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INTERFACE EFFECTS AND FRACTURE IN NICALON/SiC COMPOSITES

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ABSTRACT

Thin coatings deposited on ceramic fibers prior to densification employing chemical vapor infiltration techniques have been used to limit fiber-matrix bonding. This has resulted in improvements in strength and toughness for Nicalon® fiber-reinforced/SiC matrix composites. The properties of the composites are influenced by the thickness of the graphitic carbon interlayer. Matrix cracking, work of fracture, and ultimate strength are controlled by the nature of the interface. Interfacial forces were measured utilizing the indentation method in which a standard microhardness indentor is used to push on fibers embedded in the ceramic matrix. Correlations between interfacial phenomena and observed mechanical behavior have been made.

INTRODUCTION

A process to more efficiently fabricate ceramic matrix composites employing chemical vapor infiltration (CVI) has been developed at Oak Ridge National Laboratory (ORNL).[1-5] The ORNL process permits rapid fabrication of continuous fiber reinforced ceramic composites and simultaneously utilizes thermal and pressure gradients to reduce infiltration time. Densification times for centimeter-thick composites have been reduced from weeks to less than 20 h and thus the process has been termed "forced" chemical vapor infiltration (FCVI). Disk shapes up to 2.5 cm in thickness and one centimeter wall-thickness tubes[6,7] have been routinely infiltrated with a silicon carbide matrix using the FCVI technique.

The baseline reinforcement used in the development of the FCVI process has been the ceramic grade Nicalon fiber, a polymer-derived

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Nicalon®, Nippon Carbon, Tokyo, Japan.

EXPERIMENTAL

Composite Fabrication

Fibrous preforms were fabricated by stacking multiple layers of Nicalon plain-weave fabric rotated in a 0° ± 30° sequence within the cavity of a graphite holder. The layers were hand compressed to produce a preform with a nominal loading of 40 vol % fiber and were held in place by a perforated graphite lid pinned to the holder. The cloth sizing was removed through multiple washings with acetone. The nominal size of the fibrous preforms was 45 mm in diameter and 12.5 mm thick.

Preforms were next precoated with thin layers of carbon. The coatings were deposited from an argon/propylene mixture at 1375 K and 3.3 kPa pressure. The thickness of the carbon layer was varied by changing deposition time and was measured from weight gains and using polarized-light optical microscopy of polished metallographic cross-sections. A control sample of uncoated fibers was prepared for comparison.

The preforms were densified with SiC produced by the decomposition of MTS in hydrogen at a hot-surface temperature of 1473 K and atmospheric pressure using the FCVI process. The densification of porous structures using the FCVI process has been previously described in detail.[1-5]

Mechanical Property Measurements

Bars were cut from the samples parallel to the 0° orientation of the top layer of cloth using a diamond saw, and tensile and compression surfaces were ground parallel to the long axis of the specimen. The average dimensions of the test bars from the composite samples were 2.5 × 3.3 × 40 mm and all specimens were measured and weighed to determine densities.

Bend bars prepared from a composite containing fibers with a 0.52 μm carbon coating were oxidized to remove the interlayer, thus producing a composite with no interfacial bonding or frictional

Si-C-O material.[8,9] The fiber consists primarily of SiC, which is well known for its exceptional high temperature properties and oxidation resistance, making it an excellent candidate as an elevated temperature reinforcement. Silicon carbide was also selected as the matrix to be investigated. The SiC matrix is deposited from the decomposition of methyltrichlorosilane (CH_3SiCl_3 or MTS) in hydrogen, typically at a hot surface temperature of 1473 K and atmospheric pressure. Uniformly infiltrated composites with good strengths and exceptional toughness have been fabricated using the FCVI process.[4,5]

Typically, a thin pyrolytic carbon layer is deposited on the fibrous preforms prior to densification to provide a uniform interface. The carbon is deposited from the decomposition of propylene in argon and deposition conditions were chosen to produce a graphitic coating with the basal planes parallel to the fibers.[10,11] The coatings were found to prevent chemical damage of the fibers during processing, as well as weaken the fiber-matrix interface, enhancing fiber debonding and slip.[12,13] Initially, there was little concern with regard to the thickness of the carbon coating, the strength of the fiber-matrix bond, or about excess slip. It was assumed that the fibers were long and entanglements in the fiber bundles would allow the tows to act like ropes thus providing sufficient reinforcement to carry the load at the onset of matrix failure.[14,15] Further examination of the interlayer revealed significant differences in mechanical properties that were strongly correlated with the thickness of the fiber coating.[12,13]

