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# INTERFACIAL PUSH-OUT MEASUREMENTS OF FULLY-BONDED SIC/SIC COMPOSITES*

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## ABSTRACT

The direct measurement of interfacial bond strength and frictional resistance to sliding in a fully-bonded SiC/SiC composite is measured. It is shown that a fiber push-out technique can be utilized for small diameter fibers and very thin composite sections. Results are presented for a 22 micron thick section for which 37 out of 44 Nicalon fibers tested were pushed-out within the maximum nanoindentor load of 120 mN. Fiber interfacial yielding, push-out and sliding resistance were measured for each fiber. The distribution of interfacial strengths is treated as being in Weibull form.


## INTRODUCTION

Ceramic composite strength and fracture toughness are very sensitive to the type and magnitude of bonding at the fiber/matrix interface[1,2]. This interface has the dual roles of being strong enough to transfer load between matrix and fiber to take advantage of the high fiber strength and being compliant enough to inhibit crack propagation, thus adding toughness to an otherwise brittle composite.

Due to the fundamental importance of the composite interface and the newness of direct interfacial measurement techniques, there has been growing interest in this area. There have been several recent papers in which investigators have employed microindentation testing techniques to measure interfacial strength[3,4,5,6] . The bulk of the work for ceramic fiber composites has been on SiC/glass systems which, due to the mismatch in thermal expansions and resulting debonding during process cooling, has a pre-cracked interface yielding only frictional resistance to fiber sliding. Marshall[7,8] was the first to employ a microindentation technique to measure the interfacial frictional strength by using a Vickers pyramid indentor to apply a load and push-down fibers oriented normal to a polished SiC(Nicalon)/lithium-alumino-silicate (LAS) glass surface. The interfacial friction results were shown to compare reasonably well with data taken by matrix crack-spacing measurements.

It is clear that for a fully-bonded interface there will be at least two competing resistances to fiber sliding. First, there will be a minimum force required to initiate a crack in the interfacial chemical bond which may or may not then have the required energy to propagate along the length of the fiber. Second, there will be a frictional component to the resistance which is caused by the crack surfaces passing over each other as the fiber slides.

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Marshall and Oliver[9] were the first to use a microindentation technique to attempt to separate the effects of bonding and frictional sliding in weakly bonded composites. In their experiments, they heat treated as-processed, unbonded SiC(Nicalon)/lithium-alumino-silicate (LAS) composites in air and argon between 900 C and 1250 C, forming a carbon chemical bond at the interface. A Nanoindentor™ was used to apply loads to fibers of thick sections of the as-processed and heat-treated composite. Using the Nanoindentor, an accurate force-vs-displacement curve was generated and from the loading curve and the unload-reload hysteresis curves an estimate of the debonding and frictional resistance could be obtained.

Later researchers[10] employed a similar methodology with composite sections thin enough such that a small percentage of the fibers were "pushed-out" of the matrix, thereby simplifying analysis of the interfacial strengths. Bright et. al.[5] applied the push-out technique to fully bonded SiC(AVCO)/reaction-bonded-silicon-nitride composites and measured both bonding and frictional interfacial strength for sections as thin as 0.5 mm,. or a fiber diameter-to-thickness ratio of approximately 1/35. For fiber push-out experiments it is generally best to have as large a fiber diameter to thickness ratio as possible while not altering the fiber/matrix interface in the thinning process. By doing this the calculation of the interfacial strength is simplified.

The purpose of this paper is to present interfacial bond and frictional strengths obtained from push-out measurements for a very thin section of SiC(Nicalon)/SiC(CVD) composite material. All results reported are for a single composite section of 22 microns, which corresponds to a Nicalon fiber diameter to specimen thickness ratio approaching unity. Whereas results represent a single composite section, they are typical of other sections tested.

## EXPERIMENTAL

The composite specimen is a layered 30/60/90 weave of Nicalon cloth infiltrated with SiC using the ORNL Forced Chemical Vapor Infiltration process.[11] The fiber/matrix interface contains a 1 micron layer of graphitic carbon which was deposited onto the fibers from methane gas prior to SiC infiltration.

The composite was sliced into 250 micron sections with a low speed diamond saw and mechanically thinned and polished with 6, 1 and 1/2 micron diamond paste to a final thickness of 22 microns. The specimen was then mounted onto a polished aluminum holder such that the center of the specimen was positioned over a groove of 200 microns in width. The composite section was then held rigidly to the mount and fixed to the surface with Crystal Bond™ mounting wax. Care was taken during the thinning and mounting procedure to prevent specimen bending in order to avoid pre-cracking the interface.

