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## THE EVOLUTION OF INTERFACIAL SLIDING STRESSES DURING CYCLIC PUSH-IN TESTING OF C- AND BN-COATED HI-NICALON FIBER-REINFORCED CMCs

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### ABSTRACT

Interfacial debond cracks and fiber/matrix sliding stresses in ceramic matrix composites (CMCs) can evolve under cyclic fatigue conditions as well as with changes in the environment, strongly affecting the crack growth behavior, and therefore, the useful service lifetime of the composite. In this study, room temperature cyclic fiber push-in testing was applied to monitor the evolution of frictional sliding stresses and fiber sliding distances with continued cycling in both C- and BN-coated Hi-Nicalon SiC fiber-reinforced CMCs. A SiC matrix composite reinforced with C-coated Hi-Nicalon fibers as well as barium strontium aluminosilicate (BSAS) matrix composites reinforced with BN-coated (four different deposition processes compared) Hi-Nicalon fibers were examined. For failure at a C interface, test results indicated progressive increases in fiber sliding distances during cycling in room air but not in nitrogen. These results suggest the presence of moisture will promote crack growth when interfacial failure occurs at a C interface. While short-term testing environmental effects were not apparent for failure at the BN interfaces, long-term exposure of partially debonded BN-coated fibers to humid air resulted in large increases in fiber sliding distances and decreases in interfacial sliding stresses for all the BN coatings, presumably due to moisture attack. A wide variation was observed in debond and frictional sliding stresses among the different BN coatings.

### INTRODUCTION

Crack-growth behavior in CMCs is strongly affected by changes in crack-closure stresses. The relaxation of closure stresses exerted by crack-bridging fibers will promote crack growth.<sup>1</sup> Therefore, decreases in fiber/matrix sliding stresses (and accompanying interfacial debond crack growth) in CMCs under cyclic fatigue conditions and exposure to different environments will control long-term composite performance and service lifetimes. Previous work<sup>2</sup> has demonstrated the capability of cyclic fiber push-in testing to monitor the early stages of interface degradation under cyclic loading conditions for large-diameter SCS-6 SiC fiber reinforced CMCs. The measured response to applying load cycling to a single fiber reveals the susceptibility of the fiber/matrix interface to degrade under cyclic loading conditions. The object of the present study is to extend this approach to the evolution of frictional sliding behavior of the smaller-diameter Hi-Nicalon SiC fibers, focusing on C and BN fiber coatings, as these coatings have shown the most promise for achieving strong and tough SiC fiber-reinforced CMCs. Because earlier work<sup>2,3</sup> showed that interfacial sliding at the C-rich coating in SCS-6 SiC fiber-reinforced reaction bonded silicon nitride (RBSN) was highly sensitive to the presence of moisture, an important element of this study was to investigate whether sliding at the C and BN interfaces in the Hi-Nicalon fiber-reinforced CMCs was also sensitive to the presence of moisture at room temperature. In particular, four different BN coatings (different processing conditions), expected to have varying resistances to moisture attack, were evaluated.

## EXPERIMENTAL PROCEDURE

### Materials

Testing was performed on both C- and BN-coated Hi-Nicalon fiber-reinforced CMCs. To investigate interfacial sliding with a C fiber coating, a 2-D plain weave Hi-Nicalon (with 1  $\mu\text{m}$  thick C coating) reinforced CVI SiC matrix composite (DuPont Lanxide) was tested. To investigate interfacial sliding with BN fiber coatings, five unidirectional Hi-Nicalon fiber-reinforced barium strontium aluminosilicate (BSAS) matrix composites were tested. One composite incorporated uncoated fibers, while the other four composites incorporated four differently processed CVD BN coatings: (1) 1050°C (deposition temperature) BN-SiC duplex coating (3M); (2) 1400°C pyrolytic BN (p-BN) (Advanced Ceramics); (3) 1800°C p-BN (Advanced Ceramics); and (4) 1400°C 20wt% Si-doped BN (Advanced Ceramics). Nominal coating thicknesses were close to 0.5  $\mu\text{m}$  for all the BN coatings, but showed wide variations throughout the specimens. Higher temperature deposition and Si doping were expected to increase the resistance to moisture attack.<sup>4,5</sup> Test specimens were sliced perpendicular to the axis of the fibers to be tested with a diamond saw and mechanically polished down to a 0.1  $\mu\text{m}$  finish on both faces. Final specimen thickness was typically about 3 mm.

