decreasing of in-plane shear strength depends on the spacing between the echelon cracks. Since the interface debonding/sliding mechanism occurs under both in-plane and interlaminar shear stresses, a relationship is obtained that the in-plane shear strength equals the sum of the interlaminar shear strength and the fiber bridging term under the minimum total porosity about 30.5%.

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## 1. Introduction

Carbon fiber reinforced silicon carbide composite materials (C/SiCs) are considered as advanced structural materials for hypersonic vehicles due to their high specific mechanical properties [1–3]. Generally, the shear strengths of C/SiCs, in terms of in-plane shear strength (IPSS) and interlaminar shear strength (ILSS) [4–7], are usually much lower than the tensile strength [8–10]. Therefore, shear failure is prone to occur at the stress concentration regions such as holes, notches and attachments, which reduces the structural efficiency in application of C/SiCs. In order to improve the shear strengths, the microstructural features and the shear damage mechanisms have been investigated [4, 11–14]. It was observed that large amount of residual pores are non-uniformly distributed in SiC matrix [15–17]. Around these pores, matrix microcracks initiate during the preparation of C/SiC due to the thermal misfit stress between matrix and fibers [15]. Under shear stress, these microcracks tend to be connected with the periodical matrix shear cracks, which leads to the main shear crack [4, 18, 19]. It indicates that porosity should significantly influence the shear strengths of C/SiCs.

Many researches have concluded that the mechanical properties of C/SiCs could be quantitatively characterized by their constituent properties (matrix, fiber and interface) [4, 11–14, 20–24]. It implies that porosity effects on shear properties may be demonstrated by the constituent properties and the corresponding damage mechanisms, because porosity decreases linearly with the increase of matrix volume fraction. On one hand, high density of matrix microcracks occurs with

high porosity, which always results in inferior mechanical properties of C/SiCs [15, 25, 26]. Hence, a maximum porosity should not be exceeded in order to guarantee effective load transfer between matrix and carbon fibers. On the other hand, interfaces are more prone to debonding/sliding with the existence of pores, which is beneficial for crack deflection and branching [27]. Thus, a minimum porosity should be retained to assure stress relaxation via multiple interface debonding/sliding. In conclusion, the shear strengths of C/SiCs could be improved by porosity tailoring in terms of matrix cracking and interface debonding/sliding. However, such improvements have rarely been discussed based on the corresponding shear damage mechanisms.

In this paper, porosity effects on in-plane and interlaminar shear strengths of 2D C/SiC were comprehensively investigated based on relationships between porosity and the constituent properties. Samples with various porosities have been prepared via chemical vapor infiltration (CVI). In order to accurately characterize the variance of matrix cracking spacing, a uniform pyrolytic carbon (PyC) interface was prepared with the thickness about 200 nm in all samples.

## 2. Experimental procedures

### 2.1. Preparation of samples with different porosity

The 2D C/SiC plates were prepared via chemical vapor infiltration [28]. The preform of the plates was stacked by carbon fiber plain woven cloths (1 K, T-300, Toray). The dimensions of the preform were  $260 \times 150 \times 12 \text{ mm}^3$ . The PyC interphase was firstly deposited on carbon fibers (about 200 nm) using  $\text{C}_3\text{H}_6$  as precursor at  $870^\circ\text{C}$  for 1 h with a pressure of 5 KPa. Secondly, the as-processed preform was

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heat-treated at 1800 °C in vacuum for 1 h. After that, SiC matrix was infiltrated into the preform for 480 h under the temperature of 1000 °C and the pressure of 5 KPa, using methyltrichlorosilane (MTS,  $\text{CH}_3\text{SiCl}_3$ ) as SiC precursor. Argon was employed as the dilution gas to control the reaction rate of infiltration. Finally, preliminarily densified 2D C/SiC plates were prepared. The density and the total porosity of the plates were about 1.6 g/cm<sup>3</sup> and 32%. And their fiber volume fractions were about 40%. These raw plates were used to fabricate samples of the desired dimensions. For IPSS, fifteen samples were cut off according to the dimensions in Fig. 1 (a). For ILSS, thirty samples were cut off according to the dimensions in Fig. 1 (b).

To obtain different porosities, the majority of these samples were further densified by controlling infiltration time of SiC matrix [15,28]. The additional infiltration time was 80, 160, 240, and 320 h, respectively. Note that the maximum infiltration time does not exceed 320 h because the inner pores cannot be filled any further. Finally, samples with gradually decreased porosity have been prepared. The densities of all the samples were between 1.60 and 2.25 g/cm<sup>3</sup>. Their total porosities were between 10% and 32%.

## 2.2. Tests and characterizations

Before tests, bulk density and open porosity of all the samples were measured by Archimedes' method (Mettler Toledo, AG 204, Switzerland). Total porosity and closed porosity were calculated based on the following equations:

$$V_p = 1 - \frac{\rho_{\text{test}}}{\rho_{\text{theory}}} \quad (1)$$

$$V_p = V_{cp} + V_{op} \quad (2)$$

where  $V_p$  is total porosity,  $V_{cp}$  is closed porosity,  $V_{op}$  is open porosity and  $\rho$  is density. Clearly, total porosity decreases linearly with the increase of density.

