Effects of pyrochemical treatment for producing interfacial carbon layer on tensile properties of polymer-derived-matrix-based SiC/SiC composites

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ABSTRACT
A carbon layer can be produced on the surfaces of the SiC fibers used in SiC-fiber-reinforced SiC matrix (SiC/SiC) composites via high-temperature heat treatment in a reductive atmosphere. This layer acts as an interfacial sliding layer and helps tailor the mechanical characteristics of the composites. The pyrochemical technique is more efficient and cost-effective than the more commonly used chemical vapor infiltration (CVI) for producing the aforementioned carbon layer. In this study, the effects of the pyrochemical technique on the tensile properties of polymer-derived-matrix-based SiC/SiC composites were evaluated and compared with those obtained via CVI. Although the SiC fibers tested were not weakened by the pyrochemical treatment, the composite formed using the pyrochemically treated fibers showed a lower tensile strength than that of the composite with CVI-treated fibers. This strength reduction was probably caused by a decrease in the Young’s modulus of the fibers after the pyrochemical treatment. The matrix in the former case experienced multiple fractures, leading to the decline of its load-transferring capability at lower applied stresses. This deterioration can be redeemed by increasing the fiber volume fraction (Vf), and therefore needs to be considered when designing components for composites.

1. Introduction
SiC-fiber-reinforced SiC matrix (SiC/SiC) composites, which are representative ceramic matrix composites (CMCs), are used commercially in the hot-section components of civil aircraft engines [1–3]. To extend their applicability, their effectiveness needs to be improved, and their production cost must be reduced. The chemical vapor infiltration (CVI) method is widely used to form the fiber/matrix interfacial layer, as well as the matrix in the fabrication of these composites. A highly functional interfacial layer of boron nitride or carbon must be formed on the surfaces of the SiC fibers in order to fabricate these composites [4–9]. While the CVI method allows for significant control over the composite microstructure and results in highly stable composites, it involves long processing times. Further, the costs of installing, operating, and maintaining the equipment required for this process are very high. Therefore, the simplification or absolute elimination of the CVI step of the composite fabrication process would result in significant commercial benefits.

An alternative method for forming a carbon layer on the fiber surfaces was developed by Ube Industries, Ltd. (Japan), and is known as the TM-S6 treatment method [10,11]. It is a simple pyrochemical treatment method wherein the SiC fibers are heat-treated at high temperatures (~1300°C) in a reductive (CO) atmosphere. Owing to its features, this method is essentially inexpensive. During the treatment, the Si-C structure near the fiber surface reacts with the impure O atoms through the following reaction to produce a carbon-rich layer on the fiber surfaces: SiC + O → SiO↑ + C. Thus, in this case, the interfacial layer is formed on the fiber surfaces based on the fibers. Researchers have evaluated several composites based on pyrochemically treated SiC fibers and have reported that they exhibit remarkable mechanical properties and desirable interfacial functionality [12–15]. Basically, carbon interfaces are desirable candidates for use in nuclear power systems [16,17]. Although fibers with carbon interfaces are generally unsuitable for use in aerospace applications owing to their vulnerability toward oxidative loss, very thin films (of the order of several tens of nanometers) could exhibit a certain degree of oxidation resistance. Their low thickness somewhat complicates the penetration of oxygen. Hence, certain uses such as components of a single use spacecraft are applicable in aerospace.

In this study, we examined the aforementioned alternative method for forming the interfacial carbon layer and compared it with CVI in terms of the mechanical properties of the composites produced using these methods. To analyze the basic properties of the fabricated composites, we performed tensile tests on the unidirectional composites fabricated with one fiber
bundle, which is the simplest structure possible. Further, to elucidate the primary factors determining the properties of the composites, we performed separate evaluations of each constituent; tensile tests on the individual fiber filaments and fiber bundles, and interfacial shear debonding tests were performed.

