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Effects of Fiber/Matrix Interface and its Composition on Mechanical Properties of Hi-Nicalon/Celsian Composites

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EFFECTS OF FIBER/MATRIX INTERFACE AND ITS COMPOSITION ON MECHANICAL PROPERTIES OF HI-NICALON/CELSIAN COMPOSITES

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SUMMARY: To evaluate the effects of fiber coatings on composite mechanical properties, unidirectional celsian matrix composites reinforced with uncoated Hi-Nicalon fibers and those precoated with a dual BN/SiC layer in two separate batches (batch 1 and batch 2) were tested in three-point flexure. The uncoated-fiber reinforced composites showed catastrophic failure with strength of 210 ± 35 MPa and a flat fracture surface. In contrast, composites reinforced with coated fibers exhibited graceful failure with extensive fiber pullout and showed significantly higher ultimate strengths, 904 and 759 MPa for the batch 1 and 2 coatings, respectively. Fiber push-in tests and microscopic examination indicated no chemical reaction at the uncoated or coated fiber-matrix interfaces that might be responsible for fiber strength degradation. Instead, the low strength of composite with uncoated fibers was due to degradation of the fiber strength from mechanical damage during composite processing. Despite identical processing, the first matrix cracking stresses (σ_{mc}) of the composites reinforced with fibers coated in batch 1 and batch 2 were quite different, 436 and 122 MPa, respectively. The large difference in σ_{mc} of the coated-fiber composites was attributed to differences in fiber sliding stresses ($\tau_{friction}$), 121.2 ± 48.7 and 10.4 ± 3.1 MPa, respectively, for the two composites as determined by the fiber push-in method. Such a large difference in $\tau_{friction}$ for the two composites was found to be due to the difference in the compositions of the interface coatings. Scanning Auger microprobe analysis revealed the presence of carbon layers between the fiber and BN, and also between the BN and SiC coatings in the composite showing lower $\tau_{friction}$. This resulted in lower σ_{mc}, in agreement with the ACK theory. The ultimate strengths of the two composites depended mainly on the fiber volume fraction and were not significantly effected by $\tau_{friction}$ values, as expected. The poor reproducibility of the fiber coating composition between the two batches was judged to be the primary source of the large differences in performance of the two composites.

KEYWORDS: oxide matrix composite, fiber-matrix interface, interphase composition, fiber coatings, fiber push-in, mechanical properties, microstructure, silicon carbide fiber

INTRODUCTION

Fiber-reinforced ceramic matrix composites (CMC) are prospective candidate materials for high temperature structural applications in aerospace, energy conservation, power generation, nuclear, petrochemical, and other industries. At NASA Glenn Research Center (GRC), we are investigating celsian matrix composites [1-6] reinforced with various types of silicon carbide fibers. A crack-deflecting fiber/matrix interface is required in order to produce a strong and tough composite. Towards this end, the objective of the present study was to investigate the effects of fiber/matrix interface and its composition on the mechanical properties of silicon carbide (Hi-Nicalon) fiber-reinforced celsian matrix composites.

EXPERIMENTAL METHODS

Polymer-derived, low oxygen content, Hi-Nicalon fiber tows in the as-received condition and those precoated with a dual layer of BN/SiC by chemical vapor deposition in two separate batches, were used as the reinforcements. The nominal coating thicknesses were 0.4 μm of BN and 0.3 μm of SiC. Fiber-reinforced composites were fabricated as described earlier [7]. The fiber tows were impregnated with a matrix precursor slurry and wound on a drum. The prepreg tape was cut, stacked up (12 plies) in desired orientation and warm pressed. The fugitive organics were slowly burned out in air followed by hot pressing under vacuum in a graphite die resulting in an almost fully dense composite. The composite panel was surface polished and sliced into test bars (~50.4 x 6.4 x 1.9 mm^3) for mechanical testing.

Composite mechanical properties were determined from stress-strain curves recorded in 3-point flexure using a support span of 40 mm at a crosshead speed of 0.127 cm/min. Strain gauges were glued to the tensile surfaces of the flexural test bars. Interfacial mechanical properties were determined by cyclic fiber push-in tests performed using a desktop apparatus [8]. Thin sections of the CMCs, cut normal to the fiber axis and polished down to a 0.1 μm finish on both top and bottom faces, were tested. Fibers were pushed with a conical diamond indenter (70° included angle) with a 10 μm diameter flat base.

