THE MONOTONIC AND FATIGUE BEHAVIOR OF CFCCs AT AMBIENT TEMPERATURE AND 1000°C - 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 continuous fiber-reinforced ceramic-matrix composites (CFCCs) at ambient and elevated temperatures.

SUMMARY

Metallographically polished flexure bars of Nicalon/SiC and Nicalon/alumina composites were subjected to monotonic and cyclic-fatigue loadings, with loading either parallel or normal to the fabric plies. The fabric orientation did not significantly affect the mechanical behavior of the Nicalon/SiC composite at ambient temperature. However, the mechanical behavior of the Nicalon/alumina composite was significantly affected by the fabric orientation at ambient temperature in air and at 1000°C in argon atmosphere. In addition, there was a significant degradation in the fatigue performance of the alumina matrix composite at the elevated temperature, owing to creep in the material and degradation in the fiber strength.

PROGRESS AND STATUS

Introduction

It is generally observed that unidirectionally reinforced composites exhibit good strength and toughness when the load is applied parallel to the fiber orientation, but perform poorly when the load is applied normal to the fibers. Hence, in applications where multi-axial stresses are expected, two dimensional (2-D) or three dimensional (3-D) reinforcement is commonly used [1, 2]. The 2-D composites are usually manufactured by first making a woven or braided-fabric preform, which is then infiltrated with the matrix, using liquid or vapor infiltration techniques [3, 4]. However, the interlaminar strength of these composites is often poor, which may lead to significant differences in the mechanical behavior of the material, depending on whether the load is applied parallel or normal to the fabric plies. Accordingly, it is the objective of the current study to systematically investigate the effects of fabric orientation on the mechanical behavior of two of the commercially available 2-D CFFCs, namely, Nicalon/silicon carbide (SiC) and Nicalon/alumina composites manufactured by DuPont-Lanxide Corporation.

In our previous studies [5, 6], the monotonic and fatigue behavior of the two composites was studied at ambient temperature. It was observed that the fabric orientation significantly affected the mechanical behavior of the Nicalon/alumina composite, while the effects were insignificant for the Nicalon/SiC composite. However, it was not possible to develop an understanding of the damage mechanisms in the two materials, as unpolished specimens were used in the experiments. In the present study, carefully polished flexure specimens were used to perform the mechanical tests. The monotonic and fatigue behavior of the Nicalon/SiC composite was studied at ambient temperature. For the Nicalon/alumina composite, the monotonic and fatigue tests were performed at ambient temperature in air, and at 1000°C in argon atmosphere. The specimen fracture surfaces were examined under a scanning electron microscope to understand the damage mechanisms operative under both static and cyclic-fatigue loadings.

Experimental Details

Materials

The material details have already been described in previous progress reports [5, 6] and, hence, only a brief mention of them will be made here. The Nicalon/SiC composite, fabricated by an isothermal chemical vapor infiltration (ICVI) process, was provided by AlliedSignal Engines, Phoenix, AZ. 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.

The Nicalon/alumina composite, fabricated by a direct molten metal oxidation (DIMOX) process, was supplied by Westinghouse Electric Corporation, Orlando, FL. 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). 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.

Flexure testing

The Nicalon/SiC flexure bars were 50 mm long, 2 mm wide and 2 mm thick, while the Nicalon/Alumina samples were 50 × 3 × 3 mm [5, 6]. The details of the MTS servohydraulic system used to perform the mechanical tests can be obtained from references 5 and 6. 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 normal to the fabric plies, respectively [6]. The ambient-temperature monotonic and fatigue tests were conducted in air, while the high-temperature tests were performed at 1000°C in argon atmosphere. The monotonic tests were conducted under displacement control at a crosshead displacement rate of 0.5 mm/min. The cyclic-fatigue tests were conducted under load control using a sinusoidal wave form. The loading frequency was 0.5 Hz up to 1000 cycles, and then gradually increased to 5 Hz and maintained at that frequency till the completion of tests. A load-ratio (minimum load/maximum load) of 0.1 was used in all the cyclic fatigue tests. The fatigue run-out was set at one million cycles, which corresponded to approximately 56 hours of testing.

Metallographic Examination

Unsevered composite specimens, after unloading at periodical intervals during the fatigue tests, as well as the specimen fracture surfaces following the monotonic and fatigue tests, 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), to observe the damage mechanisms in the two materials investigated. A Macintosh personal computer was interfaced to the SEM to digitally capture the images using the Adobe Photoshop software (version 2.5).