2. Experiments

2.1. Sample fabrication

Samples with a length of 150 mm in the forms of filaments, fiber bundles, and bundle composites were prepared for tensile tests and related evaluations such as microscopic analysis and nanoindentation (Table 1). In this study, for composite reinforcement, we used bundles of Tyranno Fiber® (ZMI grade, Ube Industries, Ltd., Japan). These fiber bundles nominally consisted of 800 filaments with a diameter of 11 μm, as per the manufacturer’s catalog [18]. All the samples were prepared using fiber bundles from the same roll.

The individual filaments were picked from the bundle, fixed on a carbon jig in a straight manner, and then subjected to one of the two interface-forming treatments. The fiber bundle samples were subjected to the same treatment. The bundle composite samples were produced by fixing a bundle on the carbon jig, subjecting them to interface-forming treatments and densifying them with a polymer-derived matrix using the polymer impregnation and pyrolysis (PIP) method. As stated above, an interfacial layer was formed on the fiber surfaces either through the pyrochemical or CVI treatment. The pyrochemical treatment was performed by Ube Industries, Ltd. The fibers were statically heat-treated in a CO atmosphere at a temperature exceeding 1300°C. The CVI treatment was performed by IHI Inspection & Instrumentation Co., Ltd. (Japan). During the treatment, pyrolytic carbon (PyC) was deposited on the fibers at temperatures exceeding 1000°C using CH₄ gas. To compare the properties of the samples treated using the two methods, the CVI method was used to prepare samples with carbon layers of two different nominal thicknesses: 50 nm and 1000 nm.

The PIP treatment was performed in our laboratory. During the treatment, bundles of reinforcing fibers were impregnated with a SiC precursor polymer, allylhydridopolycarboasilane (SMP-10, Starfire® Systems Inc., US) [19], and heated to 800°C in an argon atmosphere. This treatment was repeated eight times. During the first treatment, SiC particles with an average diameter of 0.24 μm (Beta random ultra-fine grade, Ibiden Co., Ltd., Japan) were mixed with the polymer in an inner percentage of 30 wt% [20], and heat treatment was performed under vacuum. Finally, after the final PIP treatment, the samples were heated to 1000°C in an argon atmosphere. Details of the treatment conditions are available elsewhere [21]. Hereafter, composites fabricated with the pyrochemically treated fiber and the CVI-treated fibers of 50 nm and 1000 nm thicknesses are denoted as “TM-S6”, “CVI_50” and “CVI_1000”, respectively.

2.2. Evaluation of samples

All the samples were subjected to tensile testing at approximately 25°C. The samples were also subjected to Auger electron spectroscopy (AES) and Raman spectroscopy. A scanning Auger microprobe (Model 680, ULVAC-PHI, Inc., Japan) was used for AES. The surface of the test sample was sputtered with Ar⁺ ions at energy of 3 kV, and the sputtering depth was estimated based on the correlation data for SiO₂. A triple laser spectrometer (Ramanor T-64,000, HORIBA Jobin Yvon SAS, France) with an Ar laser (514.5 nm) having a beam diameter of 100 μm was used as the light source for Raman spectroscopy. Further, the samples were analyzed using digital microscopy (VHX-200, KEYENCE Corp., Japan), field-emission scanning electron microscopy (FE-SEM, S-4700 Hitachi High Technologies Corp., Japan), and high-resolution transmission electron microscopy (HR-TEM, EM-002B, TOPCON Corp., Japan). The thin-film samples used for the TEM observations were prepared using a focused ion beam system (SMI 2050, SII Nanotechnology Inc., Japan).

Prior to tensile testing, the samples were glued to a 150 mm × 25 mm supporting paper with a 90 mm × 15 mm hole in the middle. The sample was glued to the supporting paper such that a gauge length of 90 mm and tab lengths of 30 mm at both ends were obtained. All the samples were handled carefully to prevent damage, maintain their straightness, and ensure that all the fibers in the bundles were under the same tension. Once the test sample was fixed in the testing machine, the side margins of the paper were cut off using scissors.