This report describes a study of the carbon coating used to alter the fiber-matrix bond and change the mechanical behavior of a ceramic fiber-reinforced ceramic matrix composite. The effects of the thickness of the carbon interlayer on the properties of the fiber-matrix interface and the resulting mechanical properties of the composites have been examined. The influence of interlayer thickness on interfacial forces, matrix cracking, work of fracture, and ultimate strength have been investigated. The strength of the fiber-matrix bond was determined using established indentation methods.[16] Room temperature flexure strengths were used to compare the effects of the various fiber pretreatments on the fracture behavior of the composites.

stresses. Specimens were placed in a furnace and heated treated at 873 K in flowing oxygen for 50 h. The low temperature minimized damage to the fibers and matrix but allowed oxidation of the carbon interlayer. Weight losses after heat treatment were measured to ensure complete burn-out of the carbon layer.

Room temperature flexural strengths were measured in four-point bending using a support span of 25.4 mm, a loading span of 6.4 mm, and a crosshead speed of 0.508 mm/min. All specimens were loaded perpendicular to the layers of cloth. Load-displacement curves were recorded to examine the fracture process and were used to determine the loads for matrix fracture and ultimate strength. In general, a single matrix crack was observed in the tests and was noted as the sudden drop in the load-displacement curve and/or deviation from linearity.

Work of fracture was measured for un-notched bend specimens that failed in a controlled manner and was determined from the area under the load-displacement curves and the area of the fracture surface approximated as twice the cross-sectional area of the bend specimen.[17,18] To obtain controlled failure of most materials, and thus measure the work of fracture, it is usually necessary to notch the bend specimens. As the fracture energy of a material increases, such as for fiber-reinforced composites, it becomes unnecessary to notch the specimens.[17,18]

The fracture surfaces of the specimens were examined using a Hitachi S-800 scanning electron microscope (SEM). Specimens that did not completely part during flexure testing were broken by hand so that the fracture surfaces could be examined.

Fiber-Matrix Bond Measurements

Several methods have been developed to quantify the strength of interfacial bonding in fiber-reinforced composites.[12,16,19] Such tests permit a semiquantitative determination of interfacial stresses derived from relatively simple load and displacement relationships. A common technique is the indentation method, which has been thoroughly examined.[16] This technique involves using a microhardness indentor to

apply a force to the end of a fiber embedded in a matrix. Interfacial shear stresses can be evaluated from the applied load and the displacement of the fiber.

A 6.0-mm-thick cross-sectional specimen was cut from each completed composite sample to be used for indentation testing. The specimens were cut along the 0° orientation of the top layer of cloth to ensure that a portion of the exposed fibers would be oriented perpendicular to the cut surface. This alignment is essential for proper implementation of the indentation mechanics. The specimens were mounted and polished using standard metallographic techniques. Loads were applied to fiber ends using a Vickers diamond indentor and a Shimadzu Type M instrument. Loading to the fiber ends was progressively increased until debonding was observed and continued until contact of the indentor with the edge of the fiber cavity was evident. Loads of up to 3.0 N were required to displace the fibers. Indents were also placed in longitudinally polished fibers to determine fiber hardness values. The dimensions of the fibers and indent impressions were measured using the ocular scale of the indentor.

RESULTS

Composite Fabrication

The fabric preforms contained a nominal 41.7 ± 0.9 vol % fiber. Carbon-layer deposition times were varied to produce coatings that ranged in thickness from 0.10 to ≈ 1.0 μm. The preforms were readily infiltrated with SiC to a maximum of 85 to 90% of theoretical density. The theoretical density is defined as the sum of the product of volume fraction and reported density of each component of the composite (fibers, interlayer, and infiltrated SiC). Infiltration of these preforms required 16 to 24 h. Individual composite specifications are summarized in Table 1.

