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Crack initiation and growth characteristics in SiC/SiC under indentation test

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Abstract

The mechanical behavior of ceramic matrix composites (CMC) is known to be strongly influenced by fiber-matrix interfacial properties and there have been many efforts to clarify the interfacial characteristics. To understand the fracture mechanism of the materials it is necessary to clarify how the cracks initiate and propagate among fibers, interphase (coating) and matrix. The objective of this study is to investigate crack initiation and growth characteristics in SiC/SiC composites with variations in coating thickness and coating methods by means of micro-indentation technique. Micro-indentation tests and hardness tests were carried out on SiC/SiC composites produced by the chemical vapour infiltration (CVI) process. The intrinsic catastrophic mode of failure of the brittle composite was prevented by application of single carbon and multiple coatings on fibers. Thinner coatings are sensitive to make fibers debonded and may improve the toughness of the composites.

1. Introduction

SiC/SiC composite materials are potential structural materials for fusion reactor because of their high temperature strength, low induced radioactivity, non-catastrophic failure mode, high plant heat efficiency and good irradiation resistance [1]. Nevertheless, composites brittleness is still a problem. For improving the toughness of the materials, a key element is the fiber/matrix interfacial layer which is deposited on the fibers prior to chemical vapor infiltration (CVI) processing, which was reported in Ref. [2]. In that work, carbon layer was introduced to be interphase and the relation between flexural strength of SiC/SiC composites and carbon layers thickness was clarified. As the interphase thickness increased, a reduction in the ultimate bend strength occurs accompanied by a more gradual load drop-off as load is no longer effectively transferred from matrix to fibers. Crack behaviors among fibers, interphase and matrix influence the fracture performance of SiC/SiC composites significantly [3–6]. However, up to now, there are almost no reports about crack behavior under indentation test. In this work, push-in test and microhardness test were carried out on specimens with various thickness carbon layer and multiple layers to investigate the effect of carbon layer and multiple layers on crack initiation and propagation.

2. Experimental procedure

2.1. Material fabrication

For investigating the effects of fiber coatings on crack behavior of SiC/SiC composites, different thickness carbon coating and multiple coatings were employed prior to SiC CVI processing. The thickness of coating ranged from 50 to 5500 nm. Multiple coatings refers to consecutive depositions of C, then SiC, then C.

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Seven layers 2D woven fabrics of SiC (Hi-Nicalon) were stacked one layer by one layer and then pressed to the thickness of 1.8 mm. After 1 h heat treatment at 1000°C in vacuum, carbon coating or multiple coatings were deposited on fibers (CVI). Following the process, 3 h SiC CVI was utilized to preform of specimens. In the end, 22 h SiC CVI was applied for each side of a specimen respectively. The seven types of SiC/SiC composites were produced in NRIM. With the exception of specimen CR01 which was produced by Ube Industries Corporation. The specimens types are listed in Table 1.

2.2. Specimens preparing

Rectangular bars were cut from the composite in size of 4 (l) × 2 (w) × 0.043–0.6 (t) mm using low speed diamond saw. The surface of specimens were mechanically polished prior to micro-indentation. Mechanical grinding was employed for polishing with 3 μm diamond slurry at first and then polishing with 1 μm diamond slurry.

2.3. Testing

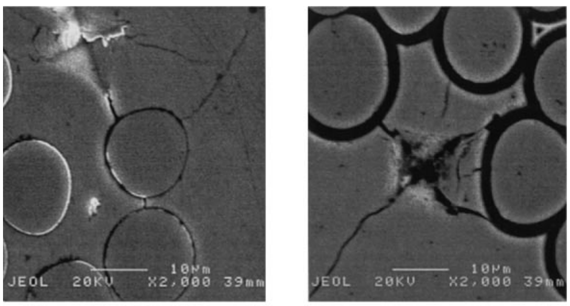
A scanning electron microscope with in situ micro-indentation test capability (SEMITEC) and a micro-Vickers hardness testing machine were utilized to introduce cracks in the composites. The newly developed ultra micro-indentation test machine locates the indentor with in an accuracy of 0.5 μm. This accuracy makes the apparatus possible to indent at the center of the fibers with an average diameter of about 14 μm. The indentor tip is the Berkovich pyramid with 68°. Push-in test was done at 90 g-f for observing fibers debonding behavior. The micro-Vickers hardness testing machine with the maximum load of 1000 g-f and a quadrangle indentor tip was applied to introduce cracks in matrix around fibers. An SEM was used to observe the morphology of specimens surface after the push-in test and the micro-hardness test. Transmission electron microscope (TEM) examination was also carried out to study the microstructure after thin-film specimen preparation with the focused ion beam processing device (FIB). The place where fibers debonded in push-in test was milled to about 60 nm by the FIB system for observation using TEM.

