THE MONOTONIC AND FATIGUE BEHAVIOR OF CFCCs – N. Miriyala, P. K. Liaw, and C. J. McHargue (University of Tennessee) and L. L. Snead (Oak Ridge National Laboratory).

OBJECTIVE

To develop a fundamental understanding of the fabric orientation effects on the monotonic and fatigue behavior of Nicalon/Al_2O_3 and Nicalon/SiC composites.

SUMMARY

Flexure tests were performed to study the fabric orientation effects on the monotonic and fatigue behavior of two commercially available continuous fiber reinforced ceramic composites (CFCCs), namely, (i) Nicalon fiber fabric reinforced alumina (Al_2O_3) matrix composite fabricated by a direct molten metal oxidation (DIMOX) process and (ii) Nicalon fiber fabric reinforced silicon carbide (SiC) matrix composite fabricated by an isothermal chemical vapor infiltration (ICVI) process. The fabric orientation effects on the monotonic and fatigue behavior were strong in the Nicalon/Al_2O_3 composite, while they were relatively weak in the Nicalon/SiC composite.

PROGRESS AND STATUS

Introduction

As structural materials, monolithic (unreinforced) ceramics inherently suffer from two important reliability issues; high sensitivity to processing and service generated flaws (low fracture toughness) and the inability to tolerate stress overloads without catastrophic failure. Although whisker reinforcement enhances toughness, it does not remove the possibility of catastrophic failure, a problem that severely restricts the use of whisker-toughened ceramics in aerospace engines. Continuous fiber reinforced ceramic matrix composites (CFCCs), on the other hand, can provide significant toughness increases along with the ability to fail in a non-catastrophic manner similar to a metal [1, 2].

Woven fiber fabric reinforcement of the matrix has been a popular choice of process engineers attempting to develop CFCCs with improved toughness. Although most of the advanced CFCCs developed to date are 2-D reinforced, the effect of fabric orientation on their mechanical behavior has received scarce attention of the material testing community. Hence, it has been decided to investigate the effects of fabric orientation on the monotonic and fatigue performance of two of the advanced CFCCs, namely, (i) Nicalon fiber fabric reinforced alumina (Al_2O_3) matrix composite fabricated by a direct molten metal oxidation (DIMOX) process and (ii) Nicalon fiber fabric reinforced silicon carbide (SiC) matrix composite fabricated by an isothermal chemical vapor infiltration (ICVI) process. Flexural specimens were performed to obtain the stress-strain curves and fatigue stress-life (S-N) curves for both composites. In addition, the progression of damage during cyclic loading was assessed by measuring the "effective modulus" of the composite at regular intervals of the fatigue tests. Fracture surfaces of both composites were examined under a scanning electron microscope to gain an insight into the damage mechanisms operative under static and cyclic loadings.

Experimental Details

Materials

The Nicalon fiber fabric reinforced alumina (Al_2O_3) matrix composite, fabricated by a direct molten metal oxidation (DIMOX) process, was donated by Lanxide Corporation. The as-received material was in the form of plate, approximately $150 \times 150 \times 3.2$ mm. A 12-harness satin woven Nicalon fabric in a 0/90 sequence was used as the preform in this composite. The fabric preform was given a duplex coating of boron nitride (BN) and silicon carbide (SiC) via chemical vapor deposition (CVD). While BN acts as the interlayer between the fiber and the matrix, the overlaying SiC coating protects the fiber and the BN interlayer during matrix infiltration. The BN coating thickness was approximately 0.2 to 0.5 μm, while the SiC coating thickness varied from 3 to 4 μm. The duplex-coated preform was then placed in contact with molten aluminum at 900 to 1100°C in air. The matrix in the composite was formed by the growth of alpha alumina, starting from the alloy/preform interface. The nominal fiber volume percent in the composite was approximately 35.

The Nicalon fiber fabric reinforced silicon carbide (SiC) matrix composite, fabricated by an isothermal chemical vapor infiltration (ICVI) process, was donated by AlliedSignal Corporation. The as-received material was in the form of two plates, approximately 200 × 150 × 2.4 mm, manufactured by DuPont Corporation. A 2-D braided Nicalon fabric was used as the preform, which was first given an interfacial coating of carbon (approximately 0.4 to 0.5 μm) by a CVD process, prior to the infiltration of SiC matrix. The SiC matrix was infiltrated by the decomposition of methyltrichlorosilane, in the presence of hydrogen, at 1100 to 1200°C. The nominal fiber volume percent in the composite was approximately 40.

