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Microstructure and Thermal Stability of a Glass-Coated SiC/SiC Composite

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Abstract

Fully glass-coated samples of two-dimensionally layered SiC/SiC composite, which had a thin carbon interfacial layer between the fibre and the matrix, were heat-treated in air for 50 h at temperatures of 1000, 1150, 1300 and 1400°C. The mechanical properties of these heat-treated samples were assessed by means of wedge opening tests, and the microstructural changes due to different heat treatments were subsequently investigated. The glass coating prevented the environmental oxidation of the bulk material whose mechanical properties were retained after heating up to 1200°C. Heating above 1200°C drastically changed the mechanical properties of the composite. This property degradation is attributed to both the structural changes in the fibres and a greatly weakened fibre–matrix interphase. Heating uncoated SiC/SiC specimens at 800°C for 100 h resulted in oxidation of the composite up to a depth of 0·5 mm below the surface. This oxidized layer did not affect the mechanical properties of the bulk material when specimens for wedge opening tests were notched after the heat treatment. However, heat treatments at 860 and 1200°C degraded the properties of the specimens which had been notched before the heat treatment. It is believed that such degradation in mechanical properties arises from the oxidation of the carbon interphase in the unprotected notch tip region.

1 Introduction

Structural ceramic materials have become increasingly important in modern industrial and consumer technology. However, the intrinsic brittleness or low resistance to crack propagation, characteristic of most ceramic materials, severely restricts their use and applications. There is great interest, and much research effort is being invested, in developing methods to improve the fracture toughness of ceramics.[1–7] Amongst these methods the reinforcement of ceramic materials with high strength fibres is believed to offer great potential. The increase in toughness that can be achieved by means of fibre reinforcement arises from both crack deflection at the fibre–matrix interface and from bridging of the crack faces by unbroken fibres. The effectiveness of these processes depends strongly on the strength of the fibre–matrix interface.[8,9] A strong fibre–matrix bonding can produce a large increase in stiffness and strength but it is ineffective for toughness improvement. This is because matrix cracking around the fibres in such a material is followed by failure of the fibres and hence the composite breaks like a monolithic ceramic. A relatively weaker interface, on the other hand, can provide great toughness improvement since failure of the matrix around the fibres is followed by debonding and fibre sliding, or pull-out. This relaxes the stress in the fibres which can remain unbroken, providing effective crack bridging. The extra load taken by the fibres causes reductions in both the crack-tip stresses and the magnitude of the crack extension force.

An efficient way of tailoring the strength of fibre–matrix interfaces in ceramic matrix composites is to form a thin layer of adequate shear strength around the fibres. The principal challenge is to identify a coating material that has the necessary mechanical properties and is also stable in the working environment and at elevated temperatures. The thermal stability of fibre, matrix and interface should therefore be fully investigated before ceramic matrix composites can be widely used at high temperatures. In the present investigation, a study of the effect of heat treatment on the microstructure and mechanical properties has been carried out on a silicon carbide fibre-reinforced silicon carbide matrix composite, which has a thin carbon coating as the interphase.

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2 Experimental Procedures

2.1 Materials

The material investigated was a silicon carbide fibre (Nicalon fibre) reinforced silicon carbide matrix composite. Bundles of Nicalon fibres woven into two-dimensional layers were coated with a thin carbon layer by the decomposition of methane, and then embedded in a chemical vapour infiltrated (CVI) SiC matrix, during which process methyltrichlorosilane gas decomposed at high temperature to yield solid silicon carbide and HCl gas. Infiltration takes place over three stages: after the first stage the mould is removed, while machining is carried out between the subsequent stages of densification. The final material had a density of 2·55 g cm^{-3} with a volume fraction of fibre equal to 38%.

The finished samples were covered with a chemical vapour deposition (CVD) SiC layer and a proprietary glass layer to provide additional protection against oxidation when the material is exposed to high temperature in air. Information about the glass coating was not available from the supplier of the material for industrial protection reasons. However, external seal coatings based mainly on CVD SiC and high temperature glass have already been extensively studied in the case of C/C composites,[10–12] C/SiC composites[13] and SiC/SiC composites.[14,15] Oxidation of the CVD SiC of the surface forms an amorphous SiO_2 layer, which has extremely low oxygen permeability up to 1800°C and provides an effective barrier to the diffusion of the oxygen towards the carbon interphase. Scanning electron microscopy (SEM) examination of the coating in our material reveals that this is a silica-based glass containing alkali oxide. The alkali additives are most likely used to reduce the viscosity of the coating layer, which can flow at relatively low temperature (see Section 3·3) to heal any crack or surface damage which may be introduced accidentally at low temperatures, when the coating is brittle. Flux additives may compromise the apparent advantages of SiO_2 by increasing oxygen permeability.[10]

2.2 Experimental techniques

The SiC/SiC composite material was supplied in the form of square tablets of 10 × 10 × 3 mm^3 in size. Some of these samples were heat-treated at high temperatures in air in order to study the thermal stability of the composite.

