

FIBER-MATRIX INTERFACES IN CERAMIC COMPOSITES

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ABSTRACT

The mechanical properties of ceramic matrix composites (CMCs) are governed by the relationships between the matrix, the interface material, and the fibers. In non-oxide matrix systems compliant pyrolytic carbon and BN have been demonstrated to be effective interface materials, allowing for absorption of mismatch stresses between fiber and matrix and offering a poorly bonded interface for crack deflection. The resulting materials have demonstrated remarkable strain/damage tolerance together with high strength. Carbon or BN, however, suffer from oxidative loss in many service environments, and thus there is a major search for oxidation resistant alternatives. This paper will review the issues related to developing a stable and effective interface material for non-oxide matrix CMCs.

INTRODUCTION

For over two decades there has been a strong interest in ceramic materials for a variety of high-temperature, high-stress applications in aerospace, defense, heat engines, and energy conversion.¹⁻³ The driving force for these developments has been the lack of thermal stability, good mechanical properties, and sufficient corrosion resistance of traditional and advanced metallic alloys under these conditions. Advanced ceramics possess the desirable characteristics of low density and high chemical stability, hardness, strength, and modulus, good corrosion resistance, and high melting point for high temperature, structural applications. Unfortunately, these high modulus materials have limited stress relieving mechanisms, are inherently flaw sensitive and are susceptible to thermal shock. Efforts to increase the strain and thermal shock tolerance of ceramics have focussed on introducing second phase materials, such as whiskers, platelets, particles, fibers, and filaments in order to create barriers to crack propagation.⁴⁻⁹ Significant progress in improving fracture toughness has been made using all of these techniques, as well as in self-reinforcing via microstructural design. The most dramatic improvement in strain tolerance, however, has been through reinforcement of ceramic matrices with continuous fibers.^{4,5,9}

This paper will briefly review the role of the interface in continuous fiber-reinforced ceramic matrix composites (CMCs) and the requirement for properly engineered interfaces. Work at several laboratories has provided an understanding of the mechanisms of toughening in CMCs, although uncertainties in their relative importance remain. Extension of the micro-scale characteristics to prediction of macroscopic behavior is currently emerging, with predictive models being proposed. Resulting from these analyses is the realization of the key role of the fiber-matrix interface in determining material properties. In many systems it has been determined that a layer of carbon between the fiber and matrix properly adjusts the bond between

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the components, as well as imparting other benefits, yielding a CMC with desirable mechanical attributes. Yet carbon is susceptible to oxidation, and is therefore not suitable for use in many applications. This paper will therefore also consider alternative interface layers with improved stability. Finally, although there is a significant variety of fiber-reinforced materials, the efforts at the Oak Ridge National Laboratory (ORNL) and a number of other institutions have focussed on polycrystalline SiC matrices, typically reinforced with SiC-based fibers (e.g., Nicalon™, Nippon Carbon, Tokyo, Japan), and thus the Nicalon™/SiC is the subject of this work.

INTERFACE PROPERTIES AND MODELS

As noted above, it is well-understood that the interface between the fiber and matrix is important in determining the properties of CMCs, although the mechanisms that govern those properties are imperfectly characterized.¹⁰⁻¹⁴ A fiber strongly bonded to the matrix in a CMC is likely to promote effective load transfer between the fiber and the matrix. Yet a strongly bound fiber will not arrest or deflect an impinging matrix crack, and therefore the composite will exhibit brittle failure. A weak interface, however, will allow a transverse crack to be deflected with energy absorption occurring via several mechanisms: Debonding at the fiber-matrix interface, crack deflection, crack bridging by the fiber, fiber sliding, and eventual fiber fracture. These energy dissipating mechanisms provide for improved apparent fracture toughness and result in a non-catastrophic mode of failure. Thus, an optimized fiber-matrix interface offers the desired combination of strength and toughness, within the bounds of the material.

The theory of fiber reinforcement for CMCs indicates that the ultimate strength is controlled primarily by the fibers.¹⁷ That is particularly true for the exceptionally high-strength Nicalon™-reinforced SiC system. In addition, the ultimate strength is also dependent on feature/component size (e.g., fiber diameter) and the toughness of the material. Additionally, high strength ceramic fibers increase the apparent toughness of the material and make it flaw and damage-tolerant. Figure 1 is a schematic showing the means by which a relatively weak interface allows for crack deflection and the other mechanisms noted above to absorb crack energy.