Chemical composition of the fiber coatings was determined by scanning Auger microprobe analysis using a Fisons Instruments Microlab Model 310-F. Elemental analysis at the fiber-matrix interface was also done with an ARL-SEM-Q electron microprobe. Matrix phase analysis was performed by X-ray diffraction (XRD) employing copper K_α radiation using a Philips ADP-3600 automated diffractometer equipped with a crystal monochromator. Microstructures of the polished cross-sections and fracture surfaces were observed in an optical microscope as well as by scanning electron microscope (SEM). Prior to analysis, a thin carbon coating was deposited onto the SEM specimens for electrical conductivity.

EXPERIMENTAL RESULTS

Microstructure

X-ray diffraction patterns taken from the polished surface of the CMC indicated the presence of monoclinic celsian with no detectable level of the undesired hexacelsian phase. This implied that the desired monoclinic celsian was formed *in situ*, from the mixed oxide precursor,

during hot pressing of the CMC. SEM micrographs taken from the polished cross sections indicated uniform fiber distribution and good matrix infiltration within the fiber tows. Occasional pores, particularly within the fiber tows, were present. The outer SiC coating occasionally debonded from some of the fibers in both coated-fiber composites during composite processing. In addition, the BN/SiC duplex coating was occasionally completely detached (atypical) from some of the fibers in the CMC reinforced with batch 2 coated fibers.

Mechanical Properties

Typical stress-strain curves recorded in three-point flexure of the composites reinforced with uncoated and BN-SiC coated Hi-Nicalon fibers are shown in Fig. 1. The stress-strain curve for a hot pressed BSAS monolith [9] is also shown for comparison. The monolith shows a modulus of 96 GPa, flexural strength of 131 MPa and fails in a brittle mode as expected. The uncoated fiber reinforced composite also shows catastrophic failure. In contrast, the BN-SiC coated fiber-reinforced composites show initial linear elastic behavior followed by an extended region beyond the initial deviation from linearity. This indicates load transfer to the fibers beyond the proportional limit indicating a true composite behavior. Room temperature mechanical properties of the various composites of this study are given in Table 1. The CMCs containing coated fibers had lower modulus than the uncoated fiber-reinforced composite due to the presence of the low-modulus BN layer. Both the composites reinforced with BN/SiC coated fibers show high ultimate strength in accordance with the value of the fiber volume fraction. However, a large difference is observed in the values of σ_{mc} for the two coated-fiber reinforced composites.