### Testing Procedure

Cyclic fiber push-in tests were performed using a desktop fiber push-out apparatus previously described,<sup>6</sup> but with the addition of symmetrically placed capacitance gauges for displacement measurements and the incorporation of an inflatable polyethylene enclosure for tests performed in nitrogen. The Hi-Nicalon SiC fibers were pushed in using a 70°-included-angle conical diamond indenter with a 10  $\mu\text{m}$  diameter flat on the bottom. To prevent the sides of the conical indenter from impacting the matrix, push-in distances were restricted to just a couple of microns. Unless otherwise noted, each test consisted of five cycles of loading and unloading between a selected maximum load and a minimum load of 0.01 N at room temperature in either room air (20-50% relative humidity) or nitrogen.

Because the cyclic push-in behavior for the BN-coated fibers did not show apparent immediate effects of the test environment, the effect of long-term exposure to a humid environment was investigated for these materials. After initial room air testing of selected fibers for five cycles, specimens were stored for eight weeks in a room-temperature 100% RH environment, and testing of the same previously tested fibers (already partially debonded) was resumed in room air for an additional five cycles under the same loading conditions.

The data were analyzed by first subtracting the appropriate load-train compliance correction from the measured displacements. Then fiber sliding distances and frictional sliding stresses could be determined for each cycle. Both a maximum displacement,  $u_{\text{max}}$ , and a sliding distance,  $\Delta u = u_{\text{max}} - u_{\text{min}}$ , could be determined for each cycle. An estimate of frictional sliding stress,  $\tau$ , could also be determined using the constant  $\tau$  model of Marshall and Oliver<sup>7</sup> to fit each reloading curve to the relationship:

$$u = u_0 + \frac{F^2}{8\pi^2 r^3 E_f \tau} \quad (1)$$

where  $u$  is the fiber end displacement,  $u_0$  is the residual fiber end displacement after the previous unloading,  $F$  is the applied load,  $r$  is the fiber radius, and  $E_f$  is the fiber modulus. While neglecting Poisson expansion of the fibers leads to an overestimation of  $\tau$  values, the relative changes in  $\tau$  with load cycling could be followed using Equation 1. In addition, a debond initiation stress,  $\sigma_d$ , 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  $\sigma_d = F_d/\pi r^2$ .

## RESULTS

### Carbon-Coated Hi-Nicalon Fibers in SiC Matrix

Averaging values determined from initial push-in load cycles for the Hi-Nicalon/C/CVI SiC in room air gives values of  $\sigma_d = 1.54 \pm 0.39$  GPa and  $\tau = 72.1 \pm 33.5$  MPa (first reloading cycle). The most striking features of these tests are the consistently increasing fiber sliding distances associated with continued cycling in room air. Fig. 1 shows typical cyclic push-in behavior in room temperature air for 20 loading cycles up to 1.4 N. There was a clear progressive increase in both  $u_{max}$  and  $\Delta u$  with continued cycling. Remarkably, when cyclic testing in room air was interrupted after five cycles and later resumed in nitrogen (typical test shown in Fig. 2), the progressive increase in fiber sliding distances halted abruptly. In addition, cyclic push-in tests initiated in nitrogen (typical test shown again in Fig. 2) showed consistently shorter fiber sliding distances that did not exhibit the progressive increases in sliding distance observed in room air. In almost all cases, interfacial failure occurred between the fiber and the C coating.