A load-controlled Nanoindentor microindentation hardness tester was used to apply a force to fibers oriented normal to the composite surface. A good discussion of the Nanoindentor can be found elsewhere.[12] The Nanoindentor has an applied force and depth measurement sensitivity of 0.3 $\mu$N and 0.16 nm, respectively. The positioning table has sub-micron accuracy to insure loading near the center of fibers which have diameters ranging from 12 to 25 microns.

A constant loading rate of 900 μN/sec was used with a maximum loading capability of 0.12 N which corresponds to approximately 700 MPa for the average fiber. A Berkovitch pyramidal diamond indentor tip with an aspect ratio of 1:7 was used .

Once the fibers were loaded to their debond load and pushed through the matrix (or to the maximum load of the machine while remaining intact), the specimen was then demounted and turned over in the holder such that the debonded fibers again lie over the 200 micron groove. The position of the fibers, which were protruding from the matrix, were found and the load reapplied with the Nanoindentor so that the fibers passed through their original position and out the opposite side of the composite section. The individual fiber diameters were measured by utilizing the Nanoindentor optics and positioning.

## RESULTS

A total of the 44 fibers were tested of which 37 fibers debonded within the machine's maximum load of 0.12 Newtons. All of the debonded fibers that were tested in reverse loading showed frictional sliding. Figure 1 shows the load displacement curve for a fiber that did not fail, which provides a measure of the specimen compliance. This can be crucial since if there is appreciable specimen bending present, there may be a fiber pinching effect which would affect the results. It can be seen from Figure 1 that the maximum diamond indentor depth corresponding to maximum machine load is 1.5 microns (marked A) of which 0.4 microns (marked B) is plastic deformation of the fiber.

Previous researchers have noted the problems of testing very thin composite sections. [5,8,13] The interfacial bonding and sliding friction strengths can be affected by precracking of the interface during thinning and by altering the interfacial stress state as the result of specimen compliance during testing. However, by careful mechanical thinning and polishing it is possible to overcome these problems with SiC composites. It is easily shown that the 1.1 micron displacement (in Figure 1) of a large section suspended over a 200 micron groove will have a negligible effect on the results. Also, due to the fact that the matrix SiC becomes transparent for thicknesses less than (approximately) 50 microns, the absence of large matrix cracks were verified using a transmission optical microscope.

It is also seen from Figure 1 that the load-displacement (and hysterisis) curve has the parabolic shape characteristic of indentation hardness measurements of monolithic materials, which indicates an absence of fiber interfacial failure. When fiber interfacial cracking occurs, followed by a fiber debonding, there will be a noticeable departure from the parabolic load curve. The bond failure can be either a partial or total debonding depending on whether the crack front has the energy to propagate the length of the fiber. The partial debonding is measured here as 2 % departure from the parabolic load curve whereas the total fiber debond is measured as the stress at which the fiber is pushed-out of the matrix. However, it was observed that a fiber could exibit one, both or neither of the failure mechanisms. Some fibers showed no parabolic deviation prior to the point of complete push-out, which would correspond to a very rapidly propagating crack front, whereas other fibers showed a deviation at a relatively low load and no push-out within the machine's maximum load, which is indicative of an arrested crack front.

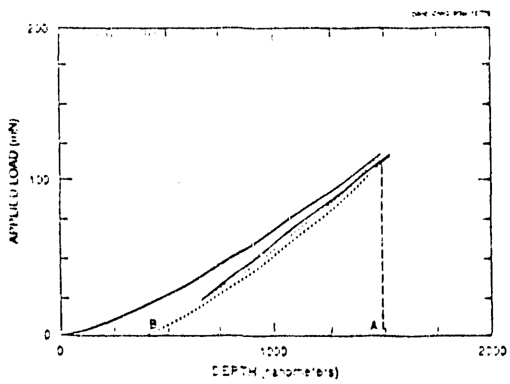


Figure 1. Loading curve for fiber remaining intact.