Flexure testing

Flexure bars were machined from the composite plates using diamond tooling. The ASTM Standard C 1161 configuration requires the specimens to be 50 mm long, 4 mm wide and 3 mm thick. However, in order to objectively study the effects of fabric orientation on the mechanical behavior of the two composites, flexural bars of squared cross-section were fabricated. The flexural bars of the Nicalon/Al_2O_3 composite used in the present investigation were 50 × 3 × 3 mm, while the Nicalon/SiC composite specimens were only 50 × 2 × 2 mm. The four-point bend monotonic and fatigue tests were performed on a MTS servohydraulic system, equipped with a graphite heating element furnace, that can be used to conduct elevated-temperature tests, up to 2000°C in vacuum or argon. To date, all the mechanical tests were performed at room temperature.

The monotonic tests were conducted under displacement control at a cross-head displacement rate of 0.5 mm/min. The midspan deflection of the specimen was monitored using a deflection gage made of SiC. The fatigue tests were conducted under load control. All the tests were conducted using a sinusoidal wave form at a test frequency of 5 Hz. The R-ratio (minimum load/maximum load) was 0.1 in all the cases. At periodic intervals during the fatigue tests, the load and midspan deflection values for two consecutive load cycles were recorded. Two hundred pairs of load and deflection values were recorded each time the data was captured. The fatigue run-out was one million cycles, which corresponds to approximately 56 hours of testing.

During the monotonic and fatigue tests, loads were applied to the specimens either parallel or perpendicular to the fabric plies. Accordingly, the specimen configurations were referred to as edge-on and transverse, depending on whether the load was parallel or perpendicular to the fabric plies, respectively (Figure 1).

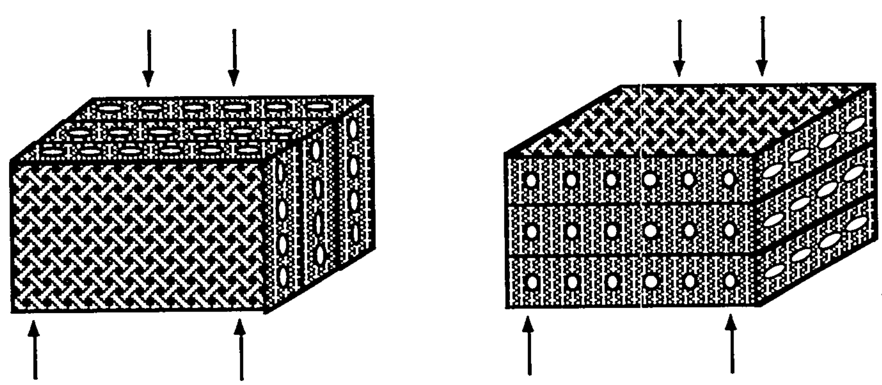

Figure 1. Geometries for testing Nicalon fabric reinforced ceramic matrix composites: (a) edge-on and (b) transverse.

Fractography

The composite fracture surfaces were examined under a Hitachi S-800 Scanning Electron Microscope (SEM), at the High Temperature Materials Laboratory (HTML) of the Oak Ridge National Laboratory (ORNL). An excitation voltage of 5 KeV was used to illuminate the electron beam on fracture surfaces. The images were directly captured onto a Macintosh personal computer using Adobe Photoshop software (version 2.5). The digital images were then processed using a public domain NIH Image software (version 1.57). The Image program was used to select areas of interest in the computer-captured images and enhance the "picture quality."

Results and Discussion

Monotonic Behavior

For the Nicalon/Al_2O_3 composite, significant differences were observed in the stress-strain curves for the two specimen configurations investigated (Figure 2). In the edge-on orientation, the ultimate flexural strength was 503 ± 11 MPa, while the strength was only 341 ± 9 MPa in the transverse orientation. However, the proportional limit was 70 MPa for both configurations, indicating that the proportional limit is an intrinsic property of the material and is independent of fabric orientation. This trend is consistent with the trends observed by Liaw [3] and Chawla et al. [4, 5] in Nicalon/SiC and Nextel/SiC composites. Also, the flexural proportional limit observed in the present investigation, 70 MPa, is comparable to the tensile proportional limit of 60 MPa reported by Fareed et al. [6]. From Figure 2, it can be seen that the strain to failure and the area under the stress-strain curve are higher for the edge-on orientation than the transverse orientation, which indicates that the edge-on orientation is tougher than the transverse orientation. It is a well known fact that the 2-D composites suffer from poor interlaminar strength. As a result, the fabric plies can slide with respect to each other in the transverse orientation, while the relative movement is not permitted in the edge-on orientation. Fractography revealed that multiple cracking (Figure 3) and interfacial debonding were observed in the composite. Interlaminar cracking was observed in the transverse specimens, while it was not observed in the edge-on specimens.