3. Results and discussion

(1) *The push-in test and crack behaviors of thin specimens under push-in test.* From the push-in test of thin specimens with thickness below 100 μm and with maximum load of 100 g-f, it was seen that the number of debonding fibers increased with the increment of the load. A bundle of fibers debonded together. In addition, indenting site is also an important factor to decide the number of debonding fibers. The phenomena mentioned above occurred at the place where the fibers distributed densely and touched each other. At the place where fibers distributed separately usually only one fiber debonded when the fiber was pushed.

Fibers debonding was very effective to reduce crack initiation. While the fiber was difficult to debond, crack would be introduced on the fiber. Cracks were observed at 50 g-f was applied to the matrix. Cracks usually initiated at the edges of the indentor and propagated along the edges direction in matrix (which were similar with the thick specimens under hardness test as shown in Fig. 1 in following section). The crack usually deflected at the interface between fiber and coating. Such crack deflection behavior is generally thought to be the reason for SiC composites toughness.

(2) *The crack initiation and propagation during hardness test.* Crack initiation and propagation were observed during hardness test on matrix. Typically cracks were introduced by 200 g-f. When hardness tests were carried out in the matrix beside fibers, cracks initiated in matrix. If cracks propagated through coating layer, fibers tended to debond. Therefore, for thin coating specimens, fibers were easy to debond. Different crack behaviors of three kinds of composites with single C-layer with thickness of 290, 1900 nm and multilayer with thickness of 3400 nm were observed. The SEM images of the first two materials after hardness test are shown in



A: c-layer 290 nm B: C-layer 1900 nm

Fig. 1. Crack initiated and propagated between matrix and fibers.

Table 1
List of the specimens used

Sample ID	CK01	CK02	CK03	CK05	CK07	CK09	CKM01	CR01
Coating thickness (nm)	890	2100	660	2900	5500	290	1650	50

Fig. 1. For composites with the thinnest carbon coating, the cracks were initiated by the indentation partially, debonded multiple fibers until the crack was arrested. In the case of fiber with C-layer thickness of 1900 nm, cracks propagated to interface and stopped there without fiber debonding. Similar phenomenon was observed on specimen with multiple layers fibers.

Generally the strength of the matrix is lower than that of the fibers. The primary function of the matrix is to transfer the load to the fibers while the debonding layer prevents catastrophic failure and provides pseudo-plasticity through fibers debonding and pull-out. In the hardness test, thinner coating appeared to produce easy fiber debonding. For improving the toughness of SiC/SiC composite, in the future, composites with single layer whose total thickness is below 1000 nm will be suggested to be produced for finding the optimum of coating thickness.

(3) *The debonding of fibers under push-in test.* For single fiber push-in tests on specimens of $\sim$ 600 μm thickness, various debond behaviors were observed. There are two kinds of fibers with a single C-layer or multiple coatings respectively. The indentor load-displacement curve of the single fiber push-in test showed that every fiber had a unique debonding load and the indentor displacement at the maximum load 90 g-f was likewise unique. Considering the debond load and maximum displacement mentioned the above with the fiber coating thickness together, three figures were drawn out. Fig. 2 shows the relation of C-layer thickness