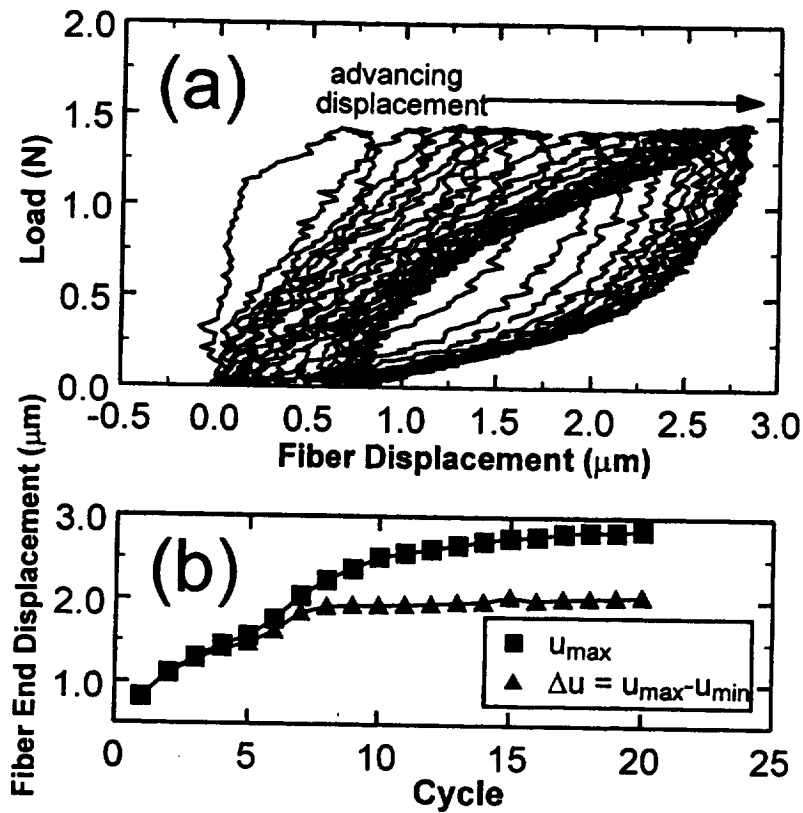
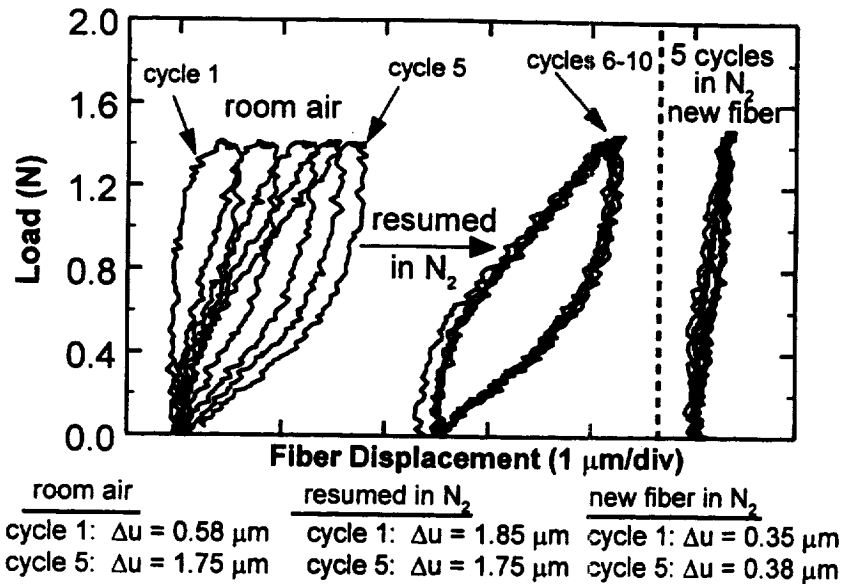


Figure 1. (a) Typical cyclic fiber push-in test for a 3.56-mm-thick Hi-Nicalon/C/CVI SiC specimen at room temperature in room air. Twenty cycles up to 1.4 N. (b) Plot of  $u_{max}$  and  $\Delta u$  vs. cycles.



**Figure 2. Effect of room temperature test environment on cyclic push-in tests to 1.4 N for a 3.56-mm-thick Hi-Nicalon/C/CVI SiC specimen. Left: Typical cyclic fiber push-in test in room air interrupted after five cycles and resumed on same fiber in nitrogen for five additional cycles. Right: Typical test of previously untested fiber performed in nitrogen for five cycles.**

#### Boron-Nitride-Coated Hi-Nicalon Fibers in BSAS Matrix

None of the BN-coated fibers showed the large progressive increases in sliding distances in room air observed for the C-coated fibers. In addition, there were no obvious differences in tests performed in room air or nitrogen for the 1050°C BN-SiC coating. These results indicated an absence of a significant immediate effect of exposure to moisture. Therefore, cyclic push-in tests interrupted for a long-term (eight-week) exposure to room temperature 100% RH air were conducted.