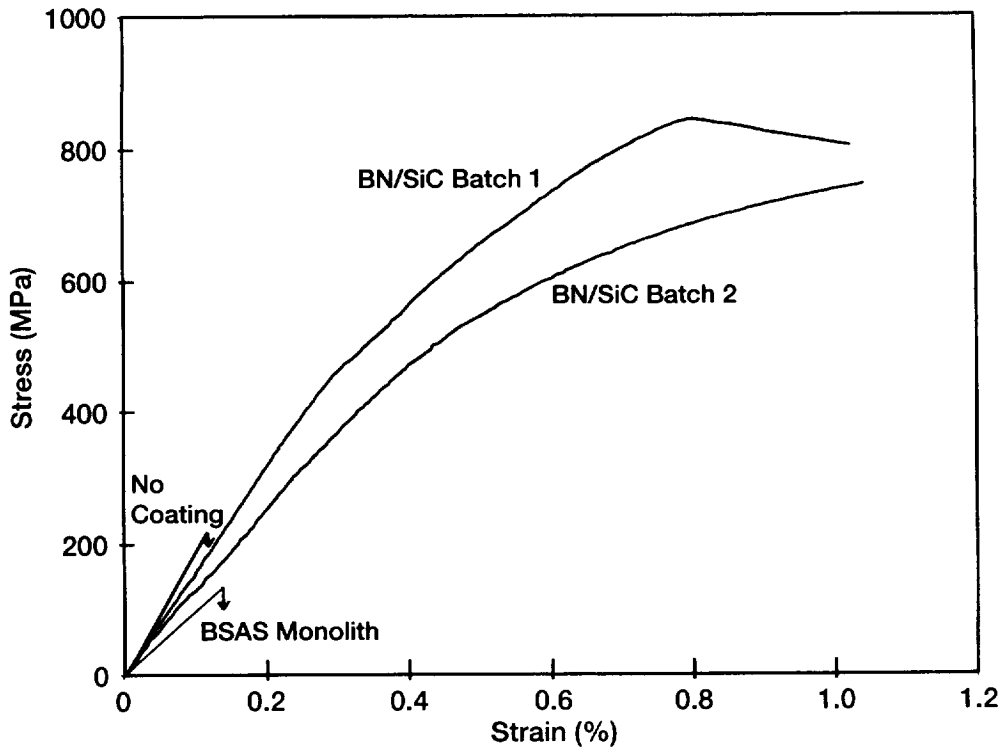

Figure 1: Apparent stress-strain curves recorded in three-point flexure for celsian matrix composites reinforced with uncoated Hi-Nicalon fibers and those coated with BN/SiC in two separate batches. Also shown for comparison are the results for a hot pressed BSAS monolith.

Table 1: Mechanical properties[a] of unidirectional Hi-Nicalon/celsian composites

Interface coatings	V_f	E, GPa	σ_{mc}, MPa	ε_y, %	σ_u, MPa	ε_u, %
None	0.45	184 ± 4	---	---	195 ± 24	0.106 ± 0.01
BN/SiC (Batch 1)	0.43	164 ± 4	436 ± 32	0.270 ± 0.01	904 ± 54	0.731 ± 0.07
BN/SiC (Batch 2)	0.32	137	122	0.091	759	1.041

[a]Measured at room temperature in 3-point flexure.

SEM micrographs of fracture surfaces of the uncoated and BN-SiC coated fiber-reinforced composites, after the three-point flexure tests, are shown in Fig. 2. Extensive long lengths of fiber pullout are observed in the Hi-Nicalon/BN/SiC/BSAS composites indicating toughening behavior. The fiber pullout lengths are larger in batch 1 composite than in batch 2 composite. In contrast, the fracture surface of the uncoated fiber-reinforced composite shows little fiber pullout, consistent with the observed catastrophic failure.