Results and Discussion

Nicalon/SiC Composite

From our previous studies on the mechanical behavior of the Nicalon/SiC composite [5,6], it was observed that the flexural strength in the edge-on orientation was 234 ± 27 MPa, while the strength was 241 ± 23 MPa in the transverse orientation. It was also evident that the difference in flexural strength between the two orientations was insignificant, considering the scatter in the strength data and the porosity content. Stress-life (S-N) tests were also performed at ambient temperature, and it was noted that specimen failure did not occur even when the maximum stress during the tests was about 190 MPa (80 % of the monotonic strength), in both orientations [6]. From the effective modulus and midspan deflection trends, it appeared that the fabric orientation did not significantly affect the fatigue behavior of the Nicalon/SiC composite. The slope of the load versus midspan deflection values decreased after the first loading cycle in all the samples. After a relatively steep drop of up to 10% in the first ten cycles, the decrease in effective modulus was less precipitous as the loading continued. It appeared that much of the damage in the composite material occurred in the first few cycles. However, an understanding of the damage mechanisms was lacking since unpolished specimens were used in performing the mechanical tests. To meet this objective, metallographically polished flexure specimens were used to perform the monotonic and fatigue tests in the present study.

The fracture surfaces after monotonic loading, in both edge-on and transverse orientations, were fibrous, indicating substantial fiber pullout. Matrix cracking was observed mostly in the transverse fiber tows (yarns oriented at 90° to the long axis of the specimen and intersecting the bottom surface in the edge-on orientation, or the cross-section in the transverse orientation). The interlaminar pores appeared to have acted as stress concentration sites, and microcracks initiated from these pores (Figure 1), and propagated primarily into the transverse fiber tows. These cracks linked up to cause specimen failure. Specimen separation was observed to be primarily along the interlaminar pores (Figure 2). Matrix cracking in the transverse fiber tows, fiber breakage and interlaminar cracking appear to be the main failure mechanisms in the composite material, under monotonic loading, in both orientations. However, from the examination of specimen surfaces, it appeared that interlaminar porosity played a bigger role than matrix cracking in the material failure [7].

The specimens subjected to cyclic-fatigue loading were periodically unloaded from the test machine to observe damage progression in the material. Although the effective modulus trends indicated that the degradation in material load bearing capability occurred during the early stages (< 10 cycles) of fatigue loading, the SEM examination did not reveal any cracking in the material. It may, however, be noted that the specimens used in the present investigation were very small (50 × 2 × 2 mm). Consequently, the damage evolution was limited, since only a small fraction of the bend bar volume is subjected to the maximum tensile stress in a flexure test. However, matrix cracking was readily observed on the specimen surfaces after about 1,000 loading cycles (Figure 3). Cracks appeared to have initiated from the interlaminar pores and propagated primarily into the transverse fiber tows in both orientations. In some samples, matrix cracking in the transverse fiber tows appeared to link up some of the smaller interlaminar pores, forming a weak layer in the material (Figure 4).

Nicalon/Alumina Composite

The flexural strength of the composite was significantly higher in the edge-on orientation, compared to the transverse orientation, at both ambient and elevated temperatures. In the edge-on orientation, the ultimate flexural strength values were 474 ± 7 MPa and 468 ± 18 MPa, respectively, at room temperature and 1000°C. In contrast, for the transverse orientation, the room-temperature flexural strength was 330 ± 20 MPa, while the high-temperature strength was 346 ± 13 MPa. The flexural stress-strain curves at 1000°C are presented in Figure 5. It can be seen from Figure 5 that the stress-strain curves were non-linear in both orientations.

While the flexural strength and the strain to failure values were higher in the edge-on orientation, compared to the transverse orientation, the proportional limit (the stress at which the stress-strain curve deviates from linearity) was approximately the same (70 MPa) for both orientations. The stress-strain curves, and the proportional limit, at ambient temperature were similar to those at 1000°C [8, 9].