Because of the small size of the specimens available, a wedge opening test method had to be developed to assess the mechanical properties of the material both before and after the heat treatment. A small notch, 3 mm deep and 1 mm wide, was cut on each tablet perpendicular to the fibre cloth layers. By forcing a wedge into the notch, a crack was made to propagate across the specimen using the experimental set-up shown in Fig. 1. The loading wedge is made of a hard steel and the wedge angle is 40°. The specimen was supported by a steel roller of 3 mm in diameter. The wedge opening tests were performed under position control mode at a crosshead speed of 0·2 mm min^{-1}. The wedge displacement (vertical displacement) was measured by a capacitance transducer with a sensitivity of 1 mm V^{-1} and the applied load vs. wedge displacement curve was recorded on an X–Y plotter. During the wedge opening test, a travelling microscope was mounted close to the specimen to observe the crack propagation.

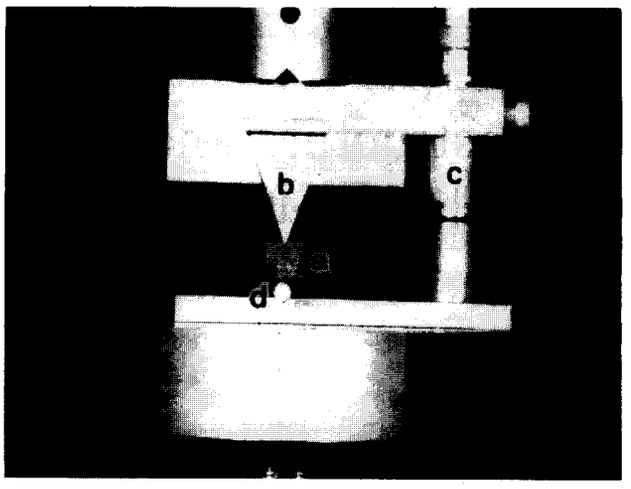

Fig. 1. Set-up for the wedge opening test showing (a) specimen, (b) wedge, (c) displacement transducer and (d) supporting roller.

After the wedge opening tests, the crack paths and fracture surfaces were examined under a scanning electron microscope (Jeol JSM-35 CF). After the specimens were split into two pieces, thin foils were prepared from different positions and orientations as shown in Fig. 2 for structural examination under a transmission electron microscope (TEM) (Jeol 200 CX). The compositions of various areas such as interphase, Nicalon fibres and CVI SiC matrix were analysed using the energy dispersive X-ray spectrometer (EDS) attached to the SEM and TEM.

3 Experimental Results

3.1 As-received material

Four phases could be identified in the as-received composite: the Nicalon fibre, the carbon interphase, the CVI SiC matrix, and pores. The range of fibre diameters was between 10 and 18 μm, while the mean value was approximately 14 μm. The selected area diffraction (SAD) pattern of the Nicalon fibre exhibited three rings, corresponding

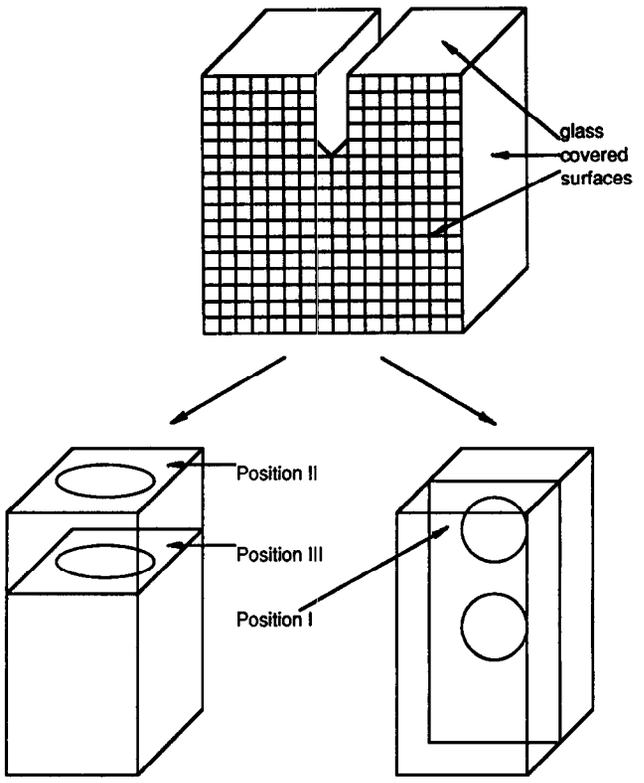

Fig. 2. Schematic diagram showing the sectioning of the SiC/SiC tablet after mechanical test for the preparation of TEM specimens.