Table 1. FCVI Nicalon/SiC composite specifications

Fiber Content (vol %)	Carbon Deposition Time (h)	Interlayer Thickness (μm)	Density (g/cm^3)	Average Porosity (%)
41.4	0.0	--	2.58 ± 0.08	11.6
41.8	0.3	0.10	2.48 ± 0.12	14.5
40.7	0.6	0.17	2.56 ± 0.11	12.3
41.3	1.0	0.26	2.61 ± 0.15	10.0
41.8	2.0	0.52	2.57 ± 0.17	10.0
40.9	3.3	0.99	2.47 ± 0.10	11.5

Fracture Behavior

Typical load-displacement curves for the Nicalon/SiC composites are shown in Figure 1. The composite prepared from uncoated fibers exhibited low flexure strength and displayed brittle failure, with no signs of toughening. The fracture surfaces were smooth and flat with no evidence of fiber debonding or pull-out. The oxidized specimens also had low strengths, but in contrast to the composites fabricated with uncoated fibers, the composites exhibited high strain to failure. A high degree of fiber pull-out at the fracture surfaces was observed.

The application of the carbon interlayer significantly altered the flexure behavior of the composites. In general, failure was gradual and the fracture surfaces of all composites fabricated with carbon-coated fiber displayed fiber pull-out. An increase in fiber pull-out length was observed with increasing interlayer thickness. The influence of the thickness of the carbon coating on the fracture behavior of the Nicalon/SiC composites is also displayed in the representative load-displacement curves in Figure 1.

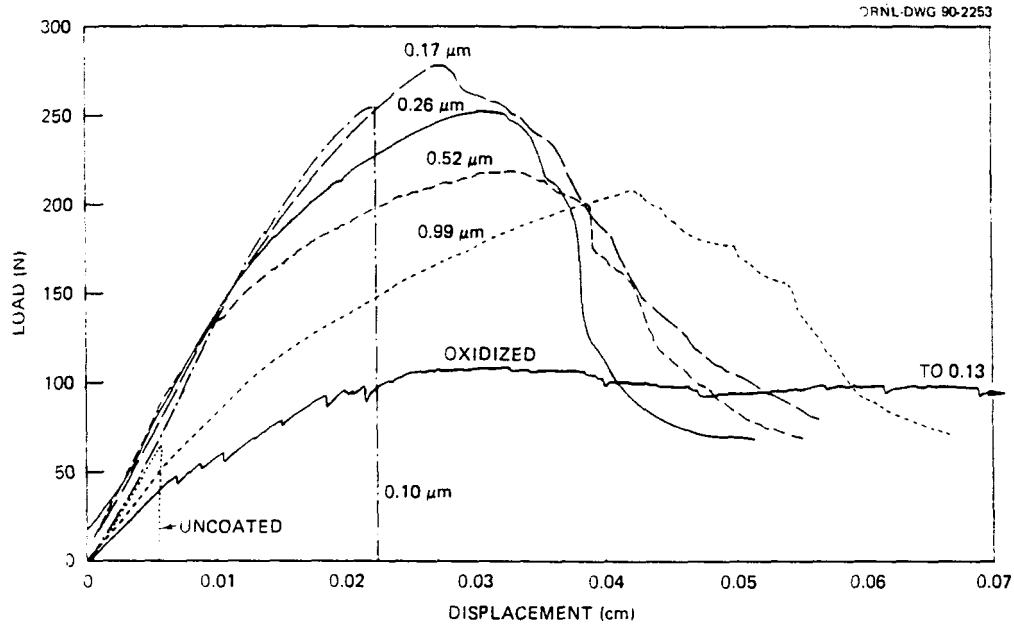

Figure 1. Representative flexure load-displacement curves for Nicalon/SiC composites with varying carbon interlayer thickness, thicknesses are shown on the curves in micrometers.

Composite Mechanical Properties

Mechanical property and interfacial shear stress measurements are summarized in Table 2. Matrix cracking stresses, work of fracture, and ultimate strength for the composites with varying interfacial pretreatments are listed. All mechanical properties were determined from the four-point flexure load-displacement curves. Shear stress was calculated from indented fibers using fiber radius and indent impressions measurements, a measured fiber hardness of 19.7 ± 0.9 GPa, and a fiber modulus of 110 GPa, as determined from previous tests and literature values.[8,9]

Interfacial Shear Stress

Composites with a broad range of interfacial shear strengths were fabricated. High interfacial shear stress was expected for the composite fabricated from uncoated fibers. A thin silica film forms at the surface of the fibers during exposure to the elevated temperatures of processing.[12,13] This layer bonds the fibers and matrix strongly and