A typical stress-displacement curve for a composite (stress in the fiber direction) is seen in Fig. 2. The initial linear region of the curve is representative of elastic deformation governed by the rule of mixtures, with the first departure from linearity at the onset of matrix cracking. As the load increases the matrix continues to microcrack, with the crack population saturating near the ultimate strength. At the maximum stress a significant fraction of the individual fibers begins to fail, resulting in the tail of the stress-displacement curve.

Non-brittle failure requires that matrix cracks that initiate at voids or flaws in the matrix should extend without breaking more than a small fraction of the fibers.¹⁸ The crack spacing is determined by the characteristic stress transfer length associated with the bridging fibers. These bridging fibers minimize crack openings, requiring greater strain for matrix crack propagation. The increasing non-linear portion of the stress displacement curve is governed by the fiber properties, initial adhesion of the fibers to the matrix, and frictional interactions between sliding fibers and the matrix. Large-scale fiber failure determines the ultimate tensile strength and the tail portion of the curve reflects fiber pullout, as fibers are separated from the matrix. Fiber pullout is seen in the fracture surface of Fig. 3, with pullout lengths many times the fiber diameter. These mechanisms are applicable only if the fiber-matrix bond is sufficiently weak, and is observed for similarly behaving brittle matrix composite materials other than CMCs, such as reinforced cements, glasses, and glass-ceramics.

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Fig. 3. Scanning electron micrograph of a typical fracture surface of a Nicalon™/SiC CMC with a mullite interface bounded by thin carbon layers, showing matrix cracks, fiber debonded regions, and fiber pull-out.

Relatively strong fiber-matrix bond strengths can yield non-brittle failure, similar strain/damage-tolerance, and high ultimate strengths if crack branching, and thus energy dissipation, can be promoted within the interface coating.¹⁹ The tensile curves exhibit continuously rising stress to failure and pullout lengths are approximately equivalent to the fiber diameter.

Hsueh, Becher, and Angelini²⁰ have described the importance of a compliant interface layer and Kerans¹³⁻¹⁵ and Singh and Reddy¹⁶ note that all ceramic composites that demonstrate good damage tolerance have at least one layer of carbon or BN between the fiber and matrix. Not only is the presence of such a layer required, ostensibly to modify (weaken) the fiber-matrix bond, but the thickness of this layer is also important in determining the performance of the composite, as quantified by Lowden.¹² The thickness dependence is likely due to a needed level of compliance in the interface layer. Both graphitic, pyrolytic carbon and hexagonal or turbostratic BN have exceptionally low moduli, and thicknesses of the order of 100-500 nm are generally required. It is expected that such a compliant layer is necessary to reduce thermal mismatch stresses which result from, for example, the significantly smaller thermal expansion of Nicalon™ fibers as compared to the SiC matrix. Such stresses have been calculated to cause the fibers to experience a several hundred MPa clamping force upon cooling from a 1000°C processing temperature. Kerans and Singh and Reddy also argue that misfit stresses due to fiber roughness are important during fiber sliding, and that a sufficiently thick compliant layer is required to accommodate that stress and to allow sufficient fiber movement.

ENVIRONMENTALLY STABLE INTERFACES

The initial occurrence of matrix cracking is an important event and signifies the onset of damage and non-recoverable displacement.²¹ Such damage allows environmental attack of the interface and fiber throughout the component. For example, Lin, Becher, and Tortorelli²² have observed substantially reduced lifetimes of NicalonTM-SiC composites with a 300 nm carbon interface layer in elevated temperature static fatigue. At <100 MPa stress there is little effect, yet at higher stresses the matrix becomes substantially microcracked and the carbon interface is lost due to oxidation at temperatures as low as 425°C. The resulting times to failure decline with increasing temperature. Studies of BN interfaces in SiC matrix material have shown improved behavior, although BN is also susceptible to oxidation and may promote the oxidation of the fibers as well.²³ BN interfaces are particularly problematical at intermediate temperatures in the presence of moisture where hydrated vapor species have substantial vapor pressures.