to β-SiC_{111}, β-SiC_{220} and β-SiC_{311} scattered beams. The silicon carbide in the fibre was not fully crystalline, therefore the hkl diffraction rings were diffuse and faint. The TEM bright-field image of the composite (Fig. 3) reveals the carbon coating layer, which ranged from 0·1 to 0·2 μm in thickness. SAD patterns from the interphase and the matrix show three primary rings of β-SiC matrix plus two unique additional rings, which are associated with the carbon coating layer [(0 0 2) and (0 1 0) graphite planes]. The chemical vapour infiltrated SiC matrix of the composite had both equiaxed and columnar grains. The initially deposited SiC

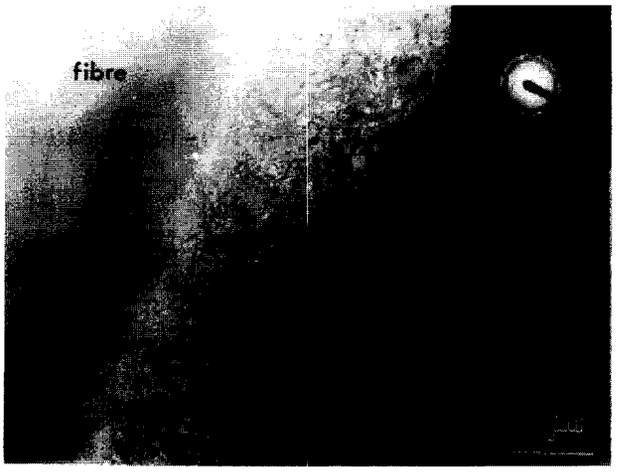

Fig. 3. TEM image of the fibre, the interfacial carbon layer and the matrix. SAD pattern corresponding to the interphase and the matrix.

Fig. 4. TEM micrograph of equiaxed β-SiC grains of the CVI matrix.

crystallites which were adjacent to the fibre coating layer were equiaxed and ranged in size from 20 to 300 nm (Fig. 4). The columnar SiC grains grew radially from the equiaxed SiC zone (Fig. 5). Every columnar grain was up to 0·3 μm wide and several μm long. The transverse cross-section of the fibres and their adjacent matrix showed the fibre–interphase (carbon coating)–equiaxed SiC zone–columnar SiC grains arrangement.

To investigate the effect of heat treatment on the mechanical integrity of the material, three as-received composite tablets were tested by the wedge opening method to obtain reference curves. The results showed good repeatability of the technique. The three specimens gave almost identical load–displacement curves as shown in Fig. 6, with a variation of maximum applied load between 1·75 and 1·88 kN. The composite exhibited an initial linear elastic behaviour followed by a non-linear load–displacement relationship until the maximum load was reached (Fig. 6). The first load drop terminated the linearity and a sound was heard coincidentally with this load drop. No crack was observed on the specimen surface, but sign of

Fig. 5. TEM micrograph of columnar β-SiC grains of the CVI matrix.

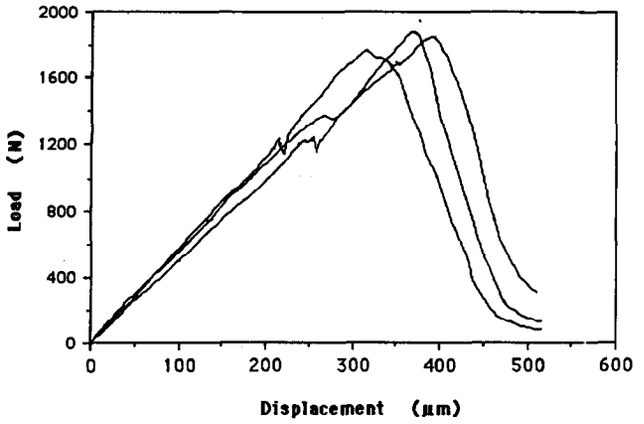

Fig. 6. Applied load vs. wedge displacement curves for as-received SiC/SiC specimens.

debris jumping from the notch tip was noted. From the first load drop to the maximum load, there was no evident main crack on the specimen surface and emission of particles from the notch front continued. The non-linearity towards maximum load is probably due to the progressive cracking of both matrix and fibres which are inside a limited damage zone just at the notch tip. It is possible that the resolution of the observation method used was not sufficient to obtain detailed image of what was happening at the notch tip before the maximum load was reached. After the maximum load, a dominant crack started to be gradually revealed, which propagated from the notch tip through the middle of the specimen to the contacting point of the supporting roller. For the SiC/SiC composite, the fracture by wedge opening test is non-catastrophic, and hence some load-bearing ability is still retained beyond the maximum load because fibres bridge the crack.

The specimen broke by the propagation of a single dominant crack (Fig. 7) and at the end of the test the two halves of the split specimen were still firmly held together by unbroken pulled-out fibres bridging the crack (Fig. 8).