Table 2. The influence of the fiber-matrix interface on the mechanical properties of Nicalon/SiC composites

Carbon Interlayer Thickness (μm)	Interfacial Shear Stress (MPa)	Matrix Cracking Stress (MPa)	Ultimate Flexure Strength (MPa)	Work of Fracture (J/m^2)
uncoated	762 ± 163	83 ± 10	83 ± 10	98 ± 24
0.10	385 ± 103	257 ± 29	343 ± 15	2590 ± 300
0.17	217 ± 48	238 ± 17	383 ± 35	4110 ± 880
0.26	127 ± 33	184 ± 22	379 ± 19	4990 ± 530
0.52	75 ± 26	122 ± 39	321 ± 25	4780 ± 300
0.99	39 ± 21	103 ± 6	293 ± 42	4530 ± 810
oxidized	0	40 ± 3	103 ± 39	4660 ± 630

does not allow for debonding and crack deflection at the interface, producing brittle fracture in the composites.

It was demonstrated that interfacial shear stresses were controllable through varying the thickness of a carbon interlayer. The relationship between interfacial shear stress and the thickness of the carbon interlayer is shown in Figure 2. The carbon interlayer reduced interfacial stresses and friction, thus required lower loads to displace the fibers within the matrix. The oxidation of the carbon layer produced a gap between the fibers and matrix resulting in no bonding or friction at the interface.

Matrix Cracking and Ultimate Strength

Provided the ratio of span to thickness ratio is high enough to minimize shear forces during testing, the determination of matrix fracture stress employing a flexure test is valid, for matrix cracking is the first damage to occur in the composite.[20] The oxidized specimens exhibited the lowest matrix fracture strengths. At this lower limit of interfacial bonding and friction, τ_i = 0 MPa, the matrix acts as a very porous material with no contribution from the fibers except to act as voids. Porosity reduces both strength and modulus in ceramics.[21] The matrix fracture stress for the oxidized composite specimens was

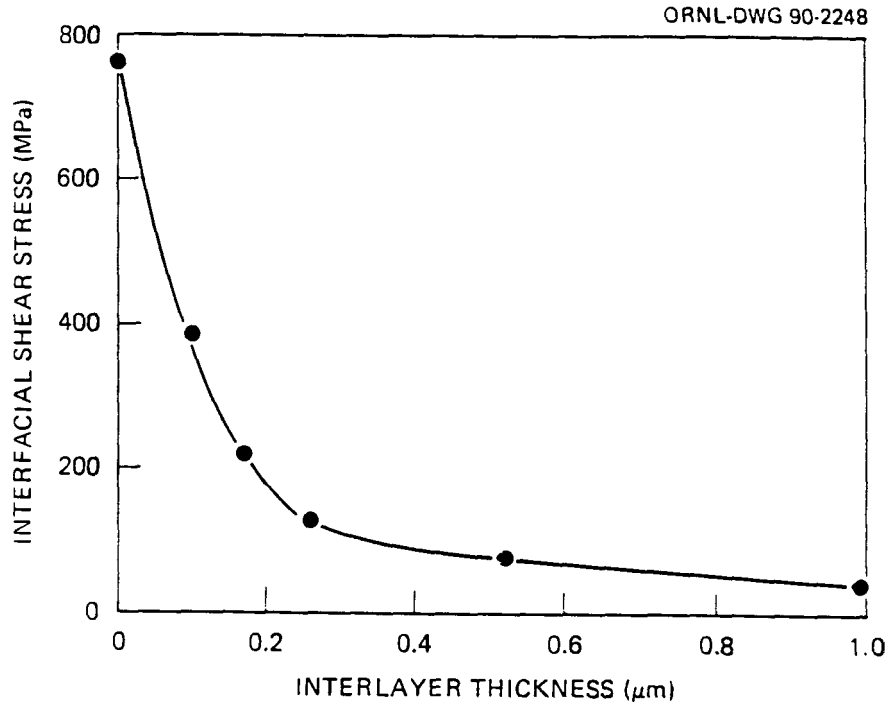

Figure 2. The influence of carbon coating thickness on interfacial frictional stress.

found to be 40 ± 3 MPa which agrees well with the strength of a CVD SiC body with 50% porosity calculated to be ≈ 40 MPa.