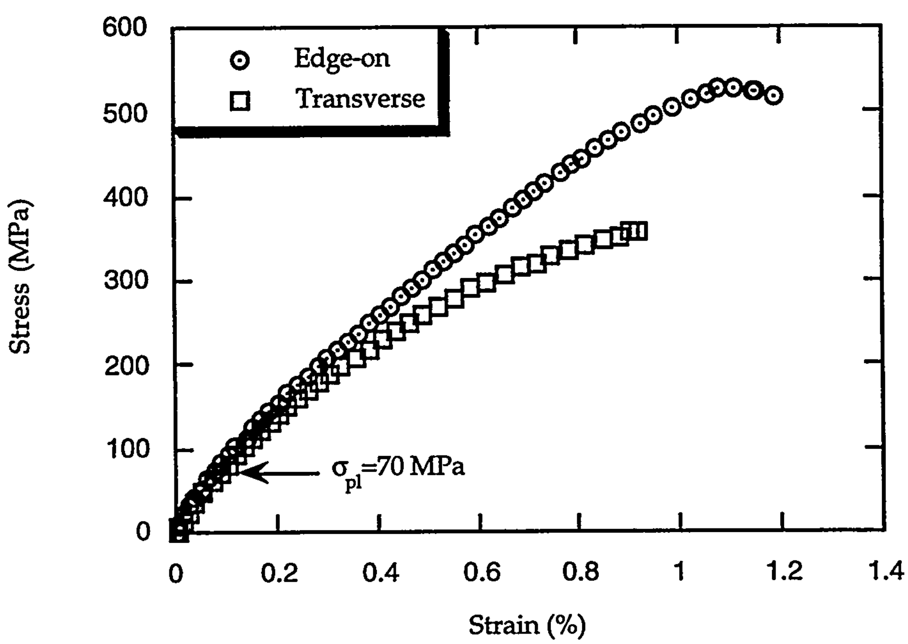

Figure 2. Stress-strain curves in the edge-on and transverse orientation for the Nicalon/Alumina composite.

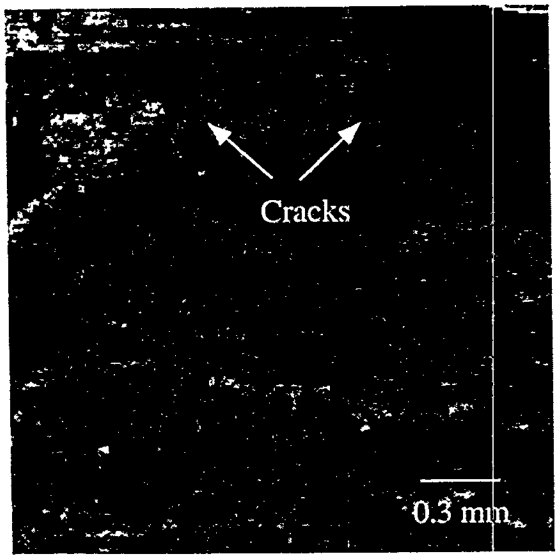

Figure 3. Multiple cracks on the tensile surface of a Nicalon/Alumina flexural specimen.

Interestingly, the interlaminar cracks did not propagate along the interface between two fabric plies. Instead, cracks were formed by the linkup of debonded interfaces within a fabric ply [7]. Fiber pullout, which contributes to composite toughness, was evident in specimens tested in both orientations (Figure 4).

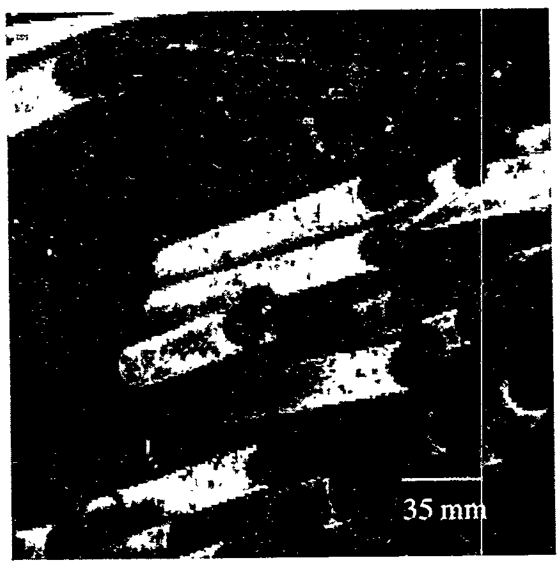

Figure 4. Fiber pullout, indicative of toughness in the edge-on orientation of the Nicalon/Alumina composite.