Table 1. List of the samples tested.

| Sample type       | Number of fiber* | Fiber/Matrix interface layer† |
|-------------------|------------------|-------------------------------|
| Filament          | 1                | Pyrochemically-formed C-rich layer (1000 nm) |
| Fiber bundle      | 800              | None                          |
|                   | 800              | Pyrochemically-formed C-rich layer (1000 nm) |
|                   | 800              | CVI-produced pyrolytic C layer (50 nm) |
| Bundle composite  | 800              | Pyrochemically-formed C-rich layer (1000 nm) |
|                   | 800              | CVI-produced pyrolytic C layer (50 nm) |
|                   | 800              | CVI-produced pyrolytic C layer (1000 nm) |

* Numbers of fiber of fiber bundle and bundle composite are based on manufacturer’s shipment inspection table.
† Values in brackets are nominal thicknesses of pyrolytic C layer.
The fiber bundle and composite samples were tested using an electromechanical testing machine (Model 5966, Instron Corp., US) fitted with a load cell that had a capacity of 1 kN and a non-contacting video extensometer (AVE 2663–821, Instron). The tests were carried out at a gauge length of 90 mm, tracking-marker interval of approximately 70 mm, and crosshead speed of 0.5 mm/min. In total, five samples were tested; the data for the samples breaking nearly at the end of the gauge section (approximately 3 mm from the end of the gauge section) were discarded to minimize the error. At least three valid data values could be obtained for all types of samples.

The fiber bundles were evaluated based on their stress; the stress was calculated by dividing the applied load by the nominal total area of all the fibers present in a single bundle. The area of the CVI-derived layer was not included in the total area. The stress-to-strain ratio was computed based on the slope for stresses of 200–300 MPa using the least-squares method. In contrast, the bundle composites were evaluated based on the load applied, as the area of the matrix varied according to the sample and test position. The load-to-strain ratio was computed for the bundle composites for loads of 7–15 N, which corresponded to stresses of 100–200 MPa based on the assumption that only the fibers were subjected to the load. The strain at the fracture was considered to be the strain at the maximum load. The proportional limit was determined as 0.005% of the offset yield load.

The tensile tests were performed on the filament samples using an electromechanical testing machine (Model 5542, Instron Corp., US) equipped with a 10 N load cell. The tests were performed at a crosshead speed of 0.1 mm/min. The stress was calculated based on the applied load divided by the cross-sectional area of the fracture fibers that was measured from FE-SEM images. The CVI-derived layer was not considered when determining the area. The strain was calculated by dividing the displacement of the crosshead by the initial gauge length, which was 90 mm. The Young’s modulus was computed for the stress range of 200–300 MPa using the same method as that used for the fiber bundle. More than 30 measurements were performed for each condition.

In addition, nanoindentation tests were performed to evaluate the elastic moduli of the fiber surfaces. A nanoindentation system (ENT-1100a, Elionix Inc., Japan) equipped with a Berkovich indenter was used for the tests, which were performed at room temperature (approximately 26°C) in air. The load was applied at 10 mN/s, held for 1 s, and then removed. The elastic modulus was determined from the load-to-displacement slope corresponding to the maximum load during the unloading stage.

Nanoindentation tests were also performed to evaluate the properties of the fiber/matrix interfaces in the bundle composites. The same nanoindentation system was used for these tests as well but with a trigonal pyramid indenter tip with a tip angle of 60° and a flat bottom measuring approximately 3 µm on each side. The flat bottom of the indenter tip was formed using a focused ion beam system (JEM-9320FIB, JEOL Ltd., Japan). For these tests, thin plate-like samples with a thickness of 100 µm were prepared, and both sides were finely polished such that all the fibers were aligned in the thickness direction. The test sample was loaded at a rate of 20 mN/s. For each test, the load at which the indenter displacement increased rapidly at an almost constant load was identified from the load–displacement curve. The fiber–matrix interface was assumed to have debonded at this point. The area of the debonded interface was determined based on the fiber diameter and sample thickness, both of which were measured using an attached video microscope. The discrete applied load was divided by the area of the debonded interface to determine the interfacial shear debonding stress (unit: MPa). More than 30 valid data points were obtained for all types of samples.

