Mechanical properties of SiC<sub>f</sub>/SiC mini-composites reinforcements for SiC<sub>f</sub>/SiC composites

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Abstract. SiC<sub>f</sub>/SiC mini-composites with single-tow microstructures are typically used as reinforcing materials for larger composites and it is essential to obtain accurate mechanical properties for such mini-composites to understand the fundamental mechanical properties of larger composites. In this paper, the mechanical properties of SiC<sub>f</sub>/SiC mini-composites fabricated through a precursor infiltration and pyrolysis process, as well as those of their constituents, are analysed. The Young’s moduli, strengths, and ultimate strains of the SiC fibres are measured through uniaxial tensile tests. The experimental results reveal that the mechanical performance of the SiC fibres is diminished during the high-temperature manufacturing process. The effects of the PyC interface on the mechanical properties of the SiC fibres and SiC<sub>f</sub>/SiC mini-composites are also explored. The results demonstrate that the influence of the PyC interface on the mechanical properties of the SiC fibres is negligible, but significantly increases the strength and failure strain of the SiC<sub>f</sub>/SiC mini-composites. A nanoindentation test is employed to measure the Young’s modulus of the SiC matrix. The relationship between the Young’s moduli of the fibre, matrix, and mini-composite is theoretically characterised and can be reasonably explained by the theory of composite mechanics based on the microstructures of the mini-composites.

1. Introduction
SiC<sub>f</sub>/SiC composites are an important class of materials that are widely used in industry for their excellent combination of mechanical properties, including low density, high toughness, and resistance to heat, oxidation, creep, and fatigue [1, 2]. Therefore, SiC<sub>f</sub>/SiC composites with various microstructures (e.g., 2D plain weaves [3, 4], 2.5D woven structures [5], and 3D braided structures [6, 7]) have received significant attention in recent years based on their potential applications at elevated temperatures. Analysis of SiC<sub>f</sub>/SiC composites can be divided into three general categories according to the characteristic length scale used: (1) microscale, (2) mesoscale, and (3) macroscale [8]. Microscopic models focus on the properties of microstructures (e.g., mechanical properties of the fibres and matrix, interface properties, and crack propagation) and their effects on the mechanical properties of mesoscopic structures. In the mesoscale, SiC yarns are typically modelled as homogeneous orthotropic materials with effective parameters determined through microscopic analysis. The repeating cells of SiC<sub>f</sub>/SiC composites are employed in some mesoscopic models to estimate the effective (macrosopic) mechanical properties of the composites. In macroscopic models, SiC<sub>f</sub>/SiC composites can be...
homogenised as anisotropic materials and their effective mechanical properties are adopted in the analysis of composite structures. Therefore, accurate simulation of composite structure macroscopic responses depends on the proper microscale modelling of mini-composites.

Most studies on SiCf/SiC mini-composites focus on damage and fracture behaviours. Various methods, such as acousto-ultrasonics [9], acoustic emission [2], and X-ray microtomography [10], have been employed by researchers to detect the distributions and propagation of internal cracks in SiCf/SiC mini-composites and to explore the relationship between crack growth and external loading. Morscher and Martinez-Fernandez [11] found that the fracture behaviours of SiCf/SiC mini-composites can be directly correlated to the strengths, moduli, and roughness values of SiC fibres. Probabilistic statistical approaches were employed by Lissart and Lamon [12], and Chateau et al. [13] to theoretically estimate damage progress in SiCf/SiC mini-composites.

The mechanical properties of the interphase of SiCf/SiC mini-composites have also been studied extensively in the literature. It has been reported that the micro-structure [14, 15] and material type [16] of this interphase affect the mechanical behaviours of SiCf/SiC mini-composites. Sauder et al. [17] found that the mechanical properties of the interphase depend on fibre surface roughness. Lissart et al. [12] investigated the relationship between the mechanical behaviours of SiC/PyC/SiC mini-composites and fracture mirrors under loading and unloading processes. Bertrand et al. [14, 18, 19] explored the influence of multi-layer interfaces on the mechanical behaviours of Hi-Nicalon(PyC/SiC)/SiC mini-composites. Single-fibre pull-out experiments have been performed in some studies [18, 19] to measure the strength of the interphase.