For the Nicalon/SiC composite, a significant scatter in ultimate strength values was observed in both edge-on and transverse orientations. The flexural strength in the edge-on orientation was 234 ± 27 MPa, while the strength was 241 ± 23 MPa in the transverse orientation. The large scatter in the strength values can be attributed to the variation in the density of the samples (2.38 to 2.64 g/cm^3). The flexural stress-strain curves for the Nicalon/SiC composites are presented in Figure 5. Although the average flexural strength is about 7 MPa higher in the transverse orientation than in the edge-on orientation, it is worth noting that this

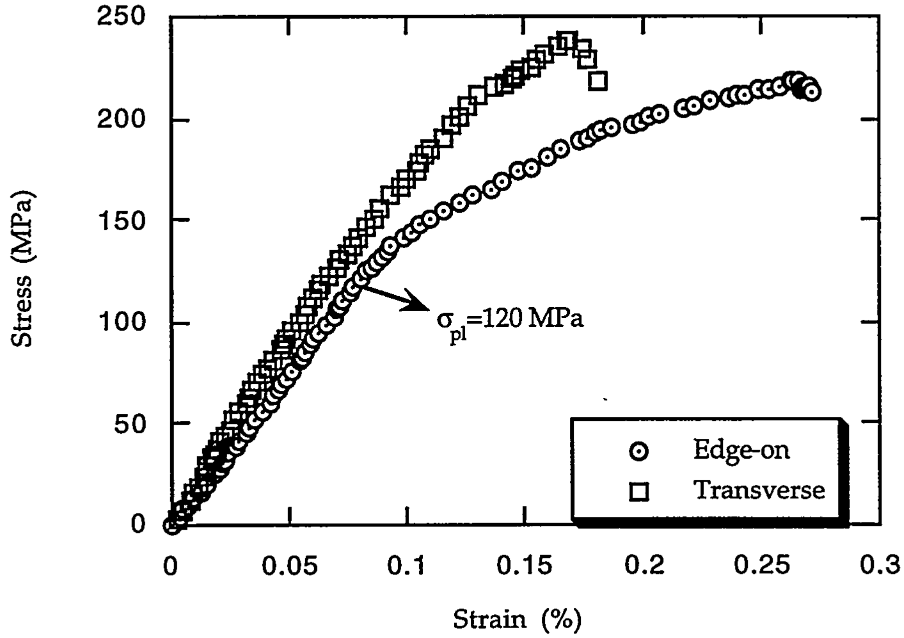

Figure 5. Stress-strain curves in the edge-on and transverse orientation for the Nicalon/SiC composite.

value is within the scatter band for the strength in both orientations. Therefore, it can be said that the strength of the composite is essentially unaffected by the orientation of fabric plies to the applied load in this composite, which is in contrast with that observed for the Nicalon/Al_2O_3 composite used in the present study. However, the proportional limit in both orientations was 120 MPa, similar to the trend observed in the Nicalon/Al_2O_3 composite. The strain to failure and the area under the stress-strain curve are higher in the edge-on orientation than in the transverse orientation, similar to the trend found in the Nicalon/Al_2O_3 composite. While multiple matrix cracking and fiber pullout were observed in the samples tested in both orientations, interlaminar cracking was observed only in the samples tested in the transverse orientation [7].

Fatigue Behavior

The stress versus number of cycles (S-N) curves for the Nicalon/Al_2O_3 composite in the edge-on and transverse orientations are shown in Figure 6. The endurance limit (the stress at which the material survives 1 million cycles) in the edge-on orientation was 318 MPa, which is about 65 % of the monotonic flexural strength. In contrast, the endurance limit in the transverse orientation was 257 MPa, which is about 75% of the monotonic strength. Thus, it appears that cyclic loading reduces the load bearing capacity of the composite in the edge-on orientation, more than it does in the transverse orientation. The proportional limit for both orientations was 70 MPa. At stresses above the proportional limit, extensive matrix cracking occurs in CFCCs. The specimens tested in the edge-on orientation were subjected to a higher level of fatigue stresses than the specimens tested in the transverse orientation (Figure 6). Hence, it could be expected that the degradation in material strength due to matrix cracking was much higher in the edge-on orientation than in the transverse orientation.