The implication of these observations is that unless the CMC experiences stresses below the linear elastic limit, the fibers and interface layers will be exposed to the ambient environment with the potential for oxidative damage. Given the relatively low stress levels for matrix cracking as compared to the ultimate strength of these materials, it behooves materials developers to devise systems which are inherently stable, and that must include a stable interface layer or set of layers. Efforts at ORNL have, therefore, focussed on developing oxidation-resistant interface layers for NicalonTM-SiC composites. Criteria for an interface system include:

- Promote relatively weak interface between fiber and matrix, but still provide for load transfer.
- Protect fibers from matrix processing and service environment, if necessary.
- Utilize a viable processing route that does not damage the fibers.
- Provide a uniform and homogeneous surface.
- Be thermochemically stable with respect to processing and service environment, fiber, and matrix.
- Possess a low modulus for stress absorption.
- Possess an appropriate thermal expansion coefficient.

Coating work to date has largely involved the use of vapor phase, solution, or sol-gel techniques to apply layers to fibers. Although it is likely that uniform, high quality CMCs will require vapor phase application of interlayers, it was determined that screening work, especially for oxides, was most easily performed using wet chemical means. The base composite system for the investigations has been ceramic grade NicalonTM densified with SiC via forced-flow, thermal-gradient chemical vapor infiltration.⁹

Walukas²⁴ performed some initial work utilizing sol-gel-derived interface layers of alumina, zirconia, and titania provided by the Westinghouse Science and Technology Center. Initial results with the alumina coatings were encouraging as they exhibited moderate strength that did not greatly suffer from exposure to 1000°C air for 1000 h. (Testing involved exposing flexure test bars having ground surfaces which allow access of oxygen to the interface. Carbon-coated fiber material shows substantial degradation after only several hours of such exposure.) Transmission electron microscopy of the coatings, however, revealed that they were very thin (< 100 nm) without uniform coverage, and that the alumina had converted to mullite, possibly via reaction with silica due to oxidation of the NicalonTM fiber.

Shanmugham et al.²⁵ evaluated the fracture behavior of CMCs with carbon, alumina, and mullite interfacial coatings as processed and after exposure to air at 1000°C. Colloidal processes

were used to prepare the oxide coatings, and chemical vapor deposition using propylene was used to form the ~300 nm carbon interface. In this case, CMCs with both carbon and alumina interfaces exhibited brittle failure after oxidation, whereas the mullite interface material retained fiber pull-out. The colloidal process, however, failed to uniformly coat the fibers, and therefore regions of pull-out were found adjacent to those exhibiting brittle fracture. Subsequent efforts to utilize sol-gel deposition of mullite and alumina-titania on Nicalon™, however, resulted in material which suffered brittle failure as-fabricated, likely due to the degradation of the fibers by the sol-gel processing technique.

With the object of protecting the fiber from damage during the sol-gel deposition of oxide coatings, and to protect the oxides from attack by the precursors for SiC matrix deposition, thin carbon films were applied to the fibers before and after mullite layer formation. The carbon was again formed by the pyrolysis of propylene for periods that allowed <50 nm to be deposited. From the earlier work of Lowden¹² such thin carbon coatings are not expected to provide the necessary interface modification to allow for good composite properties, and their thinness was also not expected to cause the composite to lose strength due to oxidation of the carbon. The results of the oxidation testing of machined surfaces exposed to 1000°C air for 500 h followed by four-point flexure testing at room temperature can be seen in Fig. 4. Although good composite behavior seems to be retained, the composites lose strength during the high temperature exposure, with the mullite interface material decreasing from 268±52 MPa to 184±28 MPa, and the alumina-titania interface material decreasing from 255±35 MPa to 159±20 MPa. This observed loss of strength may be due to degradation/oxidation of the Nicalon™ fibers by the lengthy period at elevated temperature or by interaction of the fibers with the interface material. In addition, post-processing examination of the mullite coating indicated that it did not uniformly cover the fibers.

