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Effect of fiber coating on interfacial shear strength of SiC/SiC by nano-indentation technique

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Abstract

In order to quantitatively evaluate mechanical properties of fibers, matrices and their interfaces in fiber reinforced SiC/SiC composites, fiber push-out tests have been carried out. From the indentation load vs. displacement relations, the fiber push-out process has been discussed in comparison with the C/C composites and the loads for fiber push-in and those for fiber push-out were estimated. The trends of load–displacement curve of fiber push-out process depended on specimen thickness. The curve in the case of thick specimen had a micro step indicating fiber push-in and a larger step corresponding to fiber push-out. However just a larger step indicating fiber push-out was seen without fiber push-in process in the case of thin specimen. Interfacial shear stress was discussed and defined in both cases. The effects of fiber coatings on interfacial shear stress obtained from thin specimens were analyzed. The relationship between bending stress and interfacial shear stress of $SiC_{(pcs)}/SiC_{(CVI)}$ is preliminarily postulated together with microstructural characteristics of the composites.

1. Introduction

Ceramic matrix composites are considered to be promising and potential materials durable under severe environments such as applications for super-sonic transport (SST), hyper-sonic transport (HST), space planes and fusion reactors. In addition to the many thermomechanical advantages of C/C and SiC/SiC composites, their low induced activation characteristics under neutron and high energy particle irradiation make them very attractive as fusion and fission reactor materials [1].

The importance of interfacial shear strength between reinforcement fibers and matrix on mechanical properties of CMCs has been always emphasized [2], but the clear understanding about this relation has not been established nor even provided yet. In order to quantitatively evaluate interfacial mechanical properties, pull-out [3], push-out [4,5], protrusion [6], and multiple fracture tests [7] had been applied for CMC and many accomplishments had been published. One of the important accomplishments seen in these years is the work done by Oak Ridge National Laboratory group [5]. Still, they have not been quite successful to quantitatively clarify the role of interfacial shear strength on mechanical properties of composites. The authors' group has been concentrating its efforts in utilizing the nano-indentation technique on UD and 2D fiber reinforced composites, such as C/C [8–10] and SiC/SiC [11]. One of the recent achievements is the design and installation of the new facility; named as SEMITEC (scanning electron microscope with in situ micro-indentation test capability).

The objective of this work is to establish the basis to evaluate interfacial shear strength in SiC/SiC composites and to correlate the property with other mechanical properties of the composites. Indentation load vs. indenter displacement relation obtained from the fiber push-out tests was investigated and deformation and fracture behavior of CMC was analyzed to estimate the interfacial shear strength in CMC. In order to understand roles of fiber coating on mechanical properties,

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Table 1
Properties of SiC fibers used [12]

Properties	C/Si atomic ratio	Oxygen content (wt%)	Density (g/cm^3)	Tensile strength (Gpa)	Tensile modulus (Gpa)	Max temp. for appl (K)
Nicalon	1.31	11.7	2.55	3.0	220	1473
Hi–Nicalon	1.39	0.5	2.74	2.8	270	1673
Hi–Nicalon type S	1.05	0.2	3.10	2.6	420	1873

specimens with various carbon coating thickness on fibers were used for push-out tests and bending tests. The relationship between bending strength and interfacial shear strength of $SiC_{(pcs)}/SiC_{(CVI)}$ is preliminary postulated together with microstructural characteristics of the composites.

2. Experimental procedure

2.1. Materials used and specimens

Materials used in this study were SiC fibers (Nicalon, Hi-Nicalon and Hi-Nicalon type S) reinforced SiC composites. Typical properties of polycarbosilane-derived SiC fibers are shown in Table 1 [12]. SiC/SiC materials were fabricated by chemical vapor infiltration (CVI) method, followed to various thickness of carbon coating on the fibers. Fiber coating of carbon was applied by the CVI method with the variations of coating thickness from 0.05 to 3.5 μm, prior to the CVI processing to infiltrate SiC as the matrix. These composites were used to study the effect of carbon coating on interfacial shear strength and three-point bending strength.