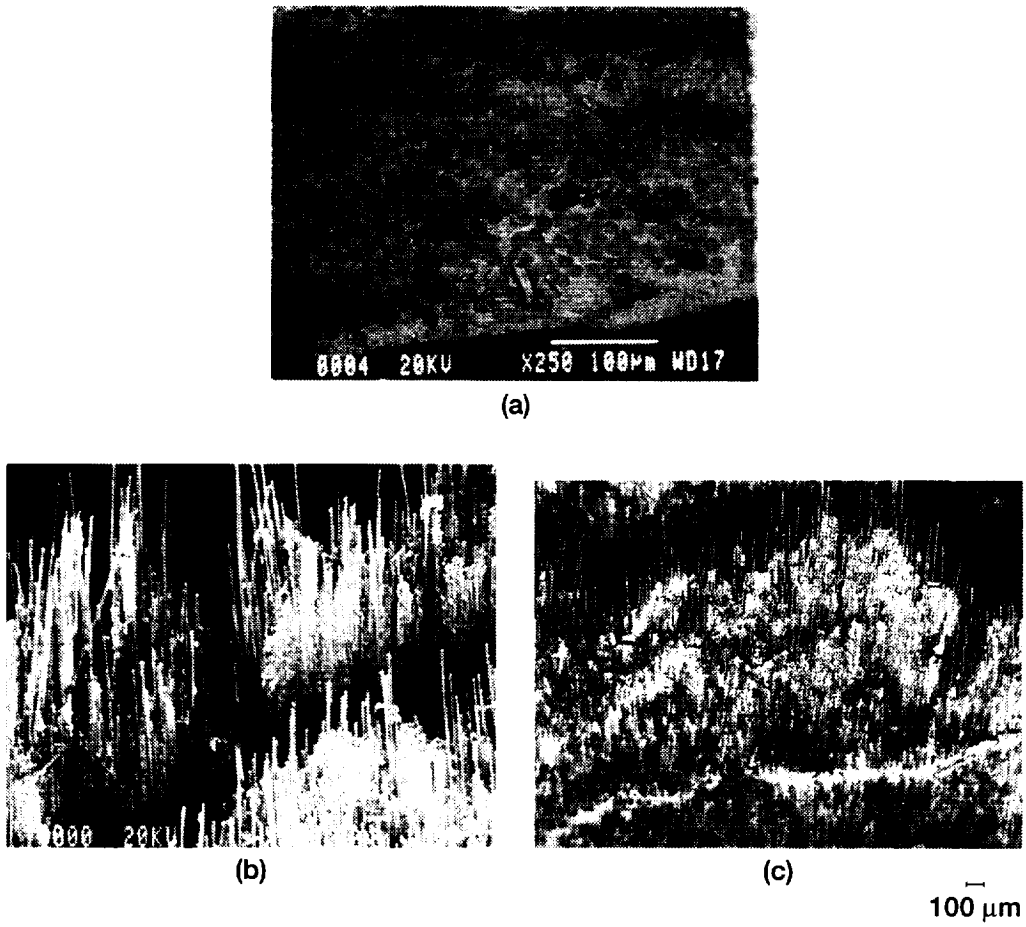

Figure 2: SEM micrographs showing fracture surfaces of celsian matrix composites reinforced with Hi-Nicalon fibers: (a) uncoated, (b) BN/SiC batch 1, and (c) BN/SiC batch 2 coatings.

Fiber-Matrix Interface

For tough composites, the fiber-matrix interface must be sufficiently weak to allow debonding at the interface, yet strong enough for effective load transfer from the matrix to the fiber. In order to determine whether differences in interfacial behavior were the source of the large disparities observed in the mechanical behavior of the composites with different interfaces, fiber debonding and frictional sliding stresses at the fiber-matrix interface were evaluated from fiber push-in tests. A number of fibers were pushed in for each composite. Typical cyclic push-in curves at room temperature for the various composites are shown in Fig. 3. The data were analyzed by first subtracting the appropriate load-train compliance correction from the measured displacements. An estimate of frictional sliding stress, $\tau_{friction}$, was determined using the constant $\tau_{friction}$ model of Marshall and Oliver [10] which includes effects of residual stresses, but does not consider fiber roughness or Poisson expansion. Values of $\tau_{friction}$ were determined by fitting the compliance corrected data from the first reloading curve to the relationship:

$$u = u_0 + [F^2 / (8\pi^2 r_f^3 E_f \tau_{friction})] \quad (1)$$

where u is the fiber end displacement, u_0 is the residual fiber end displacement after the previous unloading, F is the applied load, r_f is the fiber radius, and E_f is the fiber modulus. While neglecting Poisson expansion of the fibers leads to an overestimation of $\tau_{friction}$ values, the relative comparison of $\tau_{friction}$ for different coatings should be valid. In addition, a debond initiation stress, σ_d, could be calculated from the debond initiation load, F_d, (load at which fiber end begins to move during first loading cycle) by the relation