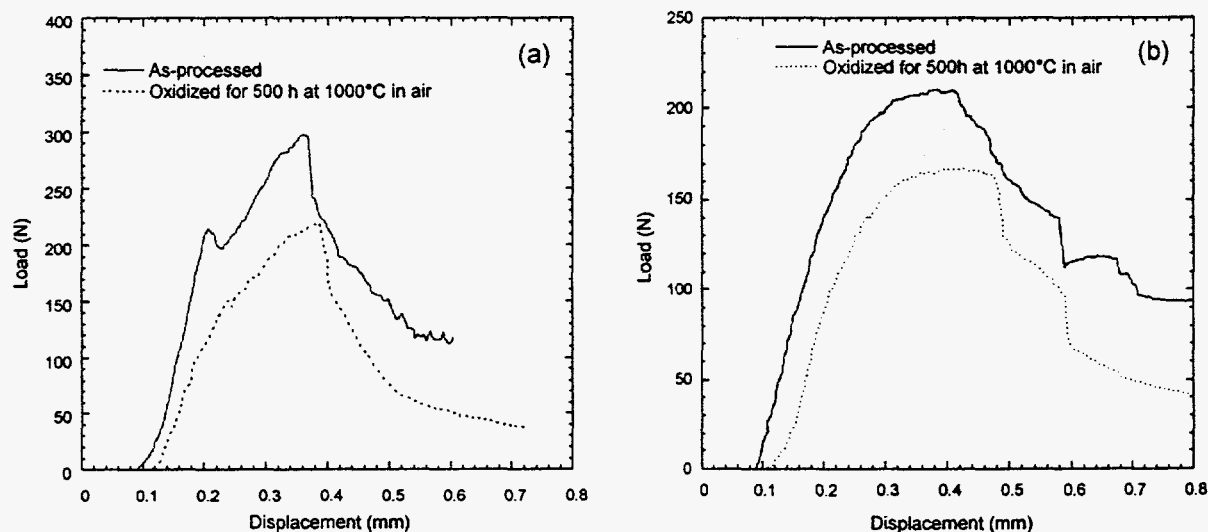


Fig. 4. Room temperature four-point flexure curves for as-fabricated and 500 h, 1000°C air exposed Nicalon™/SiC composites (a) with a mullite interface coating and (b) with an alumina-titania interface coating. (Specimens are approximately 2.5 x 3 x 33 mm.)

A transmission electron micrograph (TEM) of the alumina-titania interface material after static exposure to air at 1000°C for 500 h exhibits some interesting features (Fig. 5). The interface film is seen to be crystalline, coherent, and relatively uniform, however only 40-50 nm in thickness. The ostensibly bounding layers that had originally been carbon have been replaced by amorphous silica grown from the Nicalon fiber and the SiC matrix, with additional silica thickness grown into fiber and matrix. The mechanical properties of the system, as noted above, are promising and it appears that even the thin alumina-titania interface coating serves the purpose of blunting and deflecting cracks. The strength loss may thus be attributed to oxidation of the fibers, with even the fiber surface oxidation having a substantial effect on strength.^{16,26-28}

In another approach at ORNL, efforts are on-going to utilize porous or poorly consolidated SiC as an interlayer. Methylsilane was used as the chemical vapor deposition precursor to deposit what appears to be porous or poorly consolidated SiC.^{29,30} This precursor is sensitive to thermal decomposition to SiC and its rapid deposition does not allow high density, crystalline coatings to form. These coatings are expected to be weak and thus crack propagation is promoted within the interface material as opposed to the fiber-coating or coating-matrix interface. Scanning electron micrographs of coated fibers (Fig. 6) reveal the coating's friability and poor adhesion to NicalonTM. Fracture surfaces reveal substantial fiber pull-out both before and after oxidation (Fig. 7). Four-point flexure strength measurements indicate that the material is toughened and appears to retain toughness after oxidative exposure, although again demonstrating loss of strength (Fig. 8). As-infiltrated, the flexure strength of the CMC is 381±46 MPa, after 24 h at 1000°C in air it is 344±69 MPa, and after 100 h it is 318±76 MPa. As previously noted, it is uncertain whether the observed decrease in strength with temperature is due to thermal degradation/oxidation of the fibers since composite behavior, i.e., the strain/damage-tolerance, is retained.