The initial room air tests (six tests analyzed for each composite) showed a wide variation in  $\sigma_d$  and  $\tau$  for the different coatings (Table I). Note that, for most of the fibers,  $\sigma_d$  was too high (fiber failure occurred first) for the Si-doped BN coating to achieve fiber debonding; this absence of fiber sliding prevented investigation of the evolution of sliding stresses and distances for this composite.

Resumption of interrupted cyclic push-in tests after eight weeks storage in room temperature 100% RH air showed no significant changes in cycling behavior for the uncoated fibers. However, resumption of tests for the BN-coated fibers showed large changes in cycling behavior, depending on the specific BN coating. As an illustration, Fig. 3 shows that the 1050°C BN-SiC coated fibers show dramatically greater fiber sliding distances and lower sliding stresses (evidenced by wider hysteresis) upon resuming cycling after long-term humid air exposure. Fig. 4 summarizes the effects of the 100% RH air exposure on  $\tau$  for the three BN coatings as well as the uncoated fibers. (Six interrupted tests were analyzed for each composite.) Both the 1050°C BN-SiC coating and the 1800°C p-BN coating show large decreases from relatively high  $\tau$  values after the 100% RH exposure. The 1400°C p-BN coating also shows a significant decrease in  $\tau$ , but

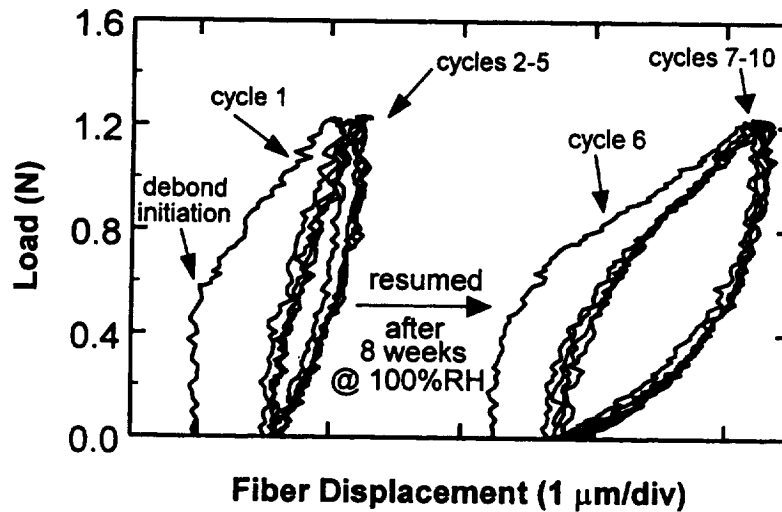
from much lower initial values. Note that modest decreases in  $\tau$  were observed during initial cycling, most notably for the 1800°C p-BN coating.

All four BN coatings exhibited interfacial failure primarily at the fiber/coating interface. The 1800°C p-BN consistently showed the greatest amount of coating debris (observed by SEM inspection) due to crumbling of the inner portion of the coating during fiber sliding.

**Table I. Interfacial Properties for Various BN Fiber Coatings in Hi-Nicalon/BSAS Composites**

Coating	$\sigma_d$ (GPa)	$\tau^a$ (MPa)
no coating	1.01 ± 0.42	25.3 ± 10.7
1050°C BN-SiC	1.82 ± 0.64	108.0 ± 39.5
1400°C p-BN	1.17 ± 0.34	29.9 ± 12.8
1800°C p-BN	2.63 ± 0.86	145.6 ± 78.2
1400°C 20wt% Si-doped BN	> 4	--

<sup>a</sup>From 1<sup>st</sup> reloading cycle.