Fig. 7. Straight crack path in the as-received material after the wedge opening test.

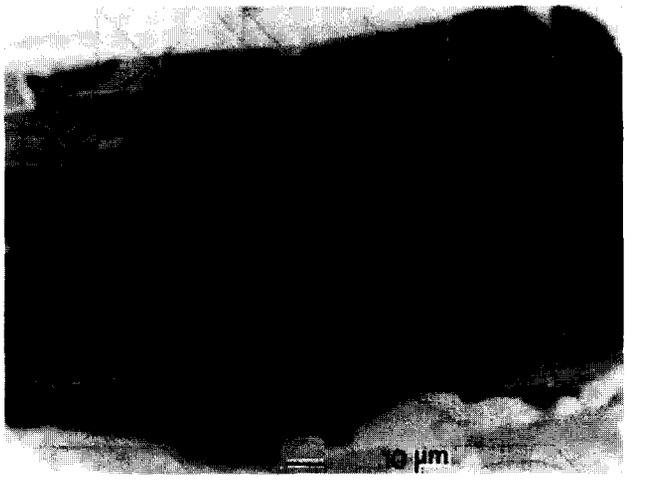

Fig. 8. Fibre bridging in the crack wake in an as-received specimen.

Fig. 9. Fracture surface of the as-received material showing various degree of fibre pull-out.

After crack path examination, the tested specimens were manually pulled apart so that fracture surfaces could be studied under the microscope. Fracture surfaces of as-received specimens were characterized by fibre pull-out (Fig. 9) and debonding between the fibre and the matrix.

Heat treatments and wedge opening tests were carried out on both fully and partially glass-coated SiC/SiC tablets.

3.2 Effect of heat treatment of fully glass-coated specimens

As-received SiC/SiC tablets were heat-treated in air for 50 h at 1000, 1150, 1300 and 1400°C. They were subsequently notched and tested using the wedge opening test method.

It could be seen from the recorded load–displacement curves (Fig. 10) that heat treatment up to 1150°C for 50 h did not significantly change the mechanical properties of the composite. The microstructures observed under SEM and TEM of the heat-treated specimens showed similar features to those of the as-received material.

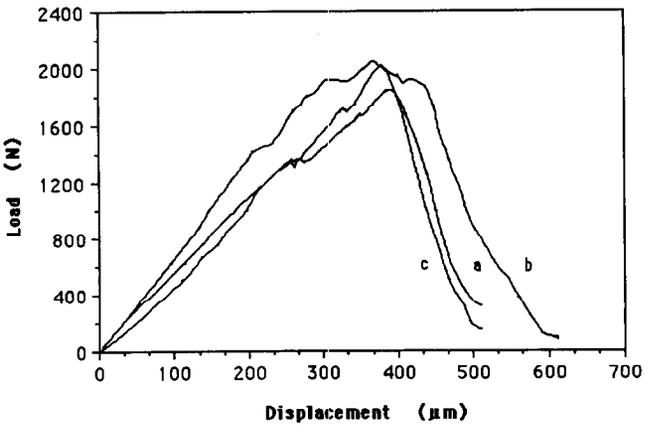

Fig. 10. Applied load vs. wedge displacement curves for SiC/SiC specimens: (a) as-received, (b) heat-treated at 1000°C for 50 h, (c) heat-treated at 1150°C for 50 h.

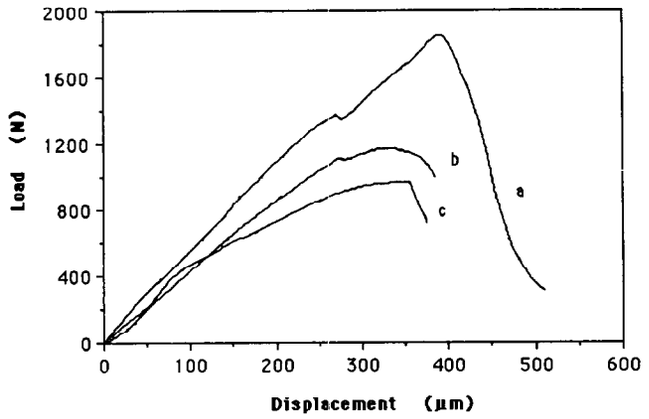

Fig. 11. Applied load vs. wedge displacement curves for SiC/SiC specimens: (a) as-received, (b) heat-treated at 1300°C for 50 h, (c) heat-treated at 1400°C for 50 h.