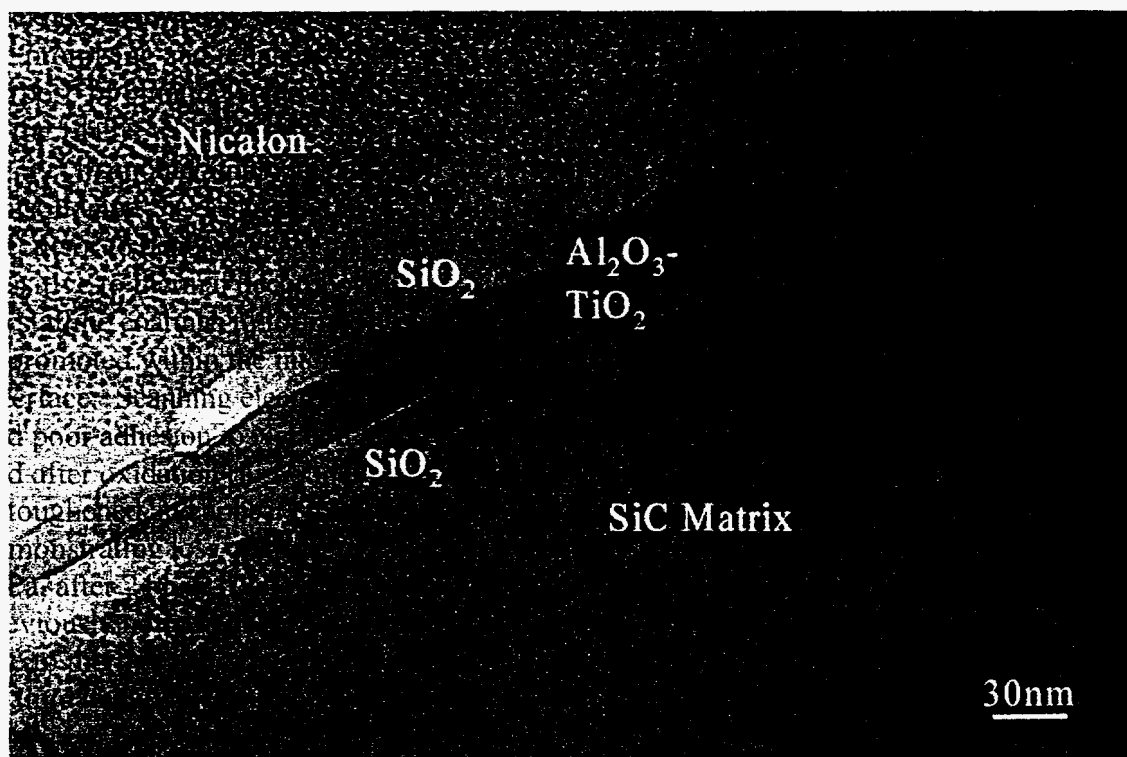
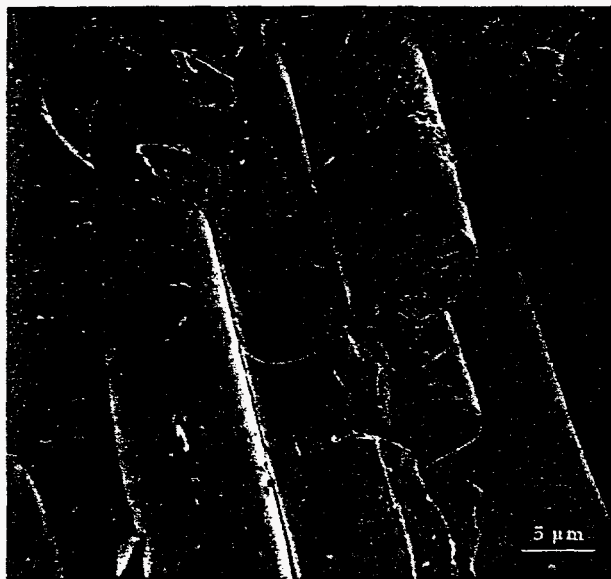


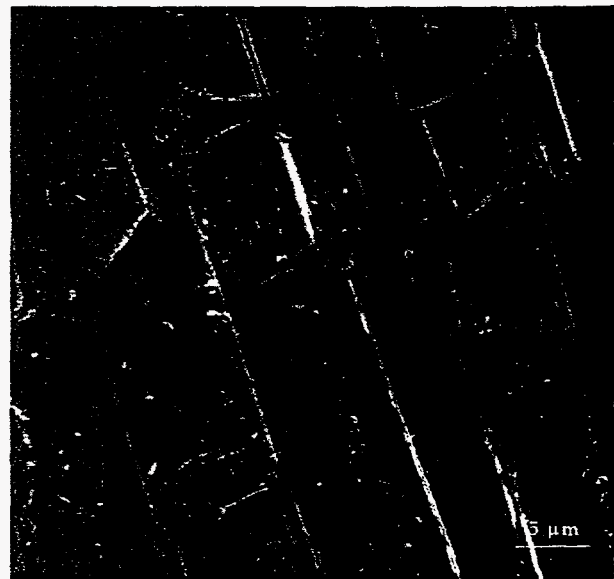
Fig. 5. TEM image of the oxidized (500 h at 1000°C in air) alumina-titania interface material with the bounding carbon layers replaced by silica grown from the SiC matrix and NicalonTM.