The Young’s moduli of SiCf/SiC mini-composites and their constituents are among the most essential material parameters in the multiscale analysis of such composites, but only a few studies have focused on these parameters. Bernard et al. [22] used acoustic microscopy to measure the local Young’s moduli of SiCf/SiC mini-composites fabricated through chemical vapour infiltration (CVI). Nanoindentation testing is a common approach to measure the local Young’s moduli of the fibres and matrices of SiCf/SiC composites [20, 21]. However, to the best of our knowledge, no comprehensive study has been carried out to explore the Young’s moduli of both SiCf/SiC mini-composites and their constituents. This paper presents a comprehensive study of the mechanical properties (including the Young’s moduli, strengths, and failure strains) of SiCf/SiC mini-composites and their constituents based on a series of experiments. To investigate the strengthening effects of SiCf/SiC mini-composites fabricated through precursor infiltration and pyrolysis (PIP), the Young’s moduli of the mini-composites are characterised theoretically based on those of the matrices and fibres in the microstructures of the mini-composites.

Table 1. General properties of SiC fibres manufactured by Boxiang New Material Co. (China).

| SiC fibre | Bulk density [g/cm³] | Diameter [μm] | C/Si atomic ratio | Oxygen Content [wt%] | Tensile Strength [GPa] | Tensile modulus [GPa] |
|-----------|----------------------|---------------|-------------------|----------------------|------------------------|------------------------|
| Boxiang III | 2.5 | 12 | 1.1 | 3.0 | 3.1 | 250 |

2. Materials and experimental procedures

2.1. Materials
SiC fibre bundles (with 500 individual unbraided fibres per bundle), manufactured by Boxiang New Material Co. (China), were used to fabricate the SiCf/SiC mini-composites. The general properties of the fibres are listed in Table 1. For the standard SiCf/SiC mini-composites, the SiC fibres were coated with approximately 300–500 nm of single-layered PyC through chemical vapour deposition (CVD) at 1100 °C (hereafter referred to as SiCf/PyC/SiC mini-composites) before the SiC matrix was fabricated using PIP (i.e., precursor infiltration and multiple pyrolysis at 1200 °C). To study the influence of high temperatures on the mechanical properties of SiC fibres during the coating process and matrix
fabrication, additional fibre bundles without a SiC matrix were processed using the same CVD coating and PIP procedures. Similarly, to explore the effects of PyC coating on the mechanical properties of SiC fibres and SiC/SiC mini-composites, additional fibre bundles and mini-composites were processed using only PIP without the PyC coating process.

Figure 1. The specimens of SiC fibre bundle (a) and SiC/SiC mini-composite (b) for tensile tests.

2.2. Specimen preparation and tensile tests
In this study, fibre bundles, rather than individual fibres, were used to measure the mechanical properties of SiC fibres. Fibre bundle samples and mini-composites are presented in figure 1. According to the Chinese national standard GB/T 34520.4-2017, the length of the specimens was 60 mm and the gauge length was 30 mm. Quasi-static monotonic tensile tests were implemented using an MTS (China) universal testing machine in strain control mode with a displacement rate of 1 mm/min in ambient air at room temperature (figure 2). First, the original (unprocessed) SiC fibre bundles were tested to measure their stiffness, strength, and ultimate strain. Next, the PIP-processed fibre bundles with and without PyC coating were tested to study the effects of the PIP process and PyC coating on the mechanical behaviours of the SiC fibres. Finally, tensile experiments were performed on the SiC/SiC mini-composites with and without PyC coating. Three specimens were used for each measurement of the fibre bundles and mini-composites.

Figure 2. The tensile test implemented by the MTS universal testing machine (a) and the zoomed-in view of the specimen response (b).

2.3. Nanoindentation testing of the matrix phase
Nanoindentation testing was employed to examine the mechanical properties of the SiC matrix phase of the SiC/SiC mini-composites. The cross sections of the SiC/SiC mini-composite samples were finely polished before they were tested by a Berkovich diamond indenter. To minimise the influence of the fibres, the indenter was positioned carefully on the surface of the SiC matrix to ensure that it was far away from the fibres. All nanoindentation tests were performed by using a loading-unloading procedure for load control with a maximum load 500 mN. An unloading curve was used to obtain the elastic modulus of the matrix.
3. Results

3.1. Mechanical properties of SiC fibres

To derive the stress-strain relationships of the SiC fibre bundles, the areas of the cross sections of the fibre bundles had to be determined. For each type of fibre, the diameters of 40 individual fibres were measured using the single-yarn strength tester. For each fibre, because the shapes of the cross sections were nearly circular, the diameters of five cross sections were measured and the average value of the areas of the cross sections (assumed to be circular) was obtained. The areas of the cross sections of the fibre bundles were then calculated as the average area of the cross-sections of the 40 individual fibres times 500, which was the number of fibres in each bundle. The areas of the cross sections of the original fibre bundles, PIP-processed uncoated fibre bundles, and PIP-processed PyC-coated fibre bundles were calculated to be 0.0608 mm², 0.0811 mm², and 0.0871 mm², respectively.