**Figure 3. Changes in room temperature cyclic fiber push-in test of a 3.22-mm-thick Hi-Nicalon/1050°C BN-SiC/BSAS specimen up to 1.2 N when testing after five cycles in room air is interrupted and then resumed in room air on same fiber after eight weeks exposure to 100% RH air.**

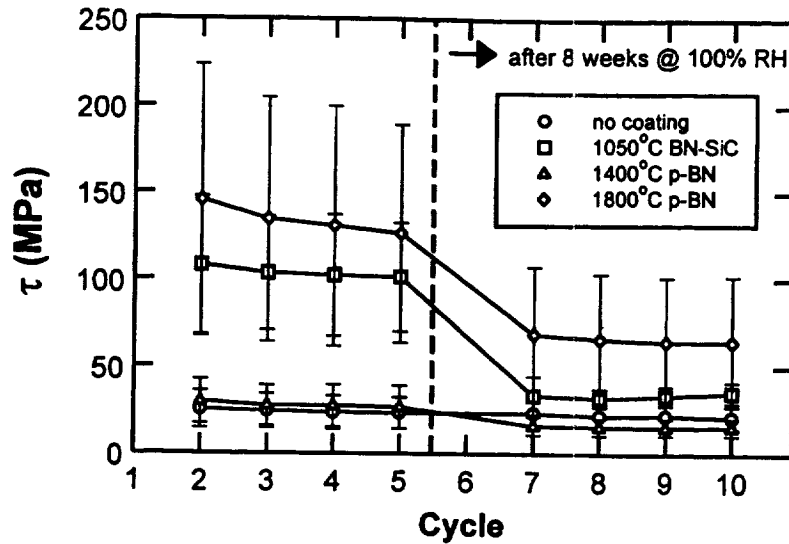


Figure 4. Summary of variation of fiber sliding stresses during cyclic fiber push-in tests for Hi-Nicalon/BSAS specimens for three different BN fiber coatings as well as uncoated fibers. Sliding stresses are shown for initial five cycles in room and for additional five cycles after eight-week exposure to 100% RH air. Error bars indicate  $\pm 1$  standard deviation.

#### DISCUSSION

The fiber sliding behavior was distinctly different for room air vs. nitrogen test environments for the Hi-Nicalon/C/CVI SiC composite material (Figs. 1-2). In room air, the cyclic fiber push-in tests show progressively increasing fiber sliding distances with continued cycling; these progressive increases in sliding distance cease immediately upon continuation of interrupted tests in a nitrogen environment and are not observed for tests initiated in nitrogen. These environmental effects are very similar to those observed earlier for the SCS-6 SiC fiber-reinforced RBSN.<sup>2</sup> The very similar behavior for these two composites, both with carbon (or predominately carbon) coatings, is consistent with the known dependence of carbon materials on adsorbed water (and secondarily oxygen) to achieve low sliding friction and wear during sliding.<sup>3</sup> Graphite and other  $sp^2$  carbonaceous materials are not good intrinsic lubricants, but depend on molecular species such as water adsorbed on the surface to provide good lubrication. The progressively increasing fiber sliding distances during cyclic push-in testing in room air can be explained by the increased exposure of the debonded interface to the ambient atmosphere, providing additional water/oxygen adsorption. These results suggest that the presence of moisture will induce relaxation of crack closure stresses (even at room temperature), and therefore, promote crack growth in composites where cracks are bridged by C-coated fibers.

The evolution of fiber sliding behavior for BN-coated fibers was investigated in Hi-Nicalon/BSAS composites. Four BN coatings processed by different routes as well as uncoated fibers were selected for comparison based on the previous demonstration that increases in deposition temperature and Si-doping dramatically increase the resistance of these coatings to attack by moisture.<sup>4</sup> The cyclic fiber push-in tests presented here show that there is also a large variation in interfacial sliding behavior (Table I) between these coatings. The 1400°C p-BN coated and uncoated fibers show similarly low  $\tau$  and  $\sigma_a$ , whereas the other BN coatings show significantly higher  $\tau$  and  $\sigma_a$  values. (It was shown earlier that the beneficial effect of the BN coating over uncoated fibers was to prevent fiber strength degradation during processing rather