Fig. 6. Poorly consolidated/amorphous SiC coating deposited on NicalonTM illustrating its friability and poor adhesion.



(a)



(b)

Fig. 7. SEM images of fracture surfaces of a NicalonTM-SiC composite with a poorly consolidated SiC interface (a) as-fabricated and (b) after exposure in air at 1000°C for 100h.

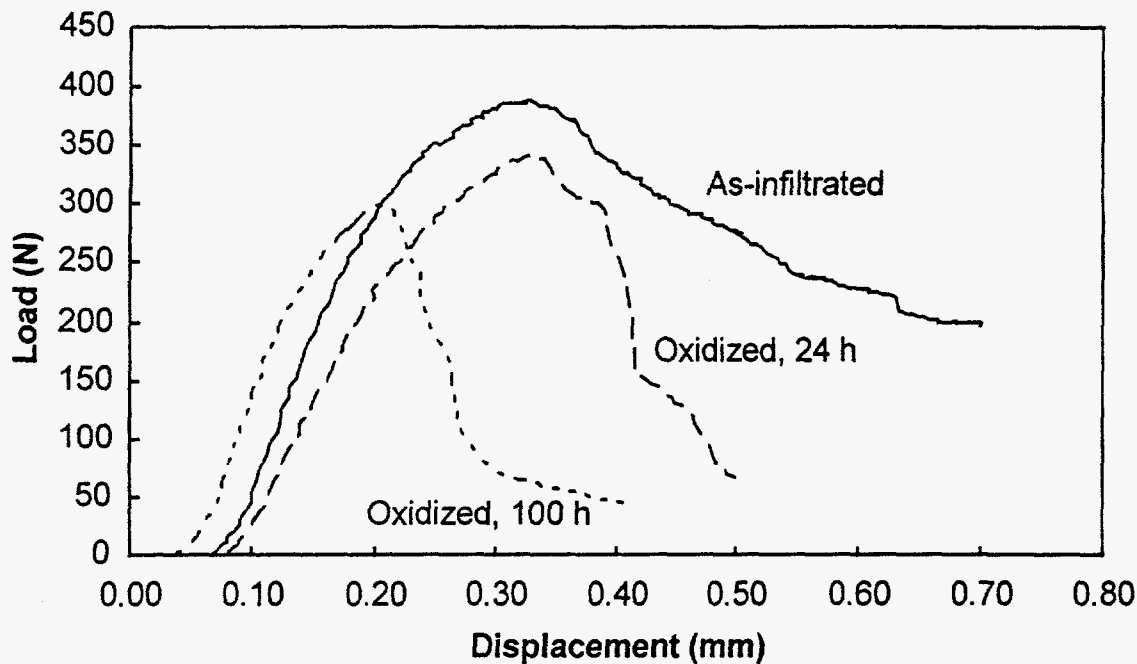


Fig. 8. Room temperature four-point flexure curves for as-fabricated and oxidized Nicalon™/SiC composites with a poorly consolidated SiC interface coating.

UNRESOLVED ISSUES IN CMC INTERFACES

As seen in the work at ORNL and elsewhere, it is possible to prepare environmentally stable interfaces for non-oxide CMCs, yet problems and questions as to desired constituent properties remain. In contemplating the unresolved issues related to interfaces they fall into several categories that include fabrication, desired failure mode, and constituent properties as they affect the stress states of the composite constituents. Although a detailed review of the background of these problems is beyond the scope of this paper, it is useful to be familiar with nature of the issues.