After the areas of the cross sections of each type of bundle were measured, their stress-strain relationships could be computed from the load-displacement results. The corresponding curves are presented in figure 3. One can see that the curves for the three samples of each of the three types of fibre bundles agree well, demonstrating good consistency between samples and the reliability of the experimental procedure. Furthermore, the strengths and ultimate strains (defined as the strains at brittle fracture) also show good consistency for the three types of fibre bundles, with maximum differences of only 9.8% and 9.2%, respectively. The strengths and ultimate strains of the three types of fibres were calculated as the average values of the three corresponding samples. The average strengths of the original fibres and PIP-processed fibres with and without PyC coating were 3072 MPa, 1704 MPa, and 1796 MPa, respectively, while their ultimate strains were 1.65%, 1.18%, and 1.26%, respectively (figure 4). These results suggest that the high-temperature PIP process has a significant influence on the strengths and ultimate strains of SiC fibre. These values diminished by approximately 42% and 24%, respectively, following the high-temperature PIP process. In contrast, the PyC coating only decreased the strengths and ultimate strains of the SiC fibres marginally (approximately 9%), which has a negligible effect on the mechanical behaviour of the SiC fibres.

![Figure 3](image_url)

**Figure 3.** The curves of the stress vs. strain for the specimens of the SiC fibre bundles: (a) original fibre, (b) processed and uncoated fibre, and (c) processed and PyC-coated fibre.
We found that all the stress-strain curves indicated a good linear relationship between strain and stress when the strain was less than approximately 0.5%. An apparent transition point can be observed for every curve at a strain value of approximately 0.5%. After this point, the slopes of the curves tend to be slightly smaller up to the point of brittle fracture. This phenomenon is caused by the progressive fracture of the fibres. Therefore, the areas with good linearity represent bundles suffering from pure elastic deformation with no structural damage. The Young’s moduli of the three types of SiC fibres were determined by fitting the data within the linear regions from the origin to the strain value of 0.5%. The average Young’s moduli are included in figure 4 and their values are 248 GPa, 184 GPa, and 184 GPa for the original fibres and PIP-processed fibres with and without PyC coating, respectively. Again, a clear decrease of approximately 26% in terms of the Young’s moduli can be observed when comparing the results of the PIP-processed and original fibres, which indicates a significant influence of the high-temperature PIP process on the Young’s moduli of the SiC fibres during matrix fabrication. The PyC coating shows a negligible effect on the Young’s moduli of the SiC fibres (the Young’s moduli of the SiC fibres with and without PyC coating are nearly identical). Therefore, damage factors should be included in the multiscale modelling of SiCf/SiC mini-composites to consider the strength, ultimate strain, and Young’s modulus losses of the fibres.

3.2. Mechanical properties of SiCf/SiC mini-composites

To analyse the effects of PyC coating on the mechanical properties of the SiCf/SiC mini-composites, two types of mini-composites with and without the PyC coating were fabricated. The areas of the cross sections of the mini-composite samples were measured using a metallographic microscope. A typical image of a cross section captured by the metallographic microscope is presented in figure 5. The areas of three cross sections of each mini-composite sample were measured and the average values were utilised to compute the stresses in the mini-composites. The stress-strain curves for the two types of mini-composites are plotted in figure 6. Again, good consistency between the stress-strain relationships,
strengths, and ultimate strains can be observed for the three samples of each type of mini-composite. The average ultimate strain for the mini-composites with PyC coating was 1.39% (figure 4), which is slightly higher than that for the PIP-processed SiC fibres with PyC coating. The average strength of the mini-composites with PyC coating was 473 MPa (figure 4), which is significantly less than that of the corresponding fibres. This implies that the strength of the SiC matrices fabricated by PIP is much less than that of the SiC fibres because they occupy 79.9% of the volume of the mini-composites. For the mini-composites without PyC coating, the average strength and failure strain were 214 MPa and 0.3%, respectively (figure 4). These values are only approximately 45% and 22% of the values for the mini-composites with PyC coating, respectively. These results indicate poor fibre-matrix interaction under tensile loading for the mini-composites without PyC coating.