The progressive damage in the composite can be *qualitatively* monitored using the slope of the linear portion in the load versus midspan deflection traces recorded at periodic intervals. The slope at any point during the fatigue test can be normalized with respect to the slope for the first cycle and plotted against the number of cycles. The normalized slope is known as the effective slope. However, it must be emphasized

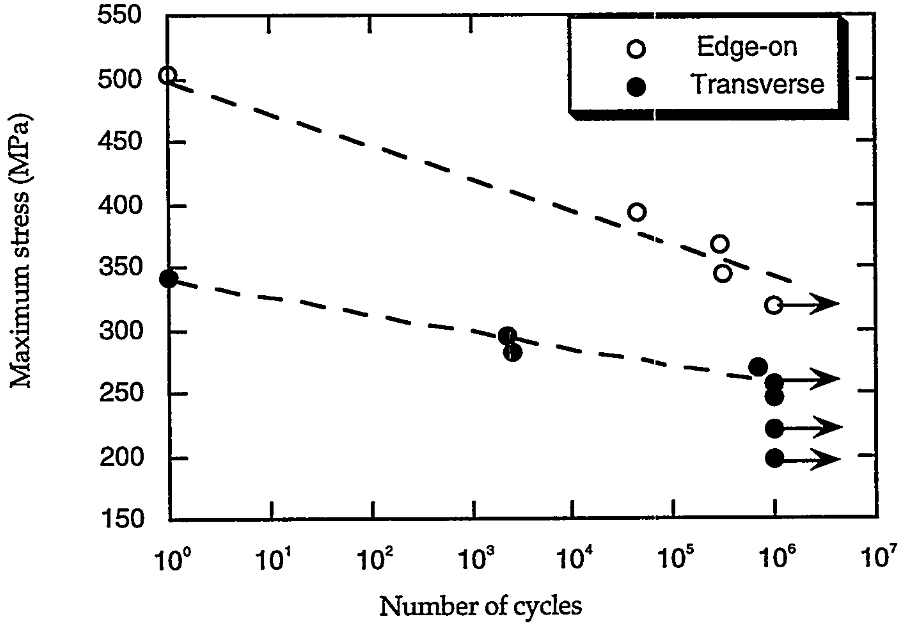

Figure 6. Stress versus life curves for the Nicalon/Alumina composite in the edge-on and transverse orientations.

that the normalized slope values do not readily translate into normalized modulus values. The effective modulus values are plotted against the number of cycles for the Nicalon/Al_2O_3 composite in Figures 7 and 8. It can seen from these figures that the effective modulus values continued to drop rapidly with increased cycling in the edge-on orientation, while the drop in the effective modulus values with cycling was more gradual for the specimens tested in the transverse orientation. However, at present, the reasons for the shapes of the curves observed are not apparent. It is hoped that *in situ* microscopy of the specimens, which will be performed shortly, might provide an insight into the damage in the material and hence help understand the stress versus life and effective modulus trends observed upon cyclic loading.

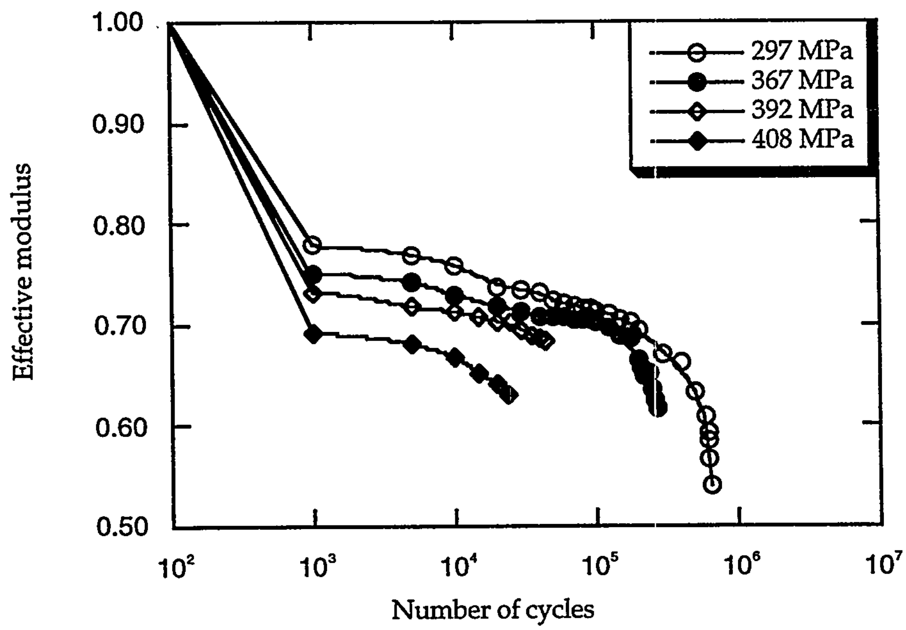

Figure 7. The reduction in effective modulus due to fatigue in the edge-on orientation of the Nicalon/Alumina composite.