$$\sigma_d = F_d/\pi r_f^2. \quad (2)$$

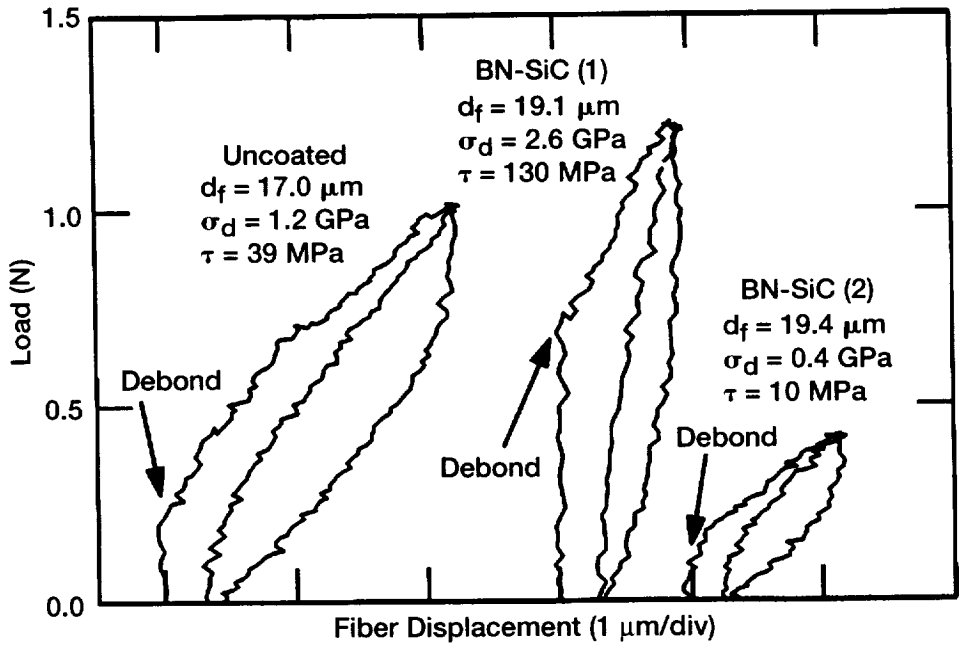

Figure 3: Load versus fiber displacement curves recorded during fiber push-in testing of celsian matrix composites reinforced with uncoated Hi-Nicalon fibers and those coated with BN/SiC in two separate batches.

The results of fiber push-in data for various composites are summarized in Table 2. For the two composites with BN/SiC interface coatings, values of σ_d are 1.95 ± 0.87 and 0.31 ± 0.14 GPa and $\tau_{friction}$ are 121.2 ± 48.7 and 10.4 ± 3.1 MPa, for batch 1 and 2, respectively. Thus, the values of debonding stress and frictional sliding stress are much higher for composites with batch 1 coated fiber than with batch 2 coated fiber. Such a large difference in the values of σ_d and $\tau_{friction}$ for the two composites, fabricated under the same conditions and having "similar" interface coatings, was unexpected. To search for differences in coating composition that could explain the disparity in fiber debonding and sliding behavior, elemental compositions of the duplex BN/SiC coatings on the fibers were analyzed by scanning Auger microprobe.

Table 2. Summary of fiber push-in results for Hi-Nicalon/celsian composites

Interface coatings	V_f	No. of tests	σ_d, GPa	$\tau_{friction}$, MPa
None	0.45	39	1.41 ± 0.67	45.4 ± 36.2
BN/SiC (Batch 1)	0.43	50	1.95 ± 0.87	121.2 ± 48.7
BN/SiC (Batch 2)	0.32	19	0.31 ± 0.14	10.4 ± 3.1

Scanning Auger Analysis

Elemental composition depth profiles obtained from scanning Auger microprobe analysis for the two batches of BN/SiC coatings on Hi-Nicalon fibers are shown in Fig. 4. The batch 1 coating consists of ~0.7 μm thick outer layer of slightly silicon-rich SiC followed by a boron-rich BN layer ~1.5 μm thick. The BN layer also contains ~ 15 atom percent of carbon and ~2 percent oxygen. The thickness of the dual coating on this filament is much higher than the nominal coating thickness of 0.4 μm BN and 0.3 μm SiC. The batch 2 coating consists of ~0.15 μm thick Si-rich SiC followed by ~0.6 μm of carbon rich "BN". In addition, unintentionally deposited carbon layers are also present between the SiC and "BN" coatings and between the "BN" and the fiber surface. Thus the coatings deposited on the fibers in the two batches differ in composition and structure.

DISCUSSION

Low strength, catastrophic failure and flat fracture surface with no fiber pullout observed for the uncoated fiber-reinforced composite could be due to strong bonding of the fibers with the oxide matrix during hot pressing. However, modest stresses required to initiate fiber-matrix debonding (Table 2) during fiber push-in, as well as microscopic examination of the pushed-in fibers indicated no chemical reaction between the uncoated or coated fibers and the matrix during composite hot pressing. Also, electron microprobe analysis [11] of a polished cross-section of the CMC with uncoated fibers indicated no interdiffusion of the elements at the fiber-matrix interface. An alternate explanation for such a low strength of the uncoated fiber-reinforced composites could be mechanical damage to the fibers during composite processing resulting in fiber strength degradation. While the BN-SiC dual layer was applied to promote a weak interface, this coating may more importantly protect the fiber surface from mechanical