Figure 5. The cross-section of the SiCf/SiC mini-composite imaged and measured by the metallographic microscope.

For the mini-composites with PyC coating, the stress-strain curves are similar to those of the fibres. A nearly linear relationship between the stress and strain can be observed when the strain is less than 0.5%. After the apparent transition point at approximately 0.5%, the slopes of the curves become slightly smaller until the point of brittle fracture. This can be attributed to the progressive fracture of the fibres. Consequently, the data within the range of approximately 0–0.5% strain were utilised to fit the Young’s moduli of the mini-composites with PyC coating. The average value was calculated to be 47 GPa (figure 4). For the mini-composites without PyC coating, good linearity with slight fluctuation was observed in the stress-strain curves up to the brittle fracture strain at approximately 0.3%. Therefore, the data within the range of approximately 0–0.3% strain were utilised to fit the Young’s moduli of the mini-composites without PyC coating. The average value was calculated to be 72 GPa (figure 4). From these results, one can see that the Young’s moduli of the mini-composites without PyC coating are much greater than those of the mini-composites with PyC coating. This can be attributed to the smaller cross-sectional areas of the mini-composites without PyC coating, which led to larger fibre volume fractions. According to the theory of composite mechanics, a larger fibre volume fraction typically results in a larger Young’s modulus because fibres are much stiffer than the matrix.

Figure 6. The curves of the stress vs. strain for the specimens of SiCf/SiC mini-composite with PyC coating (a) and without coating (b).
3.3. Mechanical properties of the SiC matrix

In the nanoindentation tests, indentations at four different positions on the matrix were measured to determine the average Young's modulus of the SiC matrix. A representative load-depth curve for the loading-unloading process of the nanoindentation tests is presented in figure 7. The average Young’s modulus of the SiC matrix was measured to be 56 GPa, which is approximately 70% smaller than that of the PIP-processed fibres (figure 4).

Figure 7. The representative load-depth curve of loading and unloading for the SiC matrix under the nanoindentation with the maximum indenting load of 500 mN.

Figure 8. The representative SEM micrographs of SiC/SiC mini-composites with PyC coating (a) and without coating (b).
4. Discussion

4.1. Young’s moduli of mini-composites

After the Young’s moduli of the SiC fibres and SiC matrix, and the volume fraction of the fibres are determined, the classical rule of mixture in composite mechanics can be used to estimate the Young’s moduli of the SiCf/SiC mini-composites as follows:

\[ E_c = cE_f + (1-c)E_m, \]

where \( E_f \) and \( E_m \) denote the Young’s moduli of the fibres and matrix, respectively, and \( c \) represents the volume fraction of the fibres. Here, the value of \( c \) is calculated as the ratio between the areas of the cross sections of corresponding fibre bundles and mini-composites. For the mini-composites with and without PyC coating, the volume fractions of the fibres \( c \) are 23% and 31%, respectively. According to (1), the Young’s moduli of the mini-composites with and without PyC coating were estimated to be 85 GPa and 96 GPa, respectively. However, the values measured in the experiments were 47 GPa and 72 GPa, which are only approximately 55% and 75% of the predicted values. This discrepancy can be attributed to the fragmented features of the SiC matrices fabricated by PIP. Scanning electron microscopy (SEM) was employed to explore the microstructures of the mini-composites. A representative image is presented in figure 8. One can see the matrix is formed by the aggregation of SiC fragments. The cracks in the matrix tend to grow, even under low stresses, which leads to a decrease in the load carrying capacity of the SiC matrix. It should be noted that the Young’s modulus of the SiC matrix measured in our experiments is much lower than that reported for bulk SiC materials (300–410 GPa) [22]. The main reason for this discrepancy is that the fractures in the SiC matrix significantly reduce the apparent stiffness of the matrix and overall structure.