The relationship between carbon layer thickness and matrix cracking stress for the Nicalon/SiC composites is graphically depicted in Figure 3. Matrix fracture stress is improved by the application of the graphite interlayer, however, the value quickly decreases with increasing carbon layer thickness. As the thickness of the layer is increased, interfacial shear stress is decreased not allowing for adequate load transfer and thus utilization of the fiber properties to increase matrix cracking stress.

The ultimate flexure strength of Nicalon/SiC composites with varying fiber treatments is also depicted in Figure 3. The lowest strengths were obtained for the composite containing uncoated fibers. The low strength of the composites is due to the strong bonding of the fibers and matrix and fiber property deterioration during processing. The oxidized specimens, not shown in the figure, also exhibited low strength. The lack of interfacial bonding and friction did not allow for load transfer thus as the fibers fractured, they were easily pulled

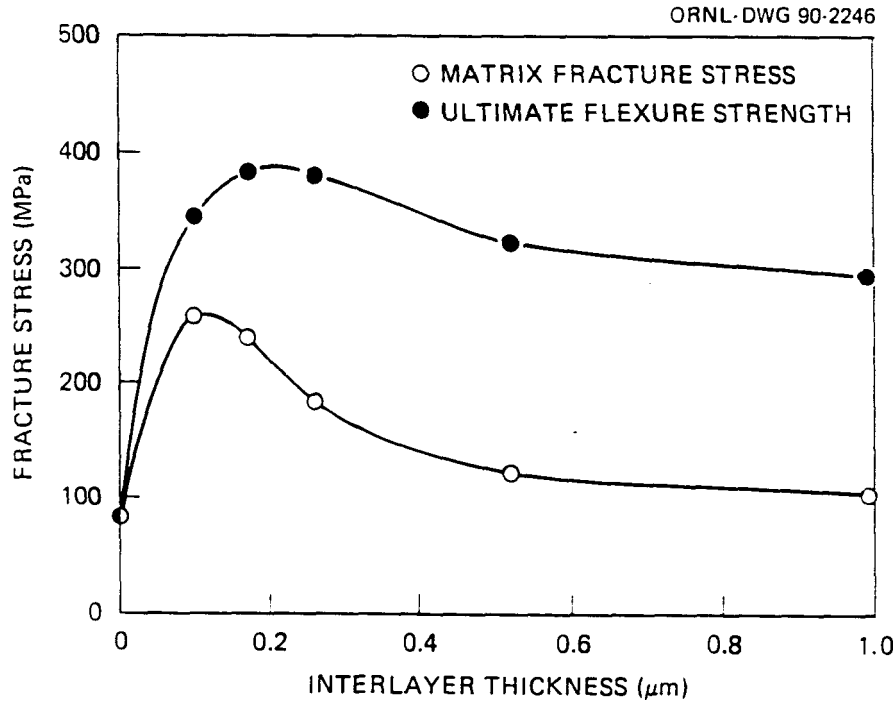

Figure 3. The effect of coating thickness on the matrix fracture stress and ultimate strength of the composites.

from the matrix. The flexure strength of the composites improved with the addition of a carbon fiber coating. An increase in strength was observed with increasing carbon layer thickness up to 0.26 μm, beyond which a degradation of strength was observed.

Work of Fracture

Work of fracture for the Nicalon/SiC composites was determined for un-notched specimens loaded in four-point bending and the results are displayed as a function of coating thickness in Figure 4. Although there was a measurable change in interfacial shear strength for the composites with the thicker interface layers, work of fracture was constant (Table 2). Upon examination of the fracture surfaces of the composites, a decrease in pull-out length with decreasing interlayer thickness was observed. Increasing interfacial shear stress results in a shorter critical length for load transfer thus a reduction in pull-out length. These concurrent changes in interfacial forces and pull-out length are equally offsetting therefore cause the work of fracture over