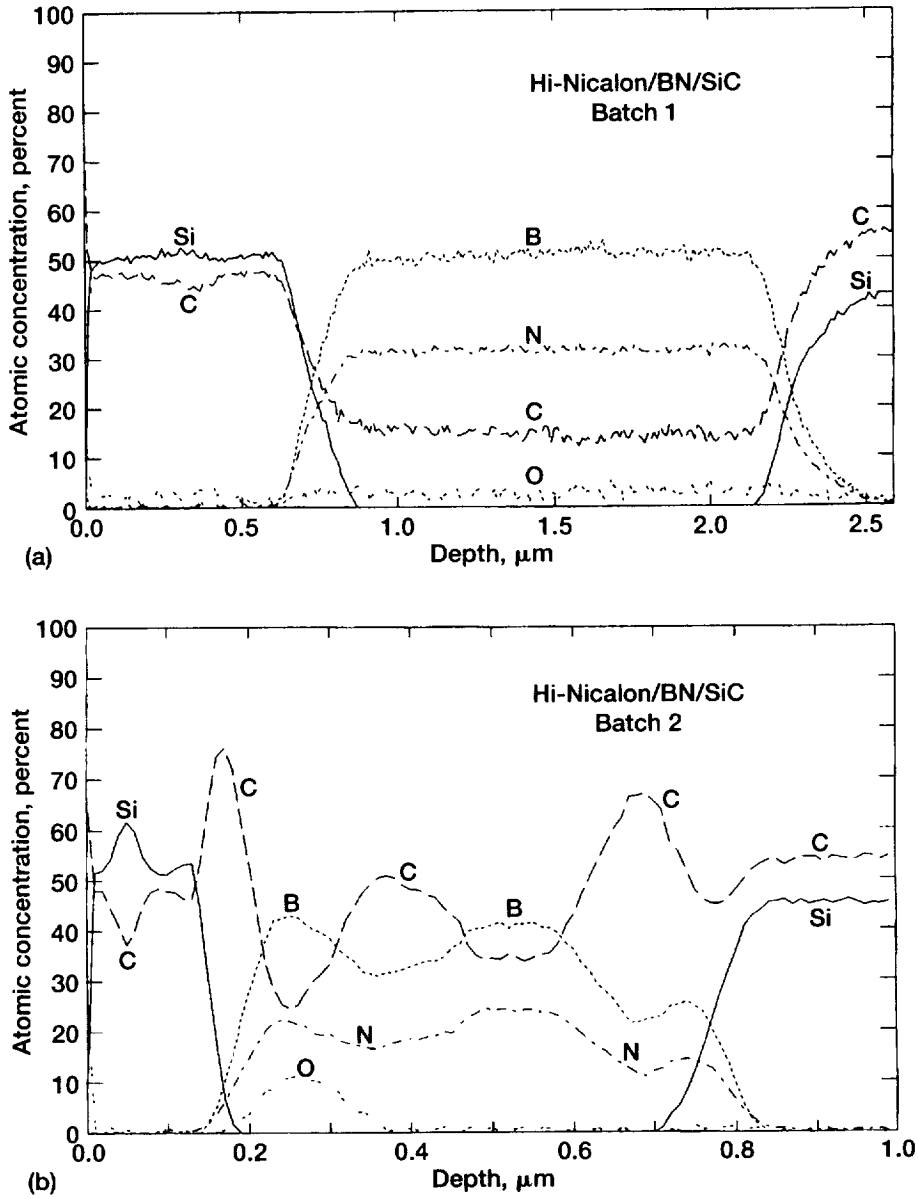

Figure 4: Scanning Auger microprobe elemental depth profiles for Hi-Nicalon fibers having a dual BN/SiC surface coating deposited by CVD: (a) coating batch # 1, and (b) coating batch # 2.

damage during processing. To substantiate this, tensile strengths of the fibers extracted from the composites by leaching away the matrix in HF acid were measured [12]. The BN/SiC coated fibers extracted from the FRC gave a tensile strength of 2.38 ± 0.4 GPa whereas the uncoated fibers fragmented into small pieces during extraction. In contrast, as-received Hi-Nicalon fibers after a similar treatment with HF acid showed no strength degradation. These results confirm that the uncoated Hi-Nicalon fibers have suffered severe mechanical damage during composite processing. In comparison, unidirectional Hi-Nicalon (uncoated) fiber-reinforced lithium aluminosilicate (LAS) glass-ceramic composites containing 50 volume per cent fibers and

processed at 1360°C for 40 min. exhibited [13] room temperature three-point flexural strength of 1158 MPa. The fibers extracted from the composite by dissolving away the LAS matrix in HF acid showed only 20-25% reduction in tensile strength [13]. Similar strength loss has also been observed for Ceramic Grade Nicalon fibers extracted from LAS glass-ceramic matrix composites [14]. The large difference observed in the strengths of the LAS and celsian matrix composites reinforced with uncoated Hi-Nicalon fibers is due to the differences in the processing of the two composites. The LAS composites are hot pressed for a short time just above the melting point of the matrix making use of viscous flow of glass for densification. In contrast, the celsian composites in the current study are hot pressed at a much lower temperature than the matrix melting point.