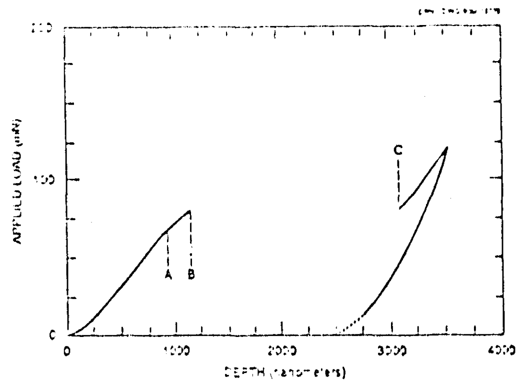


Figure 2. Loading curve for fiber debonding and push-out.

Figure 2 shows an example of a bonding failure exhibiting distinct interfacial yielding followed by total debonding. As the fiber was loaded there was a partial yield of the interface at approximately 65 mN (A) followed by a total debonding and fiber push-out at 80 mN (B). The fiber was pushed-out of the composite section approximately 2.2 microns at which point the indentor contacted the matrix surrounding the fiber. The matrix contact depth is seen in Figure 2 at an indentor depth of 3000 nm. Both partial debonding (A) and the point of fiber push out (B) are noted. The absence of data points between B abd C is evidence of the rapid fiber failure indicative of the total interfacial debonding and push-out. The stress at failure is taken to be the load applied at the point of yielding divided by the interfacial area. This, however, is an oversimplification as the load is actually concentrated at the crack front itself and not spread over the entire interface.

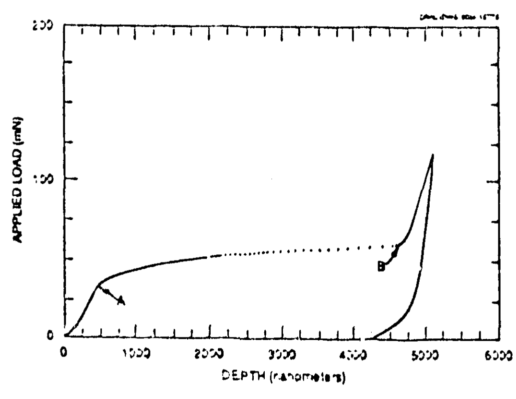


Figure 3. Loading curve for frictional sliding

The failure in the reverse loading situation is shown in Figure 3. The frictional failure is characterized by a graceful departure from the normal loading curve. The frictional stress is obtained by dividing the sliding load by the fiber interfacial area. In this case, sliding occurred at 32.5 mN (A) and was pushed a total length of 4500 nm (B). With total fiber debonding, the applied load required to keep the fiber moving at a constant rate should remain constant or changeslightly with the change in interfacial area as the fiber is pushed-out.

In Figure 3, after the point (A) when the fiber started to slide, the force continues to increase until the diamond comes in contact (B)with the channel walls left behind by the sliding fiber. The load increase occurs because the Nanoindentor was programmed to produce a constant loading rate during the indentation. After the onset of fiber sliding the load continued to increase, accelerating fiber sliding which is demonstrated by the increased spacing between the data points between A and B.

Figure 4 shows a scanning electron micrograph of a pushed-out fiber. The fiber pictured in the bottom portion of the micrograph is the fiber of Figure 1, which did not yield, and has a large pyramid-shaped imprint resulting from plastic deformation of the Nicalon at the Nanoindentor's maximum load. Pictured above this is a debonded fiber which was pushed-out and then reloaded and pushed in the opposite direction a total of 5 microns. Though the micrograph may suggest that a nearest neighbor effect may have been responsible for the adherance of the lower fiber, in the inspection of hundreds of failed fibers such a correlation was not found.

For both fully bonded and unbonded interfaces, researchers have found a great variability in measured interspecimen interfacial strengths.[10] It has been shown elsewhere, however, that a Weibull distribution is more appropriate to