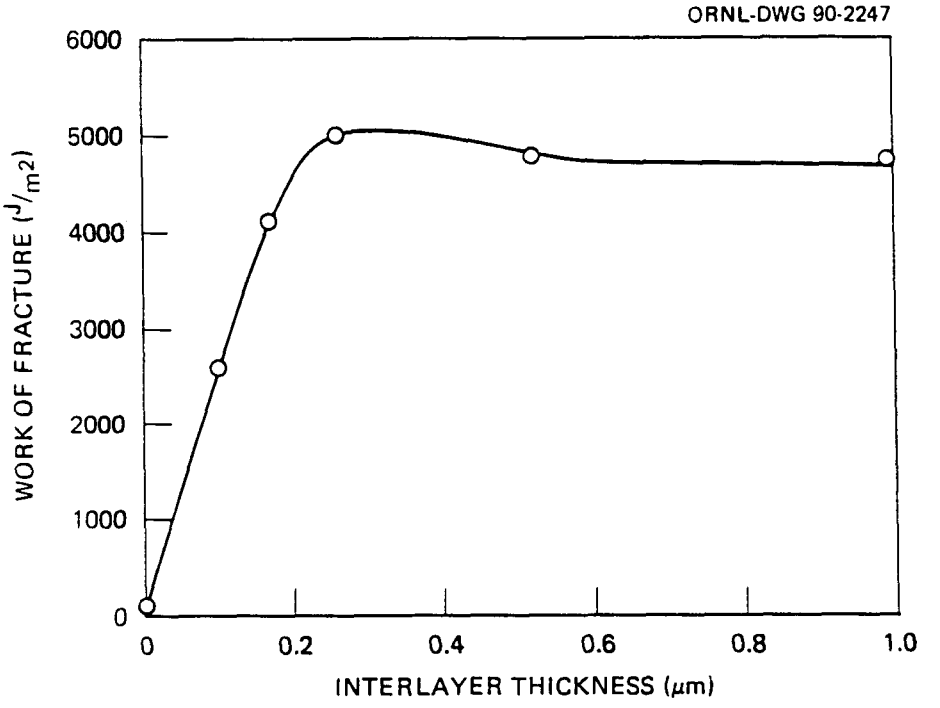

Figure 4. The influence of carbon interlayer thickness of the work of fracture for Nicalon/SiC composites.

the range of interfacial shear strengths to be constant (the same amount of energy is expended to pull a short fiber against high friction as for a longer fiber with lower friction).

The toughness of composites with thinner fiber coatings decreases with the decreasing layer thickness. As the thickness of the coating is decreased, the mechanisms of debonding and frictional sliding change. Fiber debonding and pull-out are more difficult as interfacial shear stress increases, and at a given stress, the mode of failure of the composite is changed. Fiber pull-out lengths becomes extremely short and/or fibers fracture in close proximity to the crack front, resulting in brittle failure and low toughness.

DISCUSSION

The mechanical properties of a fiber-reinforced composites are determined, to a large extent, by the degree of bonding and friction at

the fiber-matrix interface. In the case of uncoated Nicalon fibers in a SiC matrix, extensive chemical bonding and damage to the fibers during processing result in brittle failure of the composite. The application of a carbon interlayer protects the fibers and prevents chemical interaction at the fiber-matrix interface. The properties of the composites are thus influenced by the development of a mechanical bond at the fiber-matrix interface.

Interfacial shear stress in ceramic matrix composites has been described as a combination of the stress necessary to debond the interface, and the Coulomb frictional forces arising from the interaction of compressive stress acting on the fiber with the characteristic coefficient of friction of the interface.[22,23] The compressive stresses acting on the fiber can arise from a number of sources but characteristically arise from thermal expansion mismatch between the fiber and matrix. When the thermal expansion coefficient of the fibers is greater than that of the matrix ($\alpha_f > \alpha_m$), the fibers will shrink away from the matrix upon cooling. If there is poor bonding at the interface, the fiber will separate from the matrix in the radial direction, and neglecting debris or asperities at the debond surface, friction and shear stresses along the interface will be zero. When $\alpha_f < \alpha_m$, the matrix will contract more than the fibers upon cooling. The matrix will radially compress the fibers, increasing the degree of bonding and friction at the fiber-matrix interface.