Large differences in the values of σ_{mc} (Table 1) and $\tau_{friction}$ (Table 2) for the CMCs reinforced with fibers coated with BN/SiC in two different batches was very surprising because both composites were processed under the same conditions. The only difference was that the duplex BN/SiC coating on the fibers used for fabrication of the two composites were deposited in two different batches, but by the same vendor. The difference in fiber volume fraction in the two composites will have some effect on the residual thermal stresses, but not enough to account for the large difference seen in the σ_{mc} values. A high value of $\tau_{friction}$ would result in high σ_{mc} according to the ACK model [15]. By using a simple energy balance approach, in determining the stress necessary to propagate cracks in brittle solids, the following equation has been derived [15, 16] for the matrix cracking stress, σ_{mc}, in a composite consisting of a low failure strain matrix reinforced with high failure strain continuous fibers:

$$\sigma_{mc} = [(12\,\tau_{friction}\,\Gamma_m\,V_f^2\,E_f\,E_c^2)/\{r_f(1-V_f)E_m^2\}]^{1/3} \qquad (3)$$

where Γ_m is the matrix fracture surface energy, V_f is the fiber volume fraction, E_c, E_m, and E_f are the elastic moduli of the composite, matrix and fiber, respectively, and other terms have the same meaning as above. It is apparent from this equation that the first matrix cracking stress can be enhanced by increasing fiber-matrix interfacial sliding stress, by using fibers of smaller radius, and by increasing the volume fraction of fibers. It might also be increased by using low modulus matrix and high modulus fibers. The matrix microcracking may also be suppressed by placing the matrix in compression through choosing $\alpha_f > \alpha_m$, although for isotropic fibers this will result in contraction of the fibers away from the matrix and a potential decrease in fiber-matrix shear strength. It is important to optimize the fiber-matrix bond strength, as too strong a bond will result in a brittle composite with low toughness. By using values of various parameters, as given above, the ratio of $(\sigma_{mc})_{batch\ 1}$ and $(\sigma_{mc})_{batch\ 2}$ for the two coated fiber reinforced composites was calculated from equation (3) to be 3.29. This is in very good agreement with a value of 3.57 for the ratio of measured σ_{mc} values.

The question still remains as to why the $\tau_{friction}$ of composite with batch 1 coated fibers is much higher than the batch 2 coated fiber composite. Microscopic examination (Fig. 5) of the fiber pushed-in samples indicated the presence of wear debris at the coated fiber reinforced composite interface whereas the uncoated fiber reinforced composite exhibited a clean interface. The wear debris consists of thick sections of coating in batch 1 composite but only of thin sublayers of coating in batch 2 composite. The debonding occurs primarily between the innermost coating and the fiber for the composites reinforced with coated fibers. Therefore,