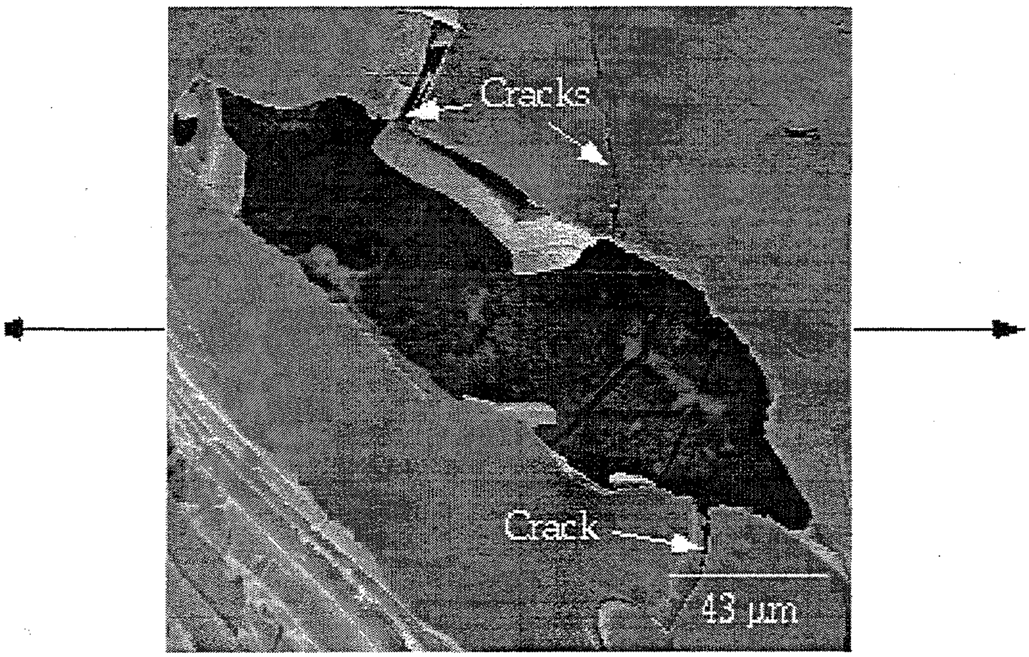


Figure 1. Crack initiation from interlaminar pore, on the tensile (bottom) surface) of an edge-on specimen monotonically loaded to failure .

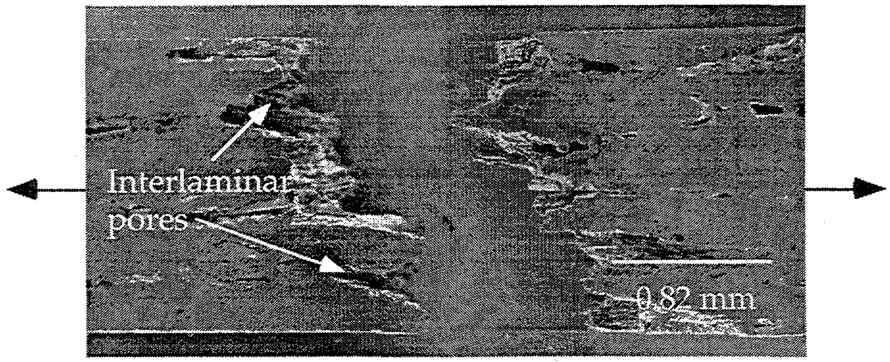


Figure 2. Tensile surfaces of an edge-on specimen separated along the interlaminar pores after monotonic loading.

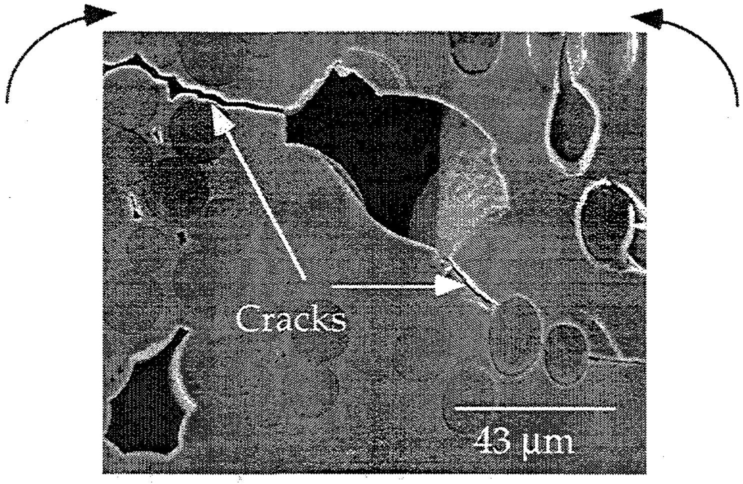


Figure 3. Cracks initiated from an interlaminar pore propagating into a transverse fiber tow in a transversely loaded specimen subjected to fatigue loading.

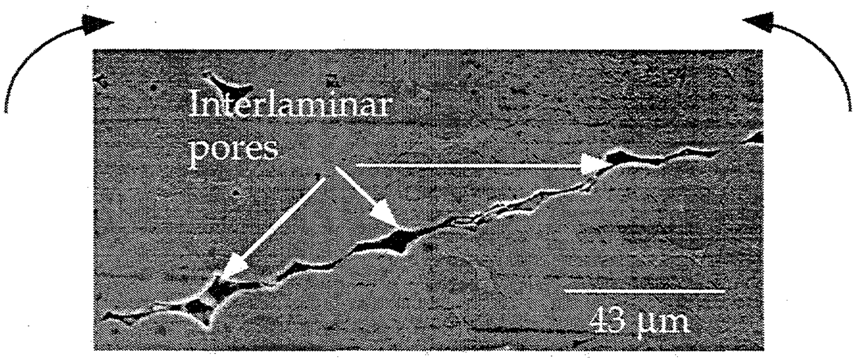


Figure 4. Matrix cracking in a transverse ply linking up interlaminar poresin a transversely loaded specimen subjected to fatigue loading.