Typically, thermal expansion mismatch effects, and thus interfacial shear stresses, have been modified by varying the composition of a matrix or by adjusting processing conditions.[16,24-27] Although these changes have a significant influence on the fiber-matrix interface and the mechanical properties of the composites, intercomparison of the influences of the interface is complicated by the consequential changes in matrix and fiber properties associated with altering compositions or processing conditions. Fiber coatings offer an alternative method of varying interfacial shear strength. Fiber coatings have been used to weaken interfacial bonding in a variety of fiber-reinforced ceramic matrix composites. Thin boron nitride coatings were found to control bonding and act as a diffusion barrier in ceramic fiber reinforced oxide matrix composites.[15,28] The

application of BN layers to the fibers prior to processing produced composites with significantly improved strength and toughness as compared to those fabricated from untreated fibers.

The influence of interlayer thickness on interfacial forces and mechanical properties of fiber-reinforced ceramic composites has received little attention. The influence of varying the thickness of a pyrocarbon fiber coating on the properties of Nicalon/LAS composites fabricated using a sol-gel/hot pressing technique has been examined.[29] Interlayer thicknesses up to 1 μm were investigated. An improvement in flexure strength was observed with increasing layer thickness up to a thickness of 0.4 μm beyond which strength decreased. Interfacial shear stresses were not measured and the variations in strength with respect to coating thickness were assumed to be due to mechanisms other than interfacial stresses.

The effects of a film at the interface on the stresses due to thermal contraction mismatch in whisker- and fiber-reinforced ceramic composites has been examined.[30] A reduction in thermomechanical stress is suggested only when a low modulus interfacial coating is present. For Nicalon/SiC composites, the matrix has a higher thermal expansion than the fiber thus upon cooling from processing temperatures, a clamping of the fibers occurs. The residual interfacial compressive stress for uncoated Nicalon fibers in a SiC matrix has been calculated to be $\approx$ 250 MPa, a significant residual compressive stress. The predicted influence of the graphitic carbon coating on the stresses at the fiber-coating-matrix interfaces in Nicalon/SiC composites is shown in Figure 5.

The presence of the carbon interlayer alters the compressive stresses at the interface. The carbon coating is able to deform and act as a buffer layer and thus is able to accommodate a large portion of the residual clamping stress of the matrix. The thickness of the carbon coating influences the clamping stress on the fiber and thus controls interfacial shear stress. Thicker coatings are able to absorb more of the stress caused by the thermal expansion mismatch of the components, therefore interfacial shear strength is inversely proportional to thickness (Figure 6).

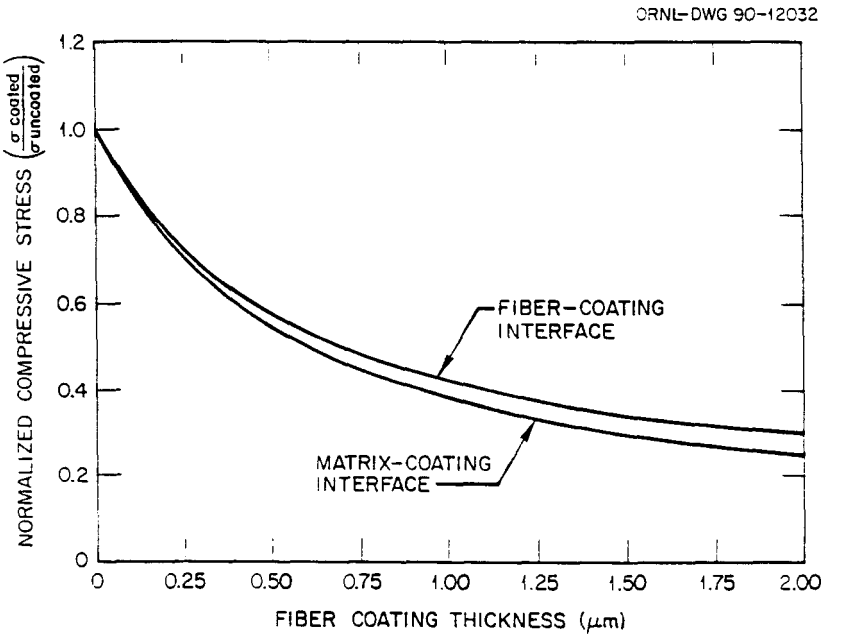

Figure 5. The predicted effects of the carbon interlayer on the compressive stresses at the fiber-matrix interface in Nicalon/SiC composites.