A heat treatment for 50 h at both 1300 and 1400°C produces a drastic degradation of the composite's mechanical properties (Fig. 11). In the wedge opening tests the specimens did not fail by the propagation of a single main crack, as observed for the material heat-treated below 1150°C, but by massive delamination of the fibre layers (Fig. 12) and severe damage at supporting points. The fracture surfaces of these specimens showed extensive fibre pull-out with segmental patches of interphase adhered to the fibre surface (Fig. 13). All these observations are clear evidence that the strength of the fibre–matrix interface has been severely degraded. It can be seen in Fig. 14 that delamination

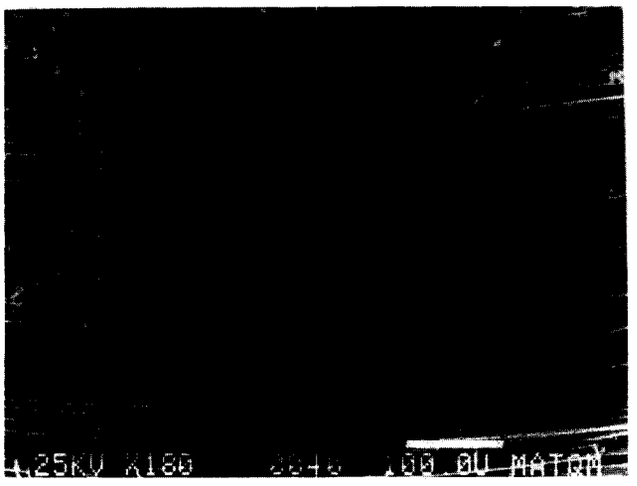

Fig. 12. Delamination surface of the specimen tested after heat treatment at 1400°C for 50 h.

Fig. 13. Extensive fibre pull-out in the SiC/SiC specimen heat-treated at 1400°C for 50 h.

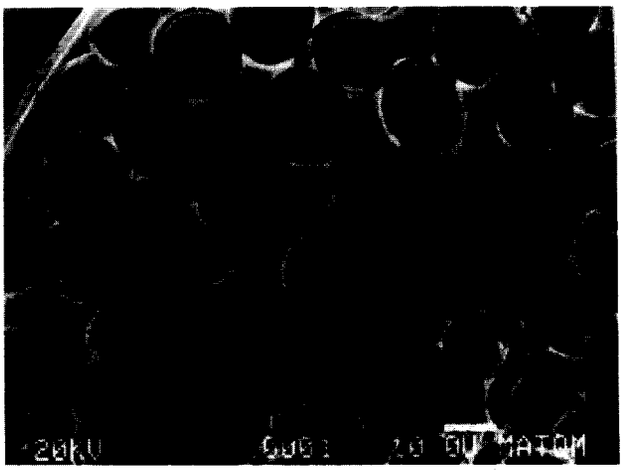

Fig. 14. Extensive debonding between the fibre and the matrix in a specimen heat-treated at 1400°C for 50 h.

Fig. 15. TEM dark-field images of the SiC fibre after crystallization caused by high temperature heat treatment (1400°C for 50 h).

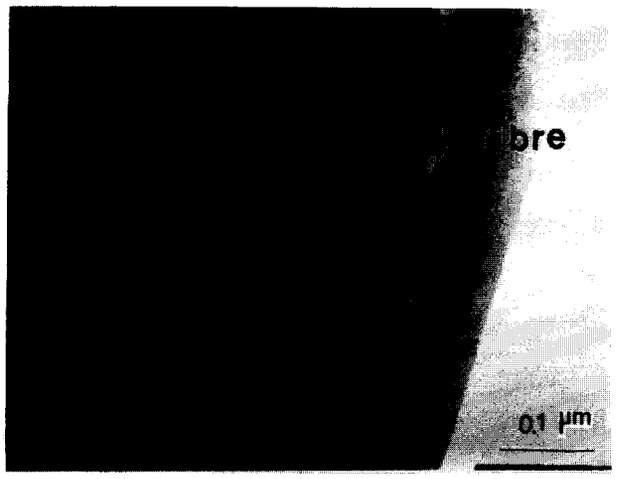

Fig. 16. Microporous carbon interfacial layer between two fibres after high temperature heat treatment.

of the material has taken place not only between the fibre layers, but also within the fibre bundles.

TEM observations made on thin foils prepared from the specimens heat-treated at 1300 and 1400°C showed that the β-SiC in the fibres had changed from an incompletely crystallized to a crystalline structure with an average grain size of 10 nm (Fig. 15). Changes also took place in the interfacial region. The carbon layer existed in the composite, but the carbon in contact with the fibres had become microporous (Fig. 16). Complete debonding of the carbon layer was also observed in some areas.

3.3 Determination of the glass flow temperature

The protection that the covering glass layer offers against oxidation of the bulk material is important for the applications of SiC/SiC composites at moderately high temperatures, and it is therefore of interest to know how efficient this glass layer can be. When local damages in the glass layer occur, the consequent oxidation of bulk SiC/SiC material should not lead to the rapid deterioration of the component.