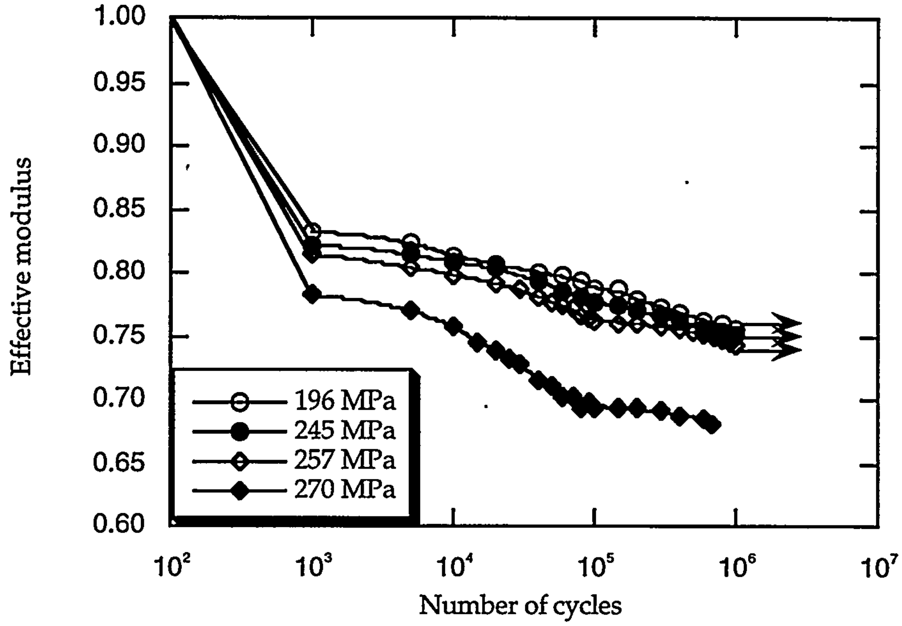

Figure 8. The reduction in effective modulus due to fatigue in the transverse orientation of the Nicalon/Alumina composite.

The S-N curves for the Nicalon/SiC composite in the edge-on and transverse orientations are shown in Figures 9. It is apparent that, in contrast to the Nicalon/Al_2O_3 composite, the endurance limit seems to be slightly higher in the transverse orientation than in the edge-on orientation. For the edge-on orientation, while specimens tested at a maximum stress of 207 MPa (which corresponds to 88% of the average monotonic ultimate strength) survived almost 1 million cycles, those attempted to be tested at a maximum stress 214 MPa failed before the maximum load could be applied. It must be mentioned here that there is a large scatter in monotonic ultimate strength values (234 ± 27 MPa) in the edge-on orientation. A stress value of 207 MPa is within this scatter band. Therefore, it can be postulated that the strength degradation due to cyclic loading is marginal in this composite. In the transverse orientation, the Nicalon/SiC specimens survived 1 million cycles at a maximum stress of 223 MPa, which corresponds to approximately 92 % of the average monotonic strength. The composite specimens survived > 10^5 cycles at a maximum stress of 231 MPa, which is approximately 96% of the average monotonic ultimate strength. A stress value of 223 MPa is within the scatter band for the monotonic strength values (241 ± 23 MPa). Hence, it can be deduced that the strength degradation due to cyclic loading is rather small in the transverse orientation as well. The effective modulus values for the edge-on and transverse orientations are plotted against the number of cycles in Figures 10 and 11, respectively. It can be seen that the effective modulus values decreased only marginally, after the initial drop, in the specimens that survived 1 million cycles, while the values dropped considerably for the samples that failed in less than a million cycles. It is interesting to note that higher monotonic strength translated into higher fatigue strength, with regard to fabric orientation, in both composites. This behavior is similar to that observed in metallic materials. Fractography revealed that a significant amount of repeated interfacial sliding was operative in the Nicalon/SiC composite specimens subjected to fatigue loading (Figure 12). While this phenomenon suggests that significant damage was induced at the fiber/matrix interface upon cyclic loading, surprisingly, however, the S-N curves indicate that strength degradation due to fatigue loading was only marginal in both edge-on and transverse orientations. In view of the small differences in the monotonic and endurance limit values in the edge-on and transverse orientations, it can be hypothesized that the fabric orientation effects are not strong in the Nicalon/SiC composite.