than providing a weak interface.<sup>9</sup>) The Si-doping appears to have the deleterious effect of such strong fiber/matrix bonding that fiber sliding could not be investigated in that composite. The high  $\tau$  observed for the 1800°C p-BN coating is most likely due to the crumbling of the inner portion of the coating during fiber sliding, producing much more debris at the sliding interface than the other coatings. This more severe coating wear is probably responsible for the greater decreases in  $\tau$  observed during initial push-in cycling of the 1800°C p-BN coated fibers. This wear will lead to relaxation of crack closure stresses upon cyclic loading of fiber-bridged cracks, and thus promote crack growth. It should be noted that a SiC overcoat on the p-BN and Si-doped BN coatings may improve the performance of those coatings by acting as a diffusion barrier.<sup>10</sup>

Although short-term exposure of the debonded interface to room air in the BN-coated Hi-Nicalon/BSAS composites did not seem to have a significant effect on interfacial frictional sliding stresses, long-term exposure of the debonded interfaces to humid air resulted in much lower frictional sliding stresses and longer fiber sliding distances (Figs. 3,4). Because the effect of exposure to humid air is not immediate, as with the C coatings, it is less likely that the changes in sliding behavior are also due to simple adsorption of water molecules on the exposed surfaces. On the other hand, while chemical attack by moisture may play a significant role for changes observed after humid air exposure for the 1050°C BN-SiC coating, the same explanation is unlikely for the other coatings, e.g., the 1800°C p-BN coating should be immune to attack by moisture at room temperature, but still shows a large decrease in  $\tau$  after exposure to humid air. Studies of friction and wear at hexagonal BN surfaces<sup>11</sup> (which are resistant to chemical attack by water at room temperature) have suggested that the lowering of friction at sliding BN surfaces in humid air environments is due to the condensation of liquid water at those surfaces. Moisture condensation would reduce friction by changing the morphology of the wear debris (by lowering the surface tension of the debris particles) to a lower energy configuration. Such a mechanism could explain both why the effect of exposure to room air is not immediate (insufficient time for liquid condensation) and why the effect of moisture does not seem to be correlated to the susceptibility of the coating to chemical attack by moisture.

## CONCLUSIONS

The early stages of the evolution of fiber sliding stresses and sliding distances under cyclic loading conditions for both C- and BN-coated Hi-Nicalon reinforced CMCs were evaluated by cyclic fiber push-in testing. Both wear at sliding surfaces and exposure of debonded interfaces to moisture were observed to increase fiber sliding distances and decrease fiber sliding stresses with the predicted consequence of decreasing closure stresses exerted by crack-bridging fibers during cyclic loading (promoting crack growth). For the Hi-Nicalon/C/CVI SiC composite (where interfacial sliding primarily occurs between the fiber and the C coating), large progressive increases in fiber sliding distances occurred immediately upon cyclic loading in room air. This behavior was attributed to the exposure of the debonded interface to moisture, since the adsorption of water molecules on carbon surfaces is known to lower friction and wear at sliding carbon surfaces. For the Hi-Nicalon/BN/BSAS composites (where interfacial failure primarily occurs between the fiber and the coating), large increases in fiber sliding distances and decreases in fiber sliding stresses were observed during cyclic loading after long-term exposure of the debonded interface to humid air. While this behavior may be due to chemical attack of the exposed coating surface by moisture for lower deposition temperature coatings, this explanation is unlikely for higher deposition temperature coatings, where the effects of humid air exposure are postulated to be due to the condensation of liquid water at the debonded interface, changing the surface tension of the coating wear debris trapped at the fiber/matrix interface.

It was also shown that changing the BN coating processing route to improve resistance to attack by moisture can unfortunately have detrimental consequences for fiber bonding and sliding behavior. This was observed for the Si-doped BN coating, where the Si addition improves

resistance to attack by moisture but results in a very strong fiber/matrix bond that would cause brittle composite behavior. In addition, although the higher temperature deposition for the 1800°C p-BN coating improves resistance to attack by moisture, it also results in a coating that crumbles during sliding more readily than lower temperature deposition coatings. This greater coating wear will have the unfortunate consequence of more rapidly decreasing crack closure stresses, promoting crack growth.

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