It was noticed that on the glass layer of the as-received materials there were microcracks and small voids in this layer, and it is possible that when the composite is under load this layer can be damaged. If the glass flows moderately at the service temperature this should have a 'smoothing' or 'healing' effect on the microcracks or small voids. The 'healed' glass layer must then lead to a decreased degree of oxygen infiltration from the environment into the bulk SiC/SiC material. From this point of view it was important to determine the temperature at which the glass coating can flow easily.

Glass flow tests were conducted on as-received SiC/SiC tablets. A scratch approximately 0·3 mm wide was made on the surface of some specimens through the glass layer and slightly into the underlying SiC/SiC material. These specimens were subsequently heat-treated in air for 5 h at 840, 860, 880 and 900°C respectively and were then examined under the SEM.

It could be seen that the glass coating started to flow at between 840 and 860°C and it flowed easily above 880°C. It can be inferred that higher temperatures above 900°C may cause sufficient glass flow to heal a damaged surface layer. A temperature of 800°C was therefore chosen for the heat treatment of partially uncoated specimens.

3.4 Effect of heat treatment of partially glass-coated specimens

Some composite tablets were polished on one surface to remove the glass layer. They were subsequently heat-treated at 800°C for 100 h. TEM specimens were prepared after the heat treatment from different positions of the tablet. Important structural changes were observed to have taken place to a depth of 0·5 mm from the uncovered surface. The original interfacial carbon layer had disappeared, having been replaced by a thin layer of amorphous structure (Fig. 17), as evidenced by a lack of contrast change during specimen tilting in the microscope and by the SAD pattern showing continuous halos. EDS analysis revealed the presence of oxygen and silicon peaks in this layer, which probably consists of silica.

It is thought that this superficial oxidation process, which occurs at the uncovered surface up to a limited depth, will not affect the mechanical properties of the composite in the wedge opening test, which measures resistance to crack growth in the bulk material that is not oxidized, provided that the specimens are notched after the heat treatments. Hence wedge opening tests were not performed on these partially glass-coated samples heat-treated at 800°C.

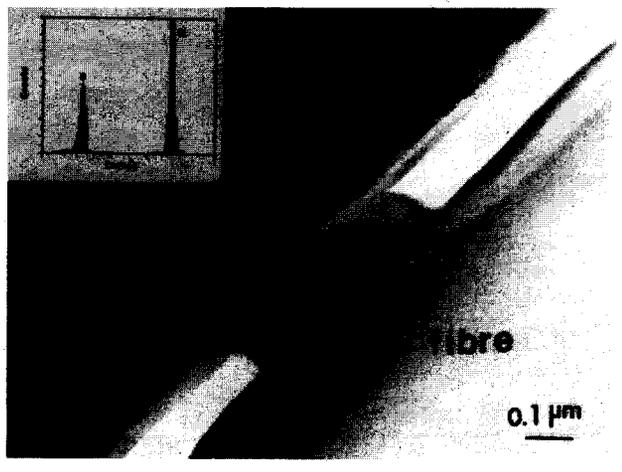

Fig. 17. TEM image and EDS spectrum of the new interphase (silica) in the oxidized specimen heat-treated at 800°C for 100 h.

However, it was considered to be of interest to investigate the effect of heat treatment on specimens which were notched before being heat-treated. During heat treatment, chemical reactions could occur in the notch-tip region and it is possible that the macrocrack initiation and its propagation may be strongly dependent on this region with respect to the first fibre bundle failure.

Notches 3 mm deep were cut into the specimens which were subsequently heat treated at 860 or 1200°C for 50 h. Wedge opening tests were then conducted on these specimens and SEM examinations were performed afterwards.

It can be seen in Fig. 18 that in the notched and heat-treated specimens both the first load drop and the maximum load were considerably lower than for the as-received specimens. However, the composite still exhibited a non-catastrophic failure behaviour as indicated by the curves.

SEM examinations revealed that a single dominant crack had propagated across the specimen for both heat treatment conditions. Fibre pull-out and debonding between the fibres and the matrix were

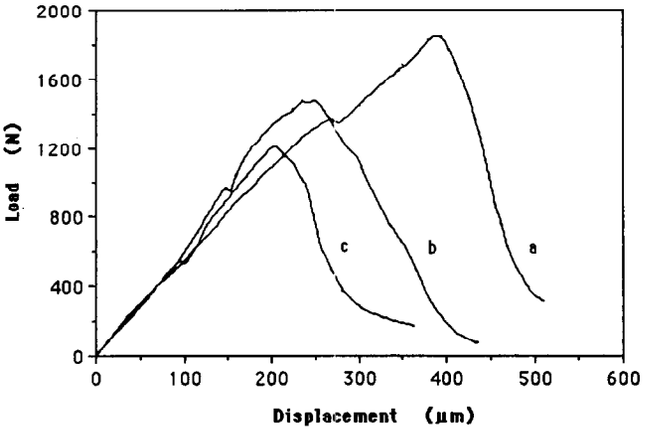

Fig. 18. Applied load vs. wedge displacement curves for SiC/SiC specimens: (a) as-received, (b) notched and heat-treated at 860°C for 50 h, (c) notched and heat-treated at 1200°C for 50 h.