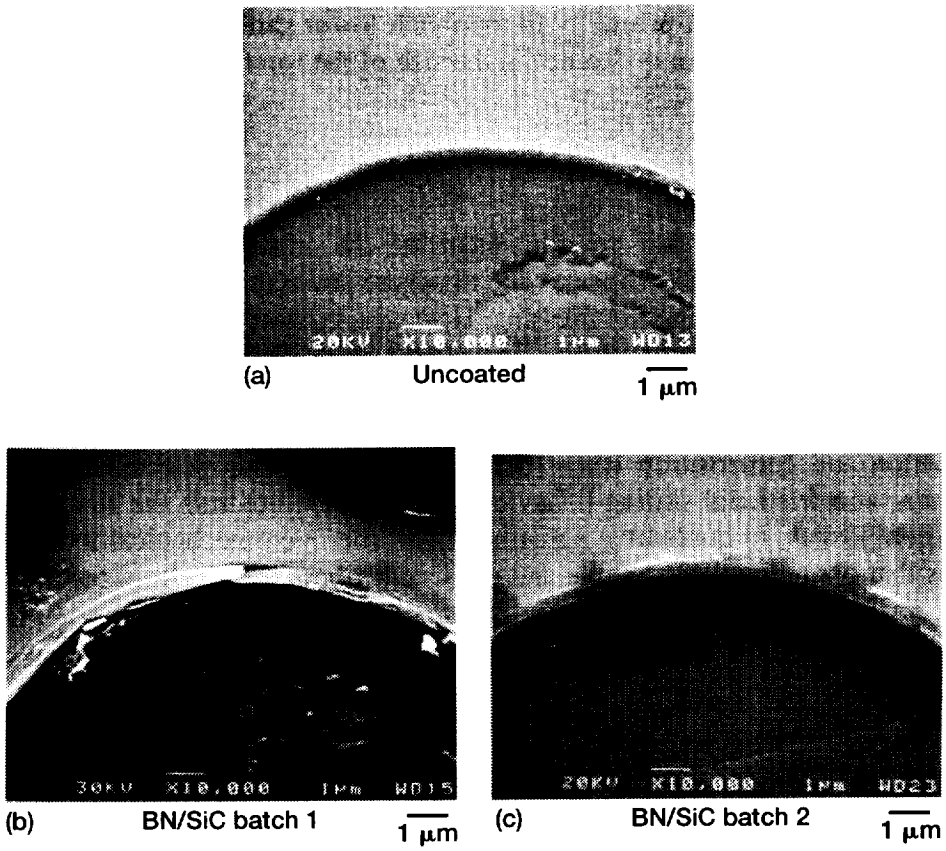

Figure 5: SEM micrographs showing interface failure during fiber push-in for celsian matrix composites reinforced with Hi-Nicalon fibers (a) uncoated, (b) BN/SiC batch 1, and (c) BN/SiC batch 2 coatings

according to the Auger results (Fig. 4), interfacial sliding occurs at the fiber(SiC)/BN interface for batch 1 composite and at the fiber(SiC)/carbon interface for batch 2 composite. Comparison of σ_d and $\tau_{friction}$ values for these two composites indicates that the interface is more strongly bonded and the frictional sliding forces are higher between fiber/BN in batch 1 composite than the fiber/carbon interface in batch 2 composite. Therefore, the additional C layers in the batch 2 coating have a very strong reducing effect on friction and wear during fiber sliding. Brennan et al [17] obtained a value of 9.9 ± 3.5 MPa for $\tau_{friction}$ for the Nicalon/LAS composites, from a similar fiber push-in method using the analysis of Marshall and Oliver [10]. This is consistent with the observation of in situ formation of a thin carbon layer at the fiber/matrix interface during processing of this composite. $\tau_{friction}$ values of 139 ± 95 and 124 ± 71 MPa have been reported [17, 18] for Nicalon/BN/SiC/BMAS and Nicalon/BN/SiC/LAS glass-ceramic matrix composites from the fiber push-in technique as used in the current study. The BN/SiC fiber coatings in these composites were also applied by 3M and the scanning Auger microprobe analysis indicated these coatings to be similar to batch 1 coatings of the present work. Several factors such as residual thermal clamping stresses, fiber roughness, and modulus and thickness of the fiber coating which can control the former factors contribute to the value of $\tau_{friction}$. However, values of $\tau_{friction}$ differing by a factor of about 12 in the two coated fiber composites of the

present study may be attributed primarily to the much lower sliding friction between C/SiC vs. BN/SiC interfaces and also to the difference in the size of the wear debris at the interface.

CONCLUSIONS

It may be concluded that reinforcement of the monoclinic celsian with uncoated Hi-Nicalon fibers yields a weak composite due to severe strength degradation of the fibers from mechanical surface damage during hot pressing. However, reinforcement with the BN-SiC coated Hi-Nicalon fibers results in strong, tough, and almost fully dense composites. While both the uncoated and BN-SiC coated fibers provide a weak interface, the BN layer is needed to protect the fibers from mechanical damage. The fiber coating plays an important role in controlling the interface location and composition where debonding occurs. This determines the fiber/matrix interfacial shear and frictional sliding behavior which in turn controls the first matrix cracking stress of the composites, in qualitative agreement with the micromechanical models. This study also indicates that obtaining reproducible and consistent fiber coatings from commercial sources is a problem.

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13. ABSTRACT ***(Maximum 200 words)***

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14. SUBJECT TERMS

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