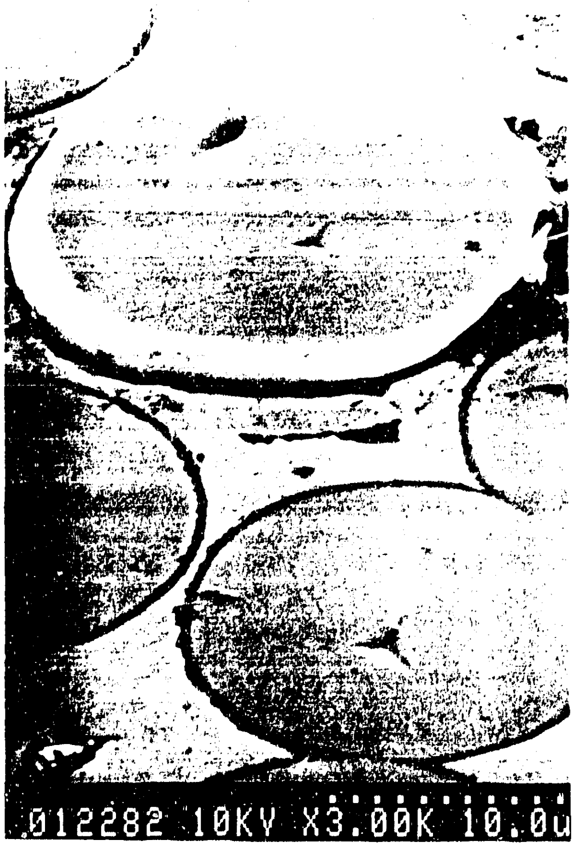


Figure 4. SEM micrograph of bonded and pushed-out fiber.

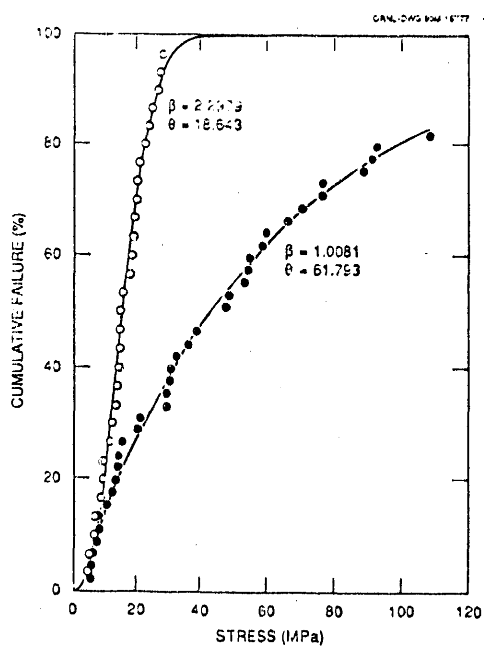


Figure 5. Cumulative Weibull Distribution of data.

represent the interfacial strengths than the lognormal, exponential or gamma functions.[14]

Here, the two parameter form of the Weibull probability distribution function and corresponding cumulative failure function is adopted and given by :

$$P = ( \beta\sigma^{\beta-1} / \theta^{\beta} )\, e^{-(\sigma/\theta)^{\beta}} \qquad F = 1 - e^{-(\sigma/\theta)^{\beta}}$$

where $\beta$ is the Weibull slope (shape parameter), $\theta$ is the size parameter and $\sigma$ is the applied variable stress.

Figure 5 shows the cumulative distribution for both the 2 % debond strength and the frictional resistance to sliding. The Weibull moduli ($\beta$) and shape parameters ($\theta$) are also given in the figure. It is seen that the 2 % yield strength is not only greater that the frictional resistance but that the Weibull modulus is less for the 2 % yield, leading to a broader distribution in strength. The Weibull mean values are calculated to be 60.8 MPa for the 2 % yield and 16.5 MPa for the sliding resistance with corresponding moduli of 1 and 2.4, respectively.. Due to the relatively low values of the Weibull moduli, the probability distribution function for the debonding yield strength and the frictional sliding resistance are rather broad. Because of this there will be noticeable overlapping of the probability functions which would become more pronounced as the average strengths of the two distributions approach each other. When the fibers are initially loaded, therefore, there will be some fraction of fibers (mathematically) which possess a higher frictional resistance to sliding than debond strength. Physically this means that the stress required to initiate and propagate a crack is less than the stress required to slide the fracture surfaces across each other. On the load curve of such fibers, the debonding is not noticeable and gradual frictional-type failure would dominate.

## CONCLUSIONS

1) It has been shown that specimens of SiC(Nicalon)/SiC composites can be thinned to a thickness approaching a fiber diameter (~ 20 μm) and push-out tests can be performed without specimen compliance limitations or matrix cracking.

2) The debond yield strength and fiber push-out strength for these small fibers can be obtained using a Nanoindentor with a high percentage of interfacial debonding. The frictional resistance to sliding can likewise be determined by reapplying the load to the opposite end of the pushed out fiber.

3) The debond yield, debonding push-out and fiber sliding stress can be adequately separated by inspection of the load-displacement curve. The debonding is characterized by an abrupt failure while the fiber sliding is a more gracefull failure.

4) The result of the analysis has shown that the interfacial frictional stress has a higher Weibull modulus than the debond stress, thus a less "brittle" failure.

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