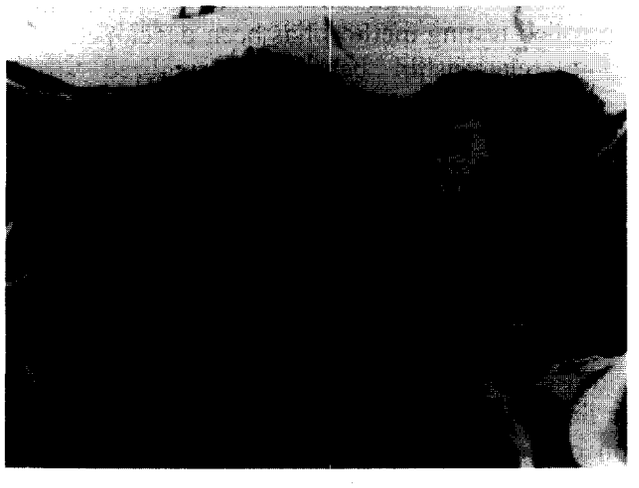

Fig. 19. Oxidized region (flat area) adjacent to the notch tip in SiC/SiC specimen of heat-treated at 1200°C for 50 h, showing strong bonding between the fibre and the matrix.

observed in the bulk fractured area. However, the fracture surface adjacent to the notch tip exhibited a flat region with no fibre pull-out (Fig. 19). The lack of mirror boundary structure in the fractured fibres seems to indicate that the fibre was perfectly bonded to the matrix in this oxidized zone, where the propagating crack may pass directly from the matrix through the fibre with little or no change in direction. The width of the flat region is about 60 μm in the specimen heat-treated at 1200°C, and 40 μm in the specimen heat-treated at 860°C.

4 Discussion

The experimental results have shown that the heat-treated SiC/SiC composite which is fully covered with a glass layer retains its mechanical properties after heat treatment up to 1150°C for 50 h. This glass surface layer drastically limits the oxidation of the bulk material when the specimen is heat-treated in air.

Heating above 1200°C drastically degrades the mechanical properties of the composite. This property degradation can be correlated with both chemical and structural evolutions of the fibre and a greatly weakened fibre–matrix interphase. It has been reported that in addition to silicon carbide, both silica and free carbon are also present in Nicalon fibres,[16,17] and more recent studies have detected a non-stoichiometric silicon oxycarbide (SiO_xC_y) which accounts for most of the oxygen content,[18,19] although the existence of a surface layer of silica is not ruled out.[8] Due to the presence of these compounds, Nicalon fibres have a non-equilibrium composition which would result in its intrinsic degradation at high temperatures.[20,21] It has also been suggested that at the beginning of the heat treatment above 1200°C, in a low partial pressure of carbon monoxide, decomposition of SiO_xC_y produces carbon monoxide,[21] according to the reaction:

$$SiO_xC_y \longrightarrow SiC + C + CO\uparrow \quad (1)$$

The evolution of carbon monoxide from the fibres creates porosity which can reduce the fibre strength.

The current TEM observations also showed that after heat treatment at 1300 and 1400°C, β-SiC in the fibres changed from an incompletely crystallized structure to grains of 10 nm in average size. Therefore, grain growth is another reason for the fibre degradation at 1300 and 1400°C.[20]

If the material is glass-coated, carbon monoxide can accumulate beneath the glass layer building up its partial pressure. Under these conditions SiO_xC_y may decompose as.[21]

$$SiO_xC_y \longrightarrow SiO_2 + C \quad (2)$$

to produce silica. This silica, or that which may be present as a product of the fibre manufacturing process, can react with the interfacial carbon layer under a low partial pressure of oxygen to produce silicon monoxide and carbon monoxide. The evolution of carbon monoxide produces the microporosity observed in the carbon interfacial layer after the heat treatment. Debonding can easily occur between the fibre and the carbon coating, or inside the carbon layer, and this is the reason for the massive delamination experienced in the wedge opening test by the specimens heat-treated above 1200°C. Hence the loss of interlaminar strength in this case is due to the thermal degradation of the fibre strength and the carbon layer, and not to inner surface oxidation.