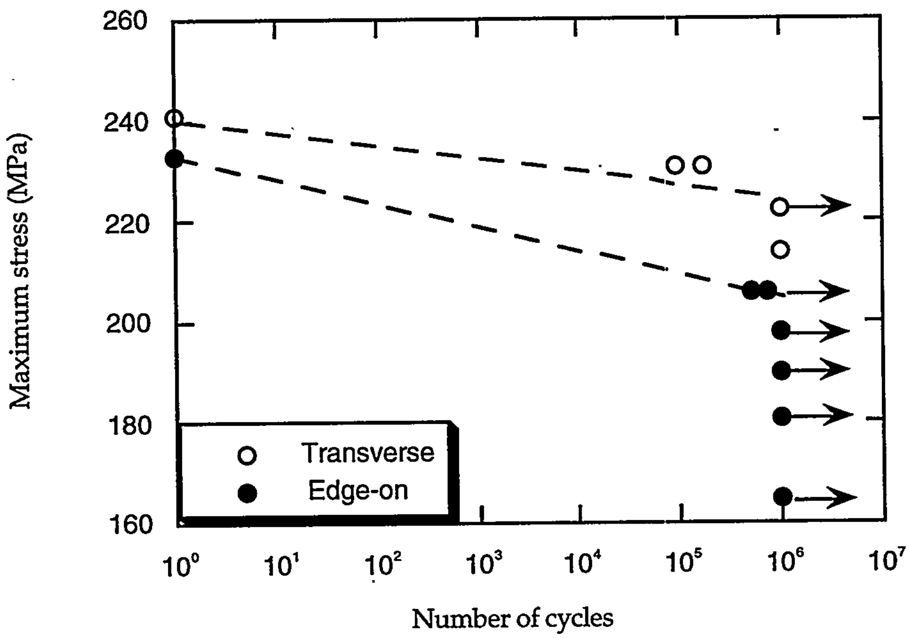

Figure 9. Stress versus life curves for the Nicalon/SiC composite in the edge-on and transverse orientations.

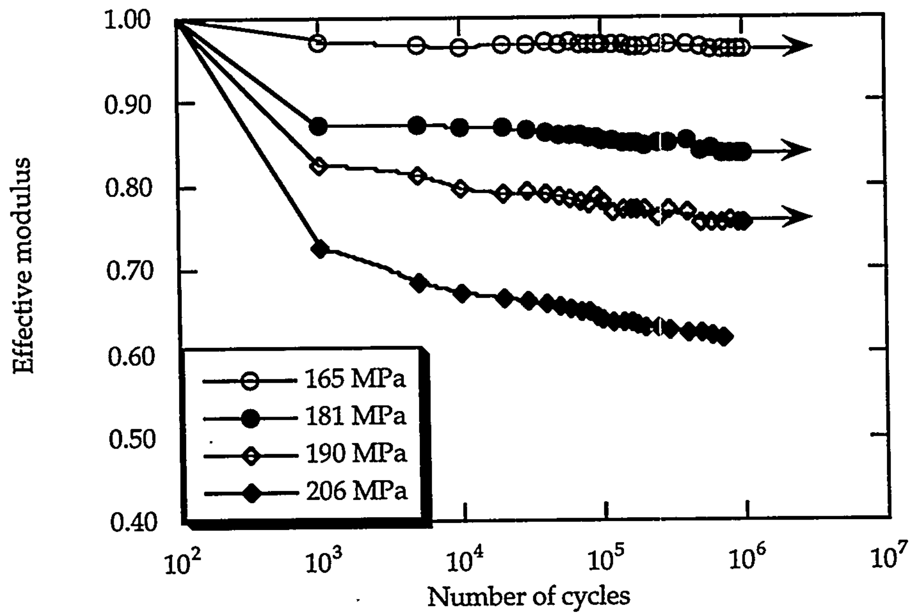

Figure 10. The reduction in effective modulus due to fatigue in the edge-on orientation of the Nicalon/SiC composite.