In-plane shear tests were conducted through Iosipescu method (ASTM: C1292–10). To obtain shear strain, BE120–2HA strain gauge was mounted at the sharp notch [19]. Interlaminar shear tests were performed according to double-notch shear (DNS) method (ASTM: C1292–10). The test configurations are illustrated in Fig. 1. All tests were monotonically performed using a universal load-frame (Instron-5567) at room temperature. The displacement control mode was chosen with a crosshead speed of 0.5 mm/min. The two kinds of shear strengths were measured according to ILSS =  $F/S$  or IPSS =  $F/S$ , where  $S$  is the shear stressed area and is independent of porosity,  $F$  is

the maximum value of the external load. After tests, fractured morphologies of all the samples were observed by scanning electron microscopy (SEM, model HITACH S-4700, Tokyo, Japan).

## 3. Results and discussions

### 3.1. Porosity and density of 2D C/SiC

Fig. 2 shows the measured porosities and densities of 2D C/SiC. As the density increases from 1.60 to 2.25 g/cm<sup>3</sup>, the open porosity decreases linearly from 27.6% to 7.4% while the closed porosity seems to be a constant of 4.3%. It implies that two kinds of pore should match the open porosity and the closed porosity respectively. Morales-Rodríguez et al. [16] characterized the three-dimensional porosity network of 2D SiC/SiC by X-ray tomography. It shows that the open porosity mainly originated from the interlayer pores (Fig. 3). Gélébart et al. [17] presented that the porosity within fiber tows of SiC/SiC was about 7.3%, which was close to the upper limit of the measured closed porosity. In addition, the pores within fiber tows (or intralayer pores) were closed when SiC matrix coating was infiltrated on the surface of fiber tows (Fig. 3) [29]. Consequently, the induced porosity should not be changed. It implies that the closed porosity mainly comes from the intralayer pores.

The decreased open porosity should be attributed to the infiltration of SiC matrix into interlayer pores (Fig. 3). During infiltration, a uniform matrix coating is progressively developed outside of the fiber tows [15, 27]. Meanwhile, the pore size is progressively decreased. It can be inferred that effects of porosity on mechanical properties of 2D C/SiC should be resulted from the interaction between matrix properties and the size of interlayer pores. Generally, mechanical properties can be improved with the increase of matrix volume fraction and the corresponding decreased pore size [15,25,28].

### 3.2. Effect of porosity on ILSS of 2D C/SiC

Fig. 4 (a) shows the typical interlaminar shear stress-displacement curves for 2D C/SiC DNS samples with different total porosities. As the total porosity increases from 12.2% to 26.1%, more flexible shear behavior is observed with lower stiffness and strength. The corresponding ILSS decreases from 44.58 MPa to 17.80 MPa according to a power law (Fig. 4 (b)),

$$\tau_{\text{ILSS}} = a_1 + b_1 (V_p)^{c_1} \quad (3)$$

where  $a_1$ ,  $b_1$  and  $c_1$  are fitting constants for ILSS (Table 1). The decreasing of ILSS is consistent with the decreased bonding area, which is between two neighbor interlayer pores (Fig. 3). Regardless of the

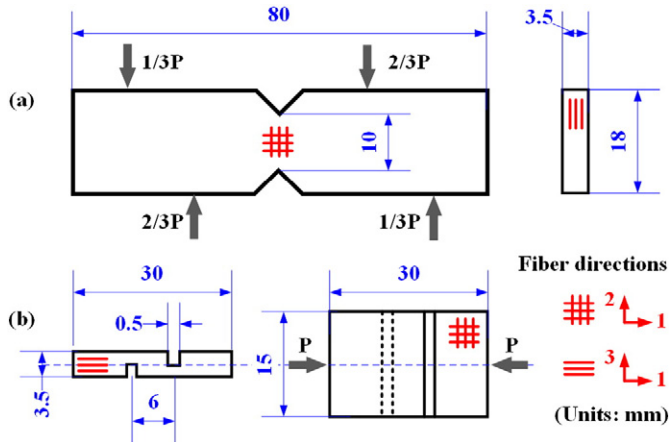


Fig. 1. Geometrical dimensions and test configurations of the two shear samples. (a) Iosipescu sample for in-plane shear test, (b) double-notch shear sample for interlaminar shear test.

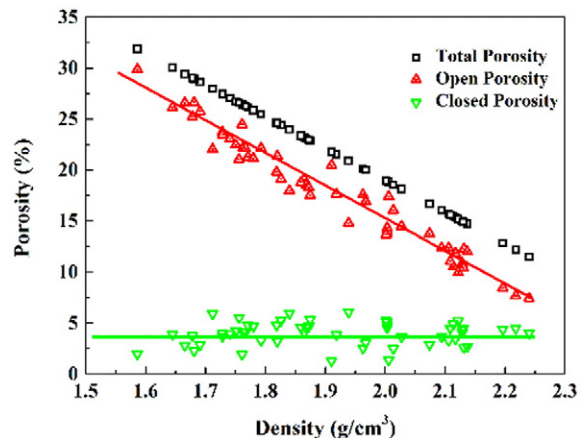


Fig. 2. Relationship between porosity and density for 2D C/SiC.

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