The microstructural examinations on glass-uncovered specimens showed that the oxidation process at 800°C only occurred to a depth of 0·5 mm (after 100 h). This limited oxidation could be explained by the fact that two chemical reactions, carbon oxidation and silica formation, occur simultaneously at high temperatures.[22] When the uncovered specimens were heat-treated above 800°C, the carbon interphase firstly oxidized starting from the exposed surface, which left interstitial cavities between the fibre and the matrix. Then oxygen easily diffused into these interfacial cavities and reacted with exposed silicon carbide, leading to the formation of silica (Fig. 17). The formation of silica layer, either on the exposed surface or in the interphase, stopped the diffusion of oxygen and hence the oxidation of carbon interphase in the bulk material. Therefore, the oxidation process only occurred over a limited depth in the composite.

It should be pointed out that the mechanical properties of specimens in which the surface had been oxidized up to a limited depth, can be strongly dependent on the testing technique. It is believed that the oxidation process discussed does not affect properties of partially glass-covered samples using wedge opening test method, because for these specimens, the bulk material is not oxidized during the heat treatment and the notch cut after the heat treatment is already inside the bulk material. It has been reported for example that for an uncoated SiC/SiC composite with a thin carbon interphase the strength, measured by three-point bending tests, dropped appreciably after heat treatment at 800°C.[23] It is important to note that the strength measured from a bending test is strongly dependent on the presence of surface flaws, where cracks can easily initiate and propagate. Therefore, even if oxidation is confined to the specimen surface, it can reduce the bending strength of the composite significantly.

The wedge opening tests also showed that heat treatment affected the properties of specimens which were notched before heat treatments at 860 and 1200°C. During the heat treatment in this experimental routine, chemical reaction occurring at the unprotected notch tip may have influenced the initiation of cracks in this region. Absence of fibre pull-out and the lack of mirror boundary structure in the fractured fibres indicated that silica layer had possibly replaced the original carbon interphase in this region, leading to strong bond between the fibre and the matrix. The maximum applied load, at which a single dominant crack started to propagate, consequently dropped. However, because the carbon interphase still remained in the bulk material, the composite retained the mechanical strength and showed non-catastrophic failure.

The toughness of a material is usually characterized on the basis of linear elastic fracture mechanics, which may not be applicable straightforwardly to ceramic matrix composites. Toughness depends on the crack length at which it is measured and materials usually exhibit 'crack growth resistance,' or increase in toughness with the length of the crack. An absolute value of toughness as an intrinsic material property of ceramic matrix composites is difficult to define. However, if toughness is measured in a consistent manner, toughness values can be used for comparison purposes to provide an idea of the resistance to crack growth. The use of the wedge opening test described before to measure fracture toughness presents various problems. The most important is the indentation of the wedge when it is forced into the notch and the uncontrolled friction which results between the wedge and the specimen. This makes it difficult to know the value of the crack opening force. However, excellent repeatability of the results seems to suggest that this testing method can be used for comparative purposes for specimens with small dimensions before a more controlled testing method has been developed.

The relationship between normalized stress intensity factor and crack length for the present wedge opening test has been obtained by finite element calculations[24] as a function of applied load, angle of the wedge and coefficient of friction between wedge and specimen. Using this information, values of fracture toughness have been calculated for the composite in different conditions. They are given in Tables 1 and 2. Two different load values have been used: one corresponds to the first load drop, which may indicate the initiation of a crack at the notch tip; the other is the maximum load. Coefficients of friction of 0·1 and 0·5 have been assumed in the calculation of the fracture toughness. The results of these tables

Table 1. Fracture toughness of the SiC/SiC composite (results based on maximum applied load)

Conditions	*Maximum applied load (KN)*	K_{Ic} *(MPa* $m^{1/2}$*)* $[\mu = 0{\cdot}1]$	K_{Ic} *(MPa* $m^{1/2}$*)* $[\mu = 0{\cdot}5]$
As-received:			
	1·75	26·76	10·27
	1·85	28·29	10·86
	1·88	28·75	11·04
Heat-treated and notched:			
1000°C	2·02	30·89	11·86
1150°C	2·05	31·24	12·03
Notched and heat-treated:			
860°C	1·48	22·63	8·69
1200°C	1·21	18·50	7·10

Table 2. Fracture toughness of the SiC/SiC composite (results based on crack initiation load)

Conditions	*First load drop (KN)*	K_{Ic} *(MPa* $m^{1/2}$*)* $[\mu = 0{\cdot}1]$	K_{Ic} *(MPa* $m^{1/2}$*)* $[\mu = 0{\cdot}5]$
As-received:			
	1·24	18·96	7·28
	1·37	20·95	8·04
	1·23	18·81	7·22
Heat-teated and notched:			
1000°C	1·37	20·95	8·04
1150°C	1·40	21·41	8·22
Notched and heat-treated:			
860°C	0·98	14·98	5·75
1200°C	0·55	8·41	3·23

illustrate both the effectiveness of the glass coating in preventing the degradation of the material heated below 1200°C, and the damaging effect of having notched the specimens before heat treatment. No results could be obtained for the material heated above 1200°C due to its total loss of interlayer strength and massive delamination.

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