Schematic flow of the specimen preparation procedure for the single-fiber push-out experiment and the shape of the nano-indenter for indentation tests are shown in Fig. 1. The specimen, with a dimension of $25^l \times 4^w \times 2^t$ mm, was fixed on a graphite plate by hot wax method and was sliced about 650 μm in thickness by a low-speed diamond saw. The final size was selected to be $4^l \times 2^w$ mm with the variation of thickness from 40 to 130 μm by mechanical polishing. A focused ion beam (FIB) device was used to prepare thin foil for TEM observation for selected specimens.

2.2. Fiber push-out test

Fiber push-out tests were carried out with a constant displacement rate by the SEMITEC and an ultra-micro indentation test machine. The specimen was placed on a specimen holder made of tungsten carbide with a groove of 25 μm in width, as shown in Fig. 1. Fibers in SiC/SiC composites above the groove were loaded by triangular diamond pyramidal indenter, where the maximum load was 1 N. The inclination of each side of the indenter to the indentation axis was 68°. The surfaces of the specimens were examined by SEM and surface profilometry before and after indentation tests.

2.3. Three-point bending test

Three-point bending tests were carried out at room temperature under the following condition.

Specimen size was $25^l \times 4^w \times 2^t$ mm, test Span was 18 mm and cross-head speed was 0.3 mm/s.

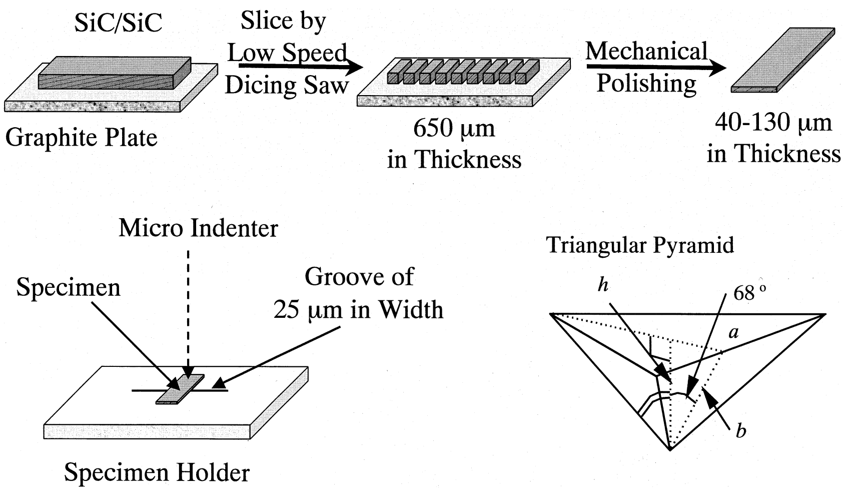

Fig. 1. Schematic flow of specimen preparation for single fiber push-out test and shape of indenter tip.

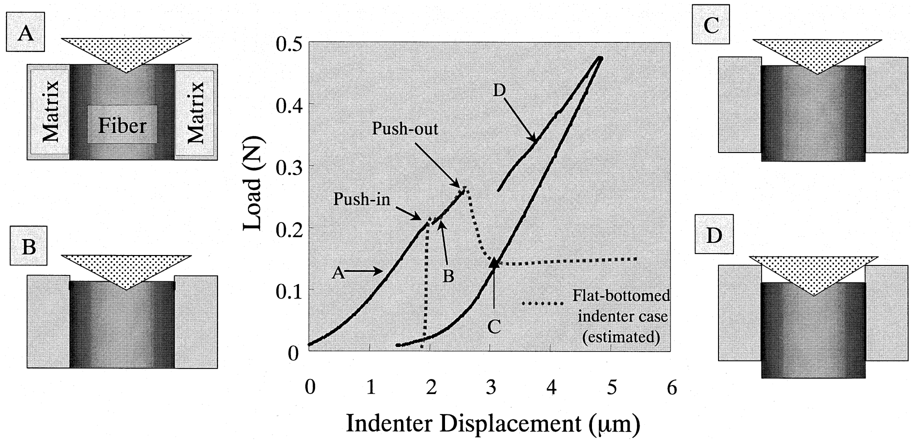

Fig. 2. Indentation curve and schematic diagram under fiber push-out test in SiC/SiC composites.

A video-microscope was used to record the deformation and fracture behavior during the three-point bend test by a side surface observation. Three-point bending stress was estimated from the maximum load.