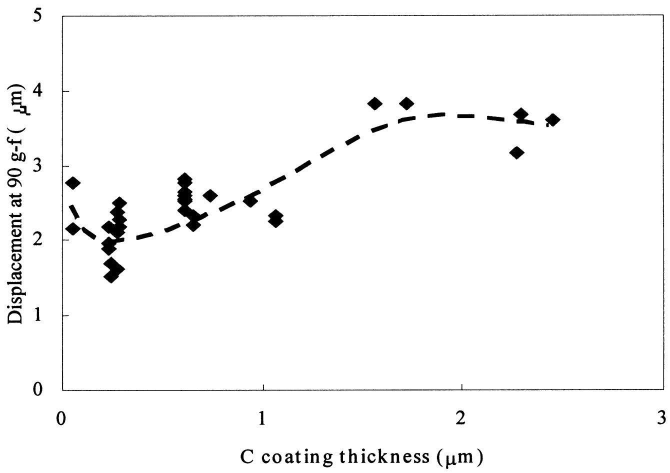


Fig. 2. The dependence of displacement of indentor at 90 g-f on carbon thickness.

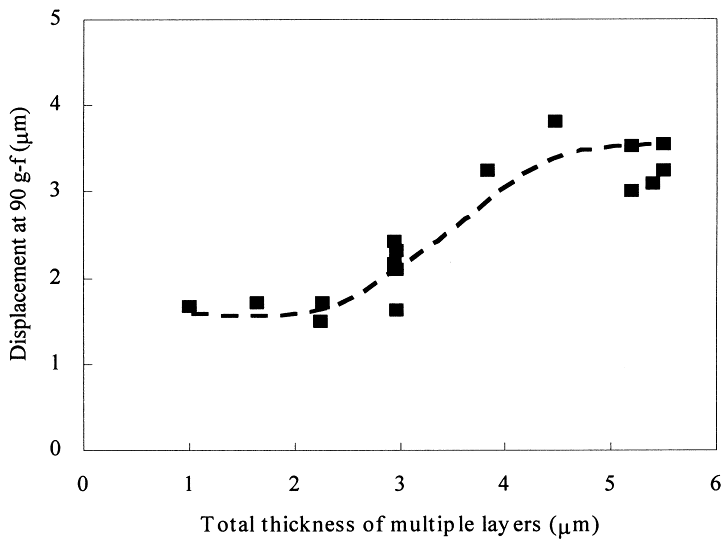


Fig. 3. The dependence of displacement of indentor at 90 g-f on coating thickness.

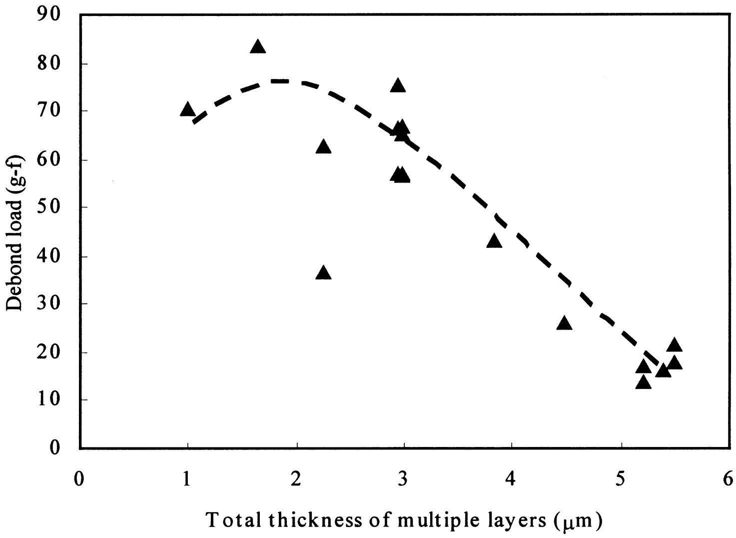


Fig. 4. The dependence of debond load on coating thickness.

with displacement of indentor at 90 g-f under push-in test. With increment of the C-layer thickness the displacement of indentor at 90 g-f increased gradually. From the push-in test on multiple coatings (C, SiC, C) specimens the same tendency was seen (Fig. 3). In addition, the load at which fibers began to debond decreased with the increase in the multiple coatings thickness (Fig. 4).