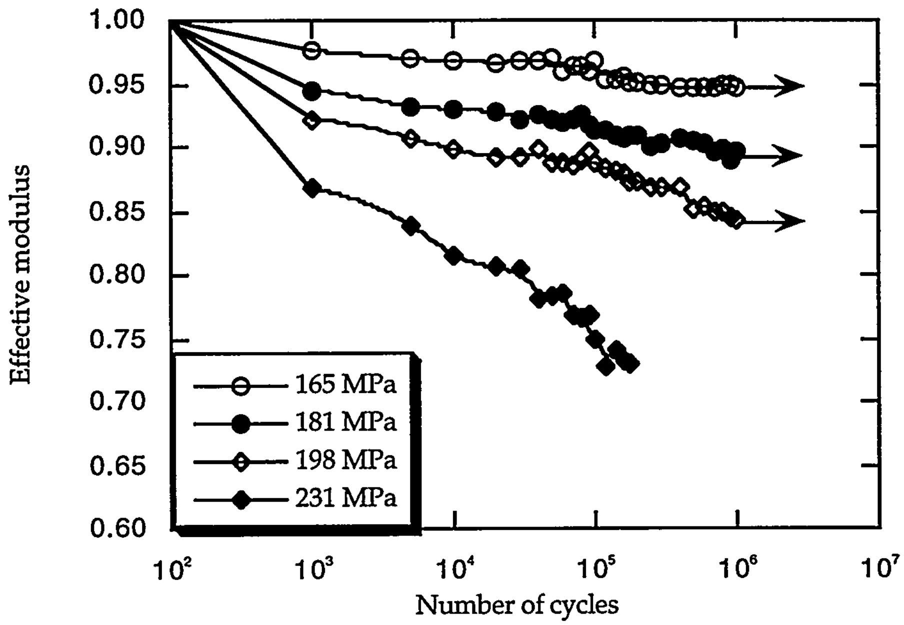

Figure 11. The reduction in effective modulus due to fatigue in the transverse orientation of the Nicalon/SiC composite.

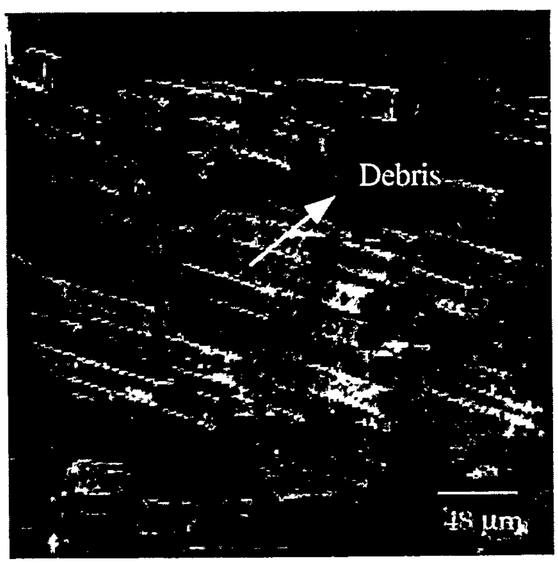

Figure12. Debris left by repeated interfacial sliding in the Nicalon/SiC composite.

CONCLUSIONS

The monotonic and fatigue behavior of the Nicalon/Alumina composite seems to be strongly influenced by the orientation of fabric plies to the applied loads, while the orientation effects seem to be only marginal for the Nicalon/SiC composite. In the Nicalon/Alumina composite, the monotónic and fatigue strengths were higher in the edge-on orientation than in the transverse orientation. In the Nicalon/SiC composite, the opposite trend was observed, i.e., the monotonic and fatigue strengths were higher in the transverse than in the edge-on orientation. However, in both composites higher monotonic strength translated into higher fatigue strength. This trend is identical to that observed for metallic materials.

FUTURE WORK

All the experiments to date have been conducted at room temperature. The modulus reduction plots indicated some interesting trends in damage accumulation upon cyclic loading in the Nicalon/Alumina and Nicalon/SiC composites. However, an understanding of progressive damage has not been achieved yet. Well polished flexural bars will be *in situ* monitored for damage development in the next few months. Elevated-temperature tests will also be performed to study the monotonic and fatigue behavior of the two CFCCs used in the present investigation, with particular regard to fabric orientation effects. In addition, the influence of span-to-thickness ratio, on the monotonic and fatigue behavior of both composites, will be evaluated. After an understanding of the damage mechanisms that operate in the two composites is developed, an attempt will be made to use finite element method (FEM) analysis to help explain some of the experimental results.

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REFERENCES

1. J. A. Dicarlo, Adv. Mater. Proc. (June 1989) 41.
2. R. W. Rice, Ceram. Eng. Sci. Proc. **2** (1981) 661.
3. P. K. Liaw, JOM (October 1995) 38.
4. N. Chawla, M. S. Thesis, University of Tennessee, Knoxville, 1994.
5. N. Chawla, P. K. Liaw, E. Lara-Curzio, R. A. Lowden, and M. K. Ferber, in **High Performance Composites - Commonalty of Phenomena**, K. K. Chawla, P. K. Liaw and S. G. Fishman (eds.), TMS, Warrendale, PA (1994) 291.
6. A. S. Fareed, D. J. Landini, T. A. Johnson, A. N. Patel, and P. A. Craig, paper EM92-216, SME, Dearborn, MI, 1992.
7. N. Miriyala, Doctoral Dissertation Proposal, The University of Tennessee, 1995.