3. Results and discussion

Figure 1 (a) and (b) show scanning electron micrographs of the polished cross-sections of the bundle composites fabricated using the pyrochemically treated and CVI-treated fibers, respectively. A distinct black interfacial layer was observed around the fibers in the CVI sample, and a blackish thin and round outline was observed around the fibers of the pyrochemically treated sample. In addition, both samples exhibited similar fiber distributions and matrix microstructures. The average diameters of the fibers in the pyrochemically treated and CVI-treated samples (more than 30 fibers were considered in each case) were 12.4 µm and 12.2 µm, respectively, indicating that the pyrochemical treatment did not cause a significant change in the fiber diameter.

To allow for a more detailed microscopic characterization, TEM images of the longitudinal cross-sections of the pyrochemically treated and CVI-processed samples were obtained, as shown in Figure 2 (a) and (b), respectively. In micrograph (b), the CVI-produced pyrolytic carbon layer is evident on the fiber surfaces. On the contrary, in micrograph (a), a light-grayish layer, which is probably the pyrochemically produced carbon-rich layer, is observed to spread gradually from the fiber surface to the fiber core. The surface appears bright to a depth of several hundred nanometers, indicating the
spreading of this carbon-rich layer from the surface to the core. In addition, both samples exhibited similar microcracks and particle distributions within their matrices, which changed with the measurement location. In addition, the CVI-produced layer exhibited a certain degree of inhomogeneity.

Figure 3 shows the atomic concentration depth profiles of the surfaces of the untreated and pyrochemically treated fibers, as determined by Auger electron spectroscopy. The contents of elemental Si, C, and O in the untreated fiber indicated the presence of stoichiometric silicon carbide and a small amount of oxygen. Conversely, the pyrochemically treated fiber was covered by a high-purity carbon layer that extended from its surface to a depth of up to approximately 150 nm. Further, the C and Si contents asymptotically approached those corresponding to silicon carbide, with the amount of O being higher than that in the former sample. This change in the elemental contents continued up to a depth of 500–600 nm; however, the contents exhibited slight variations even at a depth of 900 nm.

To compare the crystal integrities of the interfacial carbon layers, Raman spectroscopy was performed on the surfaces of the pyrochemically treated and surface-CVI-treated fibers. Figure 4 shows the obtained results; here, $\Delta \nu_{1580}$ and $I_{1355}/I_{1580}$ are the half width of the peak at 1580 cm$^{-1}$ and the ratio of the intensity of the peak at 1355 cm$^{-1}$ to that of the peak at 1580 cm$^{-1}$, respectively. $\Delta \nu_{1580}$ exhibits an inverse relationship with crystallinity in the case of graphite. Moreover, $I_{1355}/I_{1580}$ is inversely proportional to the crystallite size of graphite. The values of both factors were lower for the pyrochemically produced carbon layer than for the CVI-produced layer, indicating that the pyrochemically produced layer had a higher degree of crystallinity than the CVI-produced one. An interfacial layer of highly crystallized graphite with a well-
developed layered structure exhibiting superior sliding characteristics was possibly formed in the former case.

Figure 5 shows the typical load–strain curves of the bundle composites fabricated using the pyrochemical and CVI treatments. The data for a fiber bundle not subjected to any coating treatment are shown for reference. The two CVI composites exhibited different slopes in the first half of the curve; the slope of the composite with 1000-nm-thick fibers is relatively higher than that of the composite with 50-nm-thick fibers. Subsequently, the slope of the former decreased, with both samples eventually exhibiting similar slopes. Moreover, the slopes in the second half of the curve were similar to that of the untreated fiber bundle. The slopes differ because the first half of the curves is attributed to mechanical contribution of the thick carbon layer; discussion in this regard is beyond the scope of this article. Notably, the pyrochemically treated bundle composite exhibited an almost linear curve throughout, with a slope that was

Figure 2. Transmission electron micrographs of longitudinal cross-sections of composites consisting of (a) pyrochemically treated fibers and (b) CVI-produced-carbon-coated fibers.