Fabrication

The deposition of desired interface coatings on small diameter fibers is a substantial challenge. The coatings must be relatively uniform throughout a continuous tow or woven cloth or layup. The process must therefore allow for infiltration of the coating precursor into the fiber bundle yet not result in bridging between fibers or overcoating of the bundle. Interestingly, these challenges have been met for a variety of processes including solution, sol-gel, and chemical vapor deposition (CVD).^{19,31,32} A more difficult problem has arisen, however, and that is the degradation of the fibers by the application of the interface coating. Fiber degradation occurs for the most cost-effective fibers, such as silicon carbide-based Nicalon™ and the various oxides of the Nextel™ series of fibers, which are produced from preceramic polymeric precursors.^{23,27} Solution or sol-gel processes typically cause significant fiber strength loss, an explanation for which remains elusive. Chemical vapor deposition processes are believed to damage the fibers if the deposition temperature is too high or if corrosive or reactive precursors are used. As noted

above, protecting the fibers with a thin layer of deposited carbon has been an effective, but less than desirable solution.

Provided adequate interface coatings can be deposited on fiber tows, their incorporation into a preform may result in loss or damage to the coatings due to handling. The non-uniform stresses of weaving and other preform assembly operations would be expected to affect the coatings, and the coated fibers themselves may no longer have the necessary modulus or inter-fiber frictional properties for these operations.

Failure Mode

The mechanism of crack deflection results in a crack being diverted to propagate parallel to the fiber axis. The question, however, is along which interface, if any, do we desire the crack to travel? Typical fracture surfaces for systems such as carbon-coated NicalonTM with a SiC matrix generally reveal that the crack has uniformly followed the fiber-coating interface, due to the poor adhesion of the carbon to the fiber. Naslain¹⁹, Kerans¹⁵, and others have noted that higher strengths may be achievable provided crack energies can be dissipated within a media such as the fiber-matrix interface, or perhaps within the matrix itself. This requires that the interface be bonded sufficiently well to the matrix and fiber, yet that the interface material contains sufficient internal surfaces, e.g., crystallographic planes or interlaminar interfaces parallel to the fibers, or be inherently weak so as to undergo shear due to an impinging crack, thus absorbing the crack energy. Naslain, in particular, has described such phenomena in material in which the NicalonTM has been subjected to a surface treatment that allows carbon to strongly adhere to the fiber. Thus, the weakest interface is no longer between fiber and coating, but rather within the carbon coating itself. The material has a similar strain-to-failure as that containing untreated NicalonTM, yet the treated fiber results in significantly higher strength due to crack propagation within the carbon coating instead of at the fiber-coating interface.

Material Properties

Kerans¹⁵ and Singh and Reddy¹⁶ have suggested that a compliant interface layer is required to accommodate misfit stresses arising from fiber movement within the matrix and thermal mismatch stresses. Similar results with regard to thermomechanical stresses are obtained by Shanmugham et al.²⁵ from finite element analyses. All these calculations indicate that oxide layer thicknesses of the order of 0.5 to 1 μm , or even greater, are needed to cause a significant reduction in the radial stress on a fiber in systems with a thermal expansion coefficient mismatch equivalent to that between SiC and NicalonTM. Yet the recent work at ORNL reported here indicates that even thin (< 100 nm) oxide interface coatings have resulted in material with fiber pull-out and considerable strain-to-failure. These types of results thus leave uncertain what should be the desirable mechanical properties of an interface material or system. Finally, the chemical stability between fiber, interface layer(s), and matrix strongly effects both degradation of the fiber and the strength of the bond between surfaces. While one would expect that systems which leave the fiber unaffected and are phase-stable are desired, the reality of process/materials choices often require some compromise.

CONCLUSIONS

Observation and micromechanical modeling have revealed the necessity of an interface layer between the fiber and matrix in CMCs that has the attributes of adjusting the fiber-matrix

bond and accommodating thermal mismatch and geometrical misfit stresses. Although pyrolytic carbon has well-served that need, its susceptibility to oxidation has precluded its use in high-temperature, oxidative environments where stresses cause matrix cracking that allows ingress of air. Efforts at ORNL to replace carbon with oxidation-resistant materials have met with some initial success. Oxides such as mullite and alumina-titania have been seen to preserve composite strength and strain-tolerance after substantial oxidative exposure. A porous or poorly consolidated SiC interface layer has also shown promise. Yet there remain substantial questions with regard to acceptable fabrication techniques, optimal failure modes, and desired interfacial material properties. Further understanding of these complex systems together with continuing development of these and other interface concepts appears likely to allow CMCs to be sufficiently stable in high-temperature, oxidative environments for new applications in fossil fuel burning, heat recovery, high temperature filtration, and propulsion systems.

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