3. Results and discussions

3.1. Evaluation of interfacial shear strength

Typical examples of indentation curve for fiber push-out tests are shown in Fig. 2. At first indenter made inroads into fiber during process A. The gradient change labeled ‘push-in’ in Fig. 2 is considered to indicate the initiation of interfacial debonding. These should be basically the same results with those in Ref. [5], although the shape of indenter was not flat-bottom but triangular pyramid. Crack between fiber and matrix propagated and fiber was deformed elastically and pushed in during process B. A larger step, following process B, appeared to correspond to the fiber push-out, because fiber push-out was observed whenever this behavior was detected. Indenter touched matrix and load increased again during process D.

Fig. 3 was indentation curve in the case of relatively thin specimen. Comparing with Fig. 2, the gradient change of push-in and process B in Fig. 2 was not seen in Fig. 3. The reason of the difference is specimens used in this experiment were so thin that push-out load was smaller than push-in load in the case of thick specimen as shown in Fig. 3. This trend is quite similar with the case of C/C composites [10]. As a preliminary interpretation, it is considered that push-out in the case of thin specimen occurs when pushing stress overcomes interfacial shear stress. The meaning of load at push-out in the case of thin specimen is different from one in the case of thick specimen. Push-out load in Fig. 2 is used for elastic deformation of fiber and friction by clamping stress and Poisson expansion. From this, the load at push-out in the case of thin specimen was used to determine interfacial shear stress. Interfacial shear stress was defined as

$$\tau_{is} = P/\pi D t, \quad (1)$$

where τ_{is}; interfacial shear stress, P; load at push-out, D; fiber diameter, t; specimen thickness.

Interfacial shear stress has already defined in previous papers such as Ref. [5]. However those definitions were not shear stress but frictional shearing stress.

3.2. Effect of carbon coating on interfacial shear strength

In order to study the effect of fiber coating on interfacial shear stress, fiber push-out tests of SiC/SiC composites with varied carbon coating thickness were carried out. Each carbon coating thickness in the

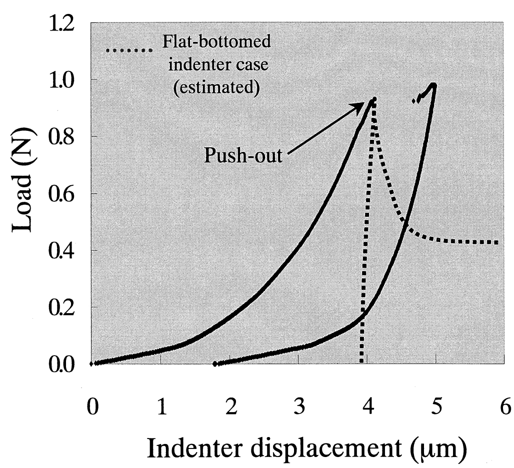

Fig. 3. Indentation curve under fiber push-out test in enough thin SiC/SiC composites.

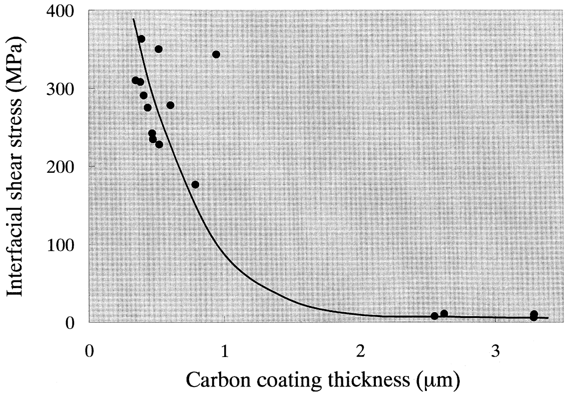

Fig. 4. Relationship between carbon coating thickness and interfacial shear stress.