From Figs. 2–4, it appears that thick coatings allowed easier debonding than thin coatings. But the debond load decreased rapidly with increasing coating thickness. If the debond load is too low, the strength of the composite will decrease. Thinner coating has a higher shear strength (ISS) and frictional sliding strength (IFS). When ISS is high, the matrix can transfer load to the high strength fibers. However, if the ISS is very high, or IFS is very high, the fibers will not debond which causes two things to happen: (1) Cracks are not tie-up at the interface and the composite fails at low load. And (2) unless fiber pull-out and fiber "bridging" occurs the strength at large numbers of fibers are not used. Comparing Fig. 3 with Fig. 4, it is found that around coating thickness of 3 μm the debond load is not so low (~70 g-f) while maximum displacement is not so deep (~2 μm). Therefore, a suitable coating thickness should be found in the future work, which has high ISS and makes fiber debond.

From this work, it is difficult to indicate the differences between single layer and multiple layers. The changing tendencies of debond load look like same. In the future, it is necessary to clarify the different function of the two type coatings by analyzing the crack behavior among the interphase after push-in test under SEM and TEM.

(4) *Observation of crack initiation and propagation under TEM*. Fig. 5 shows an SEM image of a specimen processed by FIB, which was taken by SEM. Push-in test was carried out on the fiber which is at the left side. There was a crack initiated at the interface between the fiber and the first carbon layer. Fig. 6 shows the TEM image of the area which is circled in Fig. 5. The crack propagated along the periphery of the fiber and deflected to coating and continue to propagate along the coating growth direction of synthesis.

The carbon coating and multiple coatings were introduced for transferring the load and weakening the fiber-matrix bonding (Fuse function) [7]. From the results of the push-in test and hardness test the functions mentioned above were seen. By TEM it was proved that

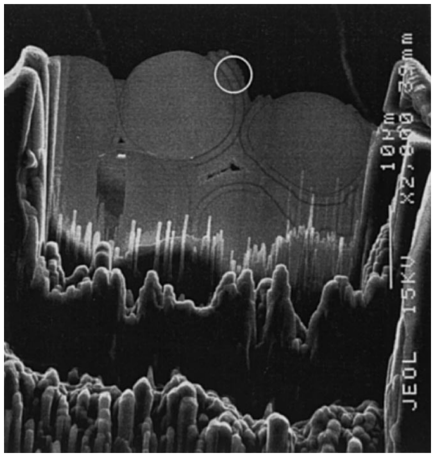


Fig. 5. The SEM image of specimen after FIB.

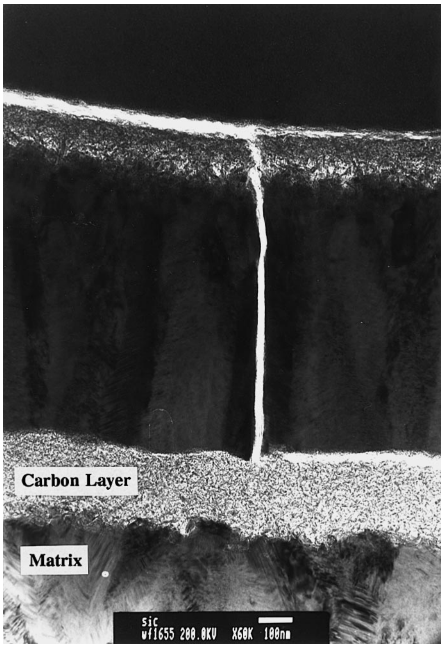


Fig. 6. The TEM image of crack propagation between fiber and coating.

crack initiated in interface and deflected to another coatings, which is considered good for improving the toughness of the composite.

4. Summary

(1) Debonding of fibers is effective to reduce crack initiation in fibers. Indentation site is also an important factor to affect the debonding behavior of fibers. The SEMITEC presented an excellent advantage on push-in test.

(2) A crack propagated from the matrix through coating, led to fiber debonding but did not introduce crack in the fiber. Thus, the catastrophic mode of failure of brittle composite was prevented by the carbon coating or multicoating on fibers.

(3) With the increment of the coating thickness the displacement of indentor at maximum load increased, the debond load of the fibers decreased. It is necessary to find a optimum coating thickness which will lead to fibers debond and also has high interface shear strength.

(4) When fibers were pushed, crack initiated at the interface between fibers and coating and propagated along the fibers periphery, or deflected to coating and propagated along the coating growth direction of synthesis, which was also considered to be good for improving the toughness of the SiC/SiC composites.

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