Figure 3. Auger-based atomic concentration depth profiles of surfaces of untreated and pyrochemically treated fibers (denoted as “TM-S6”).
relatively smaller than that of the other samples. Finally, all composites fractured immediately after the application of the respective maximum loads. The results of the tensile tests performed on the bundle composites, including the ultimate load, load-to-strain ratio, proportional limit, and strain at fracture

Figure 4. Raman spectroscopy results showing carbon crystallinities of surfaces of pyrochemically treated fibers and CVI-produced -carbon-coated fibers; $\Delta \nu_{1580}$ and $I_{1355}/I_{1580}$ represent half-width of peak at 1580 cm$^{-1}$ and ratio of intensity of peak at 1355 cm$^{-1}$ to that of peak at 1580 cm$^{-1}$, respectively.

Figure 5. Typical load-strain charts of various bundle composites; curve for untreated fiber bundle is shown for reference.

Figure 6. Results of tensile tests of various bundle composites, including ultimate load, strain at fracture, load-to-strain ratio, and proportional limit values.
value, are shown in Figure 6. Of the three composites tested, the pyrochemically treated one exhibited the lowest ultimate load and proportional limit values, with its values being approximately 60% of those of the CVI-treated composites. Further, the former exhibited a lower load-to-strain ratio, although the values of the CVI composites varied, owing to the difference in the thicknesses of the interfacial layers. In contrast, the values of the strain at the fracture of the three composites were similar. That is to say, the pyrochemically treated composite broke at a lower load after elongation to a strain level similar to the CVI composites while

Figure 7. Optical micrographs of post-tensile-test broken tips of composite specimens consisting of (a) pyrochemically treated fibers and CVI-produced-carbon-coated fibers with thicknesses of (b) 50 nm and (c) 1000 nm.
exhibiting a lower load-to-strain ratio. The reason for this result is discussed below.

Figure 7 shows the broken tips of the composites after the tensile tests; these images were obtained using a digital microscope. Micrographs (a), (b), and (c) correspond to the pyrochemically treated fibers and the CVI-layer-coated fibers with thicknesses of 50 nm and 1000 nm, respectively. In all three composites, hundreds of fibers extending to lengths of several hundred micrometers were observed. Further, the length distributions of the fibers in all three samples were similar. Based on the crystallographic data presented in Figure 4, the pyrochemically produced interfacial layer can be assumed to have exhibited more optimized sliding characteristics than those of the CVI-produced pyrocarbon layer. The results of the quantitative tests performed to evaluate the properties of the fiber–matrix interfaces are discussed later.

To further examine the tensile test results of the composites, the properties of the fibers used for fabricating these composites, as filaments and bundles, were relatively evaluated. The results obtained for the fiber bundle samples subjected to the same treatments as the composites are shown in Figure 8.

In terms of strength, the pyrochemically treated fiber bundle was similar to the CVI-treated bundle with 50-nm-thick fibers. On the contrary, the strength of the CVI-treated bundle with 1000-nm-thick fibers was approximately half. This was probably attributable to nonuniform longitudinal stress, as they contacted each other only partially through the CVI-produced pyrocarbon layer. However, this result was specific to the bundle sample in question; and was therefore disregarded when considering the fiber strength. Hence, it can be concluded that the pyrochemically treated fibers did not show a noticeable decrease in strength compared to the CVI-treated ones.