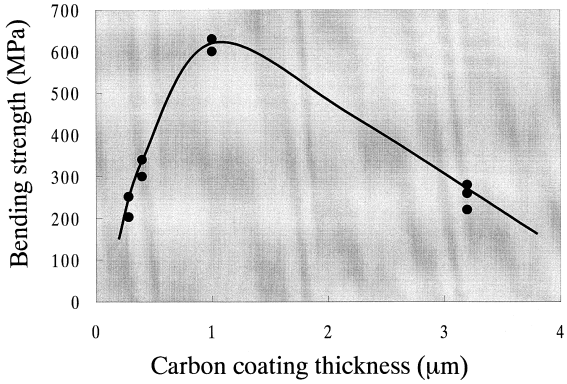

Fig. 5. Effects of carbon coating thickness on bending stress of SiC/SiC composites.

specimens was measured from SEM images. The resultant effect of carbon coating thickness on interfacial shear stress is shown in Fig. 4. Interfacial shear stress drastically decreased with increasing of carbon coating thickness. The relationship between interfacial shear stress and bending strength was discussed in Section 3.3

3.3. *Effect of fiber coating on interfacial shear strength and bending strength*

In order to see the relationship between the interfacial coating thickness of carbon and bending stress, three-point bending tests were carried out with SiC/SiC composites of varied carbon coating thickness. Fig. 5 presents data obtained by the bending tests. Carbon coating thickness in Fig. 5 was average thickness in the specimen as they were non-uniform. Using data like that in Fig. 5, composites with optimum fracture toughness may be engineered. In this result, SiC/SiC composites with around 1 μm carbon coating thickness had a peak in bending stress. Comparing with the results of previous section, appropriate decrease of interfacial shear stress improved bending stress. These results suggest that it is possible to control mechanical property by controlling interfacial shear strength. This result is consistent with the result by Snead et al. [1], that adequate carbon coating improves mechanical performance of SiC/SiC composites. Further work on improved materials will bring us a clearer insight about effects of interface on performance of SiC/SiC composites.

3.4. *Microstructure of interfaces between fibers and matrix*

In order to understand the reason for the difference of interfacial shear strength, microstructure of interfaces between fibers and matrix was observed by TEM. Fig. 6 shows the microstructures of the fiber–matrix interfaces of the specimen with 50 nm-thick fiber coating prior to and after a push-out test. Site at which the debonding occurred during the push-out test was identified as the carbon coating adjacent to the fiber.

4. Summary

In order to quantitatively evaluate mechanical properties of fibers, matrices and their interfaces in fiber reinforced SiC/SiC composites, a nano-indentation technique has been developed and applied. The following results were obtained.

1. From the indentation load–displacement curves, fiber push-out process was discussed and loads at push-in and push-out would be estimated. A definition of interfacial shear strength as obtained from the push-out test of thin specimen is proposed.
2. Interfacial shear stress drastically decreased with increasing of carbon coating thickness. And appropriate decrease of interfacial shear stress improved bending stress. These results mean that carbon coating thickness on fiber is one of the important factors to control mechanical property of SiC/SiC composites.
3. TEM examination provided useful information on microstructure across the fiber–matrix interface. Site of debonding during push-out tests was identified as the carbon coating adjacent to fibers.

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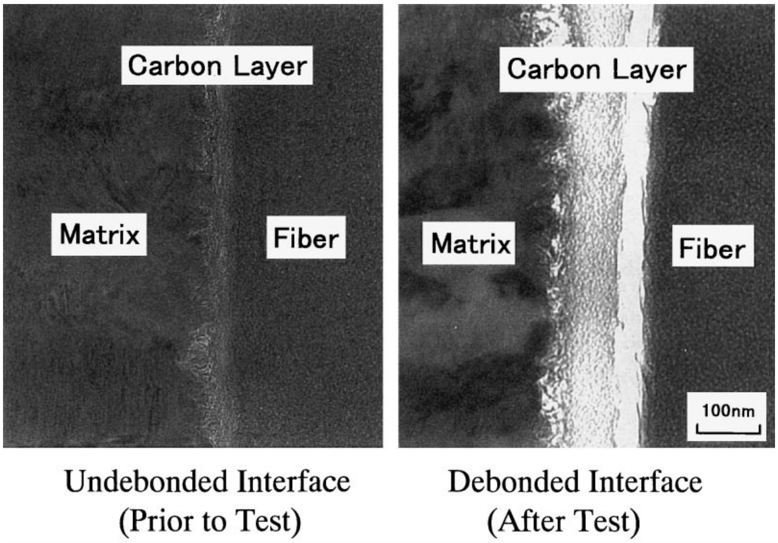

Fig. 6. Interfacial microstructure of SiC/SiC after push-out test.

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