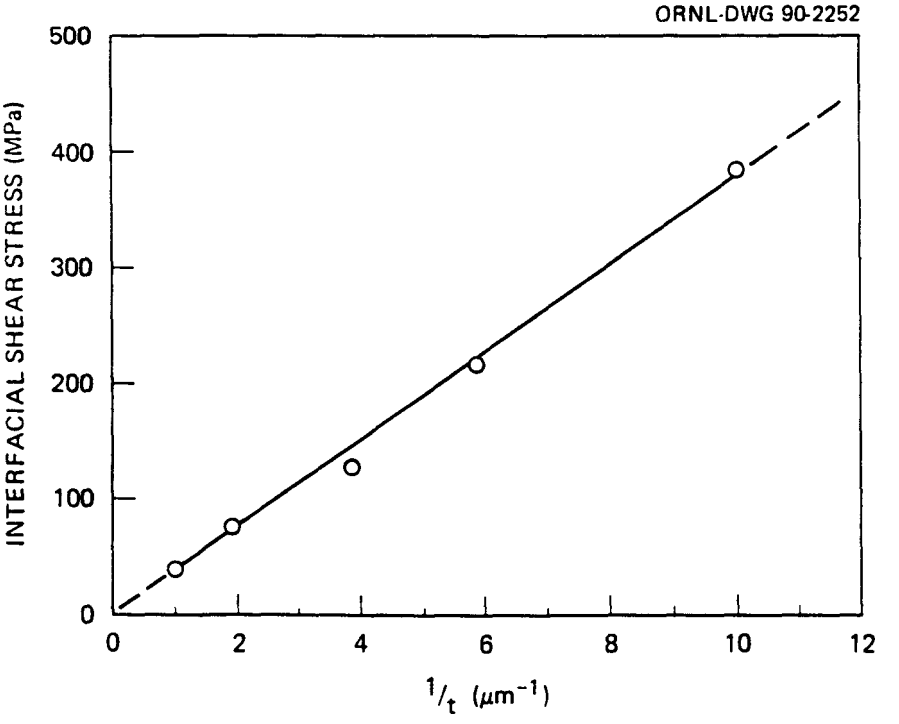

Figure 6. Interfacial shear stress was found to be inversely proportional to coating thickness.

As the thickness of the carbon layer is decreased, interfacial shear stress will increase and at some point exceed the maximum necessary for debonding and pull-out. Crack interaction at the fiber-matrix interface is altered and, in the extreme case corresponding to relatively high interfacial stresses, debonding and crack deflection do not occur, resulting in brittle failure. It may also be possible to compress the coating beyond its limit and thus change the mode of debonding and friction at the interface. In this case, excessive force must be applied to debond and slide the fibers, most likely inducing severe damage to the interlayer and possibly to the fiber and matrix contact surfaces. Interface morphology, thus sliding surface characteristics, would be changed and frictional sliding force would not vary linearly with respect to applied load. This would also result in brittle failure for the composite.

CONCLUSIONS

The fiber-matrix interface in fiber-reinforced ceramic composites controls the mechanical behavior of these materials. An extremely strong bond does not allow for crack deflection or debonding at the fiber-matrix interface therefore a crack propagating in the matrix simply passes through the fibers undisturbed resulting in brittle fracture. Conversely, an extremely weakened interface leads to a low matrix fracture stress and low ultimate strength, for as the composite is stressed, load is not transferred efficiently from the matrix to the fibers, thus the properties of the reinforcement are not utilized. Therefore, interfacial forces must be controlled to produce a composite material with good matrix failure stress and ultimate strength that also exhibits gradual composite failure through effective fiber pull-out.

Carbon has been shown to be an effective interfacial coating for the Nicalon/SiC system. The deposition of a graphitic carbon coating on the fibers prior to infiltration improved the ultimate strength and toughness of the material. The thickness of the coating affects the properties of the interface and thus can be varied to produce material with different mechanical properties. Interfacial shear stress is

inversely proportional to coating thickness. The carbon layer acts as a buffer to accommodate thermal expansion mismatch clamping stress at the fiber-matrix interface. At a thickness < 0.26 μm, the forces at the interface compress the coating beyond its limit and the failure mode of the composite becomes more brittle. This suggests interfacial shear strength, i.e., friction and debonding, is to high and does not permit crack deflection, fiber debonding, and fiber pull-out that are essential for strength and toughness in these brittle-brittle composite systems.

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