Moreover, both types of treated bundles showed higher strengths than that of an untreated fiber bundle. This can be attributed to flaws present on the fiber surfaces, which can act as initiation sites for fiber breakage. These flaws were probably eliminated by the pyrochemical treatment. Conversely, in the case of CVI treatment, the development of these surface flaws was hampered by the coating layer formed, and the layer improved the fiber strength.

The Young’s modulus of the pyrochemically treated bundle was 25% lower than those of the two CVI-treated bundles. Furthermore, it was approximately 17% lower than that of the untreated one. Therefore, it can be surmised that the pyrochemical treatment decreased the Young’s modulus of the fibers. Hence, owing to its
moderately high strength and low Young’s modulus, the pyrochemically treated bundle exhibited a strain at fracture value that was 25% higher than that of the other samples.

The tensile test results of the filament samples are shown in Figure 9. In all cases, except for the CVI-treated 50-nm-thick fibers, the tensile strength was similar to that of the corresponding bundle sample. The strength of the CVI-treated 50-nm-thick filaments was lower presumably because of the variations in the morphology of the formed coating, as confirmed by microscopic analysis. The surfaces of these filaments were lumper than that of the fiber bundle sample, probably owing to the formation of a large number of island-like structures during the environment-sensitive CVI process. Hence, the results for the fiber bundles and filaments confirmed conclusively that the pyrochemically treated fibers did not exhibit a decrease in tensile strength.

In addition, the pyrochemically treated fiber filaments exhibited a Young’s modulus that was similar to that of the corresponding fiber bundle. In other words, they showed the lowest Young’s modulus and the highest strain at fracture. Therefore, we can conclude that after the pyrochemical treatment, the fibers exhibited a decrease in their Young’s modulus; however, their strength either increased or remained unchanged.

The effects of the pyrochemical treatment on the Young’s modulus of the fibers were also examined through nanoindentation tests performed on the fiber surfaces. The depth-based elastic modulus of a pyrochemically treated fiber and an untreated one are compared in Figure 10. The untreated fiber exhibited a relatively flat profile with a slightly decreasing slope, and its Young’s modulus was approximately 100 GPa. In contrast, the modulus of the pyrochemically treated fiber was only approximately 30 GPa at a depth of approximately 100 nm. Further, it increased gradually to approximately 60 GPa as the depth increased to several hundred nanometers. This profile was consistent with the trend in the atomic concentrations of Si and C shown in Figure 3. Accordingly, it can be assumed that the Young’s modulus of the pyrochemically treated fibers decreased as the amorphous SiC was pyrochemically transformed into crystalline C, beginning at the fiber surface. The Young’s moduli were also consistent with the results of the filament tests, which showed that the Young’s modulus of the pyrochemically treated fibers was 10% lower than that of the untreated fibers when computed roughly by assuming that a decomposition proceeded to a depth of 1500 nm.

In addition, the modulus of the untreated fibers determined by the nanoindentation test was approximately half of that reported by the manufacturer (195 GPa) [18], and decreased with an increase in the indentation depth. This result may be related to the fact that the indentation direction was perpendicular to the fiber axis (transverse). Moreover, the region evaluated was very small (microscale), and the sample was curved and not flat. However, the exact reason should be investigated.

The properties of the fiber–matrix interface, which determine the mechanical properties of the composite, were evaluated via the push-out test. The individual shear debonding stresses (\(\gamma\)) of the various samples and the averaged values (\(\text{avg}\)) are shown in Figure 11. All the results were verified based on the load–displacement curves and by examining the surfaces of the

![Figure 10. Depth profiles of elastic moduli of untreated and pyrochemically treated fibers as determined by nanoindentation tests.](image-url)
fibers after testing from both sides. Despite that, the measured stresses were significantly higher compared to those reported previously for composite materials in which the matrix was formed by CVI [22]. This is conceivably attributed to the occurrence of pyrolytic shrinkage of the polymer precursor during composite fabrication, or the deformation of the thin plate-like sample during the test due to the vulnerability of the fiber-surrounding polymer-derived matrix, but it remains unknown. However, the relative relationship between the interface shear strengths of the composites is evident in Figure 11.