The contribution to the Young’s moduli from the fibres in the mini-composites is written as \( 
\bar{E}_f = cE_f \). The resulting values are 42 GPa and 57 GPa for the fibres in the mini-composites with and without PyC coating, respectively. These values are only 11% and 21% smaller than the Young’s moduli of the corresponding mini-composites, respectively. This implies that the fibres support most of the load, whereas the main function of the matrix is to constrain the fibres.

4.2. Strength of mini-composites

Analysis of the stiffness of the mini-composites revealed that tensile loads are mainly carried by the fibres. However, the mini-composites without PyC coating failed at a much lower strain level compared to those with PyC coating, whose ultimate strain was slightly larger than that of the corresponding fibres. The SEM micrograph of the mini-composites without PyC coating (figure 8b) shows that the interface between the SiC fibres and SiC matrix is less defined compared to the case of the mini-composites with PyC coating (figure 8a). This suggests that the cracks in the matrix may propagate directly into the fibres under relatively low tensile stress, which contributes to the poor strength of the mini-composites without PyC coating. However, for the mini-composites with PyC coating, the PyC coating can prevent the propagation of cracks into the fibres [23].

4.3. Comparisons to data from the literature

Regarding the mechanical properties (i.e., Young’s modulus, strength, and ultimate strain) of the SiCf/PyC/SiC mini-composites we studied, some experimental results have been reported in the literature [2, 17, 22, 27, 28]. We note that the fabrication method used to create SiC matrices in previous studies, namely CVI, is different from the process we used (PIP). Detailed comparisons between the corresponding mechanical properties in the results from the literature and our results are demonstrated in figure 9. As shown in this figure, the strength of SiCf/PyC/SiC mini-composites in previous studies ranges from 317–1030 MPa based on the types of SiC fibres used. The corresponding value (473 MPa) measured in our study is within this range. However, the Young’s modulus of the matrix (56 GPa) obtained in this study is significantly smaller than previously reported values (approximately 420 GPa) [17, 22, 28]. As discussed in Section 4.2, the SiC matrix fabricated by PIP is amorphous and filled with
concentrated cracks, which significantly diminishes the Young’s modulus of the SiC matrix. In contrast, the microstructure of a SiC matrix fabricated by the CVI process is crystalline in nature. Based on the large volume fraction of the SiC matrix (greater than 70%), the Young’s moduli of the SiCf/SiC mini-composites measured in this paper are significantly smaller than those reported in the literature, even though the Young’s moduli of the SiC fibres are comparable. In terms of ultimate strain, the values measured in this study are larger than those reported in the literature. This implies that the PyC coating performs very well in terms of deflecting crack propagation.

5. Concluding Remarks
In this study, the mechanical properties (i.e., Young’s modulus, strength, and ultimate strain) of SiCf/SiC mini-composites and their constituents were carefully measured. The matrices for the mini-composites were fabricated through PIP. Tensile tests of the SiC fibre bundles were implemented to measure the mechanical properties of the fibres. Specimens of the original fibre bundles and PIP-processed fibre bundles with and without PyC coating were examined to investigate the influence of high-temperature PIP processing (during matrix fabrication) and PyC coating on the mechanical properties of the SiC fibres. The experimental results demonstrate that high-temperature PIP processing diminishes the Young’s modulus, strength, and ultimate strain of the SiC fibres, whereas PyC coating has a negligible effect on the mechanical properties of the SiC fibres.

Two types of mini-composites (with and without PyC coating) were fabricated to explore the effects of PyC coating on the mechanical properties of mini-composites. The stiffness, strength, and ultimate strain of the mini-composites were obtained via tensile tests. Nanoindentation tests were performed to determine the Young’s modulus of the SiC matrices. The classical rule of mixture in composite mechanics was adopted to analyse the relationship between the Young’s moduli of the fibres, matrices, and mini-composite. It was shown that the Young’s moduli of the mini-composites obtained from the experimental tests are smaller than the values predicted by the classical theoretical model based on the Young’s moduli of the fibres and matrices. This can be attributed to the fragmented microstructures of the matrices fabricated by PIP, which suggests that the fibres support most of the load, whereas the main function of the matrices is to constrain the fibres. The mini-composites without PyC coating failed at a much lower tensile strain based on the propagation of cracks (originally in the matrix) through the SiC
fibres. For the mini-composites with PyC coating, this propagation was prevented by the PyC coating and the ultimate strain of the mini-composites was comparable to that of the corresponding fibres.

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