The CVI composite consisting of 1000-nm-thick fibers exhibited a significantly higher stress than that of the other two samples, which showed similar values (the value of the CVI composite with 50-nm-thick fibers was only slightly higher than that of the pyrochemically treated ones). This can be ascribed to the crystal integrity of the interfacial carbon, as shown in Figure 4, which confirms that the pyrochemically produced carbon layer resulted in lower shear strength than the CVI-produced pyrocarbon. The relative relationships between the interfacial stresses were consistent with the Young’s moduli of the corresponding composite samples, but not with their tensile strengths. Therefore, in the case of the investigated composites, the interfacial shear strength was not a significant factor influencing the tensile strength.

Figure 11. Interfacial shear debonding stresses between fiber and matrix for composites consisting of pyrochemically treated fibers and CVI-produced-carbon-coated fibers.

Figure 12. Schematics of fracture mechanisms of composites consisting of pyrochemically treated fibers and CVI-produced-carbon-coated fibers. Differences in mechanisms account for differences in tensile properties.
Based on the obtained experimental results, we propose the following fracture mechanism for the composites with different tensile properties (see schematics in Figure 12). When subjected to stress, the composites with pyrochemically treated fibers exhibit a higher strain than that of the CVI-pyrocarbon-coated-fiber-reinforced composite, as the Young’s modulus of the fibers of the former sample is lower. As the strain increases, a greater number of cracks are initiated in the matrix. This lowers the matrix-to-fiber load-transferring ability, causing the stress redistribution mechanism in the fibers to deteriorate according to the shear-lag theory [23]. Hence, the fiber breakage frequency increases. This mechanism would be in effect at lower stresses in composites consisting of pyrochemically treated fibers. Therefore, the composites with pyrochemically treated fibers showed lower tensile strength than that of the CVI-coated-fiber-reinforced composites, even though all composites contained fibers with similar strengths. According to the basic mechanics of composite materials, the strain of a composite can be suppressed (i.e. the Young’s modulus of a composite can be increased) by raising \( V_f \). Therefore, cost-effective pyrochemically treated fibers can be effectively used to fabricate various composite components by considering this deteriorative influence.

The strength of a ceramic composite is largely determined by the events that occur in the later (high stress) phase of the fracture process. During this phase, the two CVI-coated-fiber-reinforced composites exhibited slopes similar to that of the untreated fiber bundle, which was higher than that of the pyrochemically treated composite, as seen in Figure 5.

On the contrary, the Young’s moduli of the composites shown in Figure 6 were determined during the early stage of the fracture process. These relationships of the results of the Young’s modulus showed relatively good correspondence to that of the interfacial shear debonding stress shown in Figure 11. Hence, the strain behavior during the early loading stage would be affected by the interfacial shear debonding stress.

### 4. Conclusions

The effects of a carbon-layer-producing pyrochemical treatment method on the tensile properties of polymer-derived SiC/SiC composites were evaluated and compared to those of CVI treatment. The following conclusions were drawn from the results:

1. The pyrochemically treated fibers did not exhibit a decrease in tensile strength. However, their Young’s modulus was slightly lower than that of the CVI-treated samples.

2. The composite reinforced with the pyrochemically treated fibers showed lower strength and Young’s modulus than those of the composites reinforced with the CVI-coated fibers.

3. The deterioration of the strength was primarily attributed to the decrease in the Young’s modulus of the constituent fibers, which was caused by the conversion of the fiber surface layer of SiC into C of low elastic modulus.

4. The Young’s moduli of the composites were affected by the interfacial shear debonding stress.

5. The deteriorations of strength and Young’s modulus can be redeemed by increasing fiber volume fraction (\( V_f \)). Therefore, when fabricating a composite component, fiber architecture needs to be designed to have sufficient \( V_f \) in consideration of this effect.

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### Disclosure statement

No potential conflict of interest was reported by the authors.

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