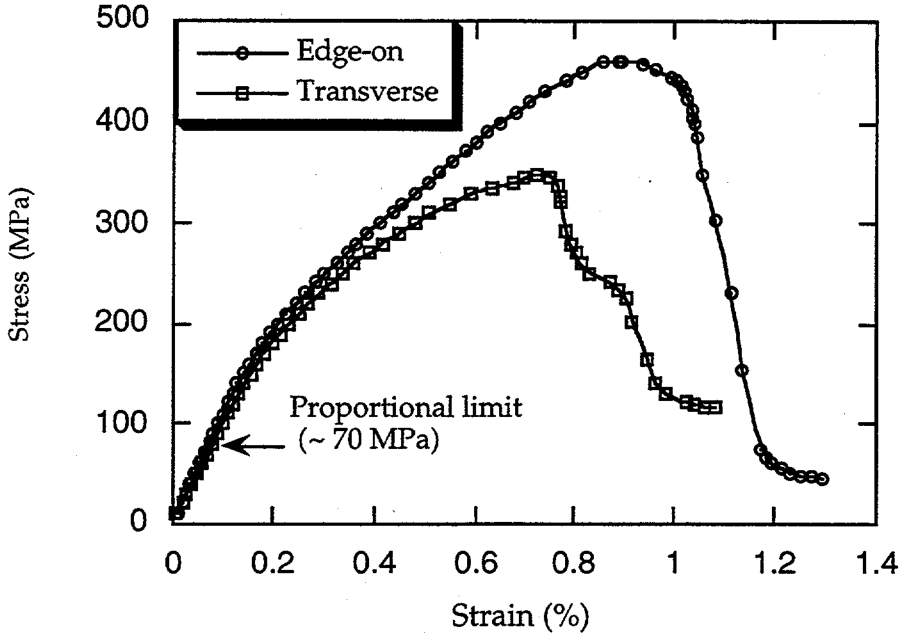


Figure 5. Stress-strain curves for the Nicalon/alumina composite in the edge-on and transverse orientations at 1000°C in argon atmosphere.

Fractography revealed that the failures modes in the edge-on and transverse orientations were significantly different. The edge-on specimens failed in a predominantly tensile mode, with cracking in the 90° fiber bundles preceding the failure of the 0° fibers. In most cases, the specimen broke into two pieces (Figure 6). In the transverse orientation, cracking initiated in the 90° tows, and some of these cracks developed into interlaminar cracks, leading to collapse rather severance of the specimens (Figure 7). Multiple matrix cracking and interfacial bonding were observed in all the samples. It was also observed that the cracks in the 90° fiber tows propagated primarily along the fiber-matrix interface. For a given orientation, the failure modes were similar at ambient and elevated temperatures.

While the monotonic behavior of the composite was not affected by the test temperature, there was a significant degradation in the fatigue behavior of the material, particularly in the edge-on orientation, due to the prolonged exposure to elevated temperatures. The stress versus number of cycles (S-N) curves for the composite at ambient and elevated temperatures are shown in Figure 8. It can be seen from Figure 8 that, at room temperature, in the edge-on orientation, the ratio of endurance limit (the stress at which the specimen survives 1 million cycles) to flexural strength was approximately 0.67, while the ratio was only 0.45 at 1000°C. The corresponding ratios in the transverse orientation were 0.77 and 0.69, respectively, at ambient and elevated temperatures. Thus, the degradation in the fatigue behavior appears to be more severe in the edge-on orientation compared to the transverse orientation. It is also apparent from Figure 5 that, at room temperature, the endurance limit was significantly higher in the edge-on orientation, compared to the transverse orientation. In contrast, the endurance limit at 1000°C was approximately the same in both orientations. It is important to note that, at room temperature, the specimens tested in the edge-on orientation were subjected to much higher stress levels than the transversely oriented specimens. At these higher stress levels, the 0° fibers failed gradually, leading to specimen severance, while the transverse specimens, subjected to much lower stress levels, failed by interlaminar cracking, followed by specimen collapse. At 1000°C, however, the stress levels used in both orientations are comparable. While the S-N line slopes for the transverse orientation at room temperature and 1000°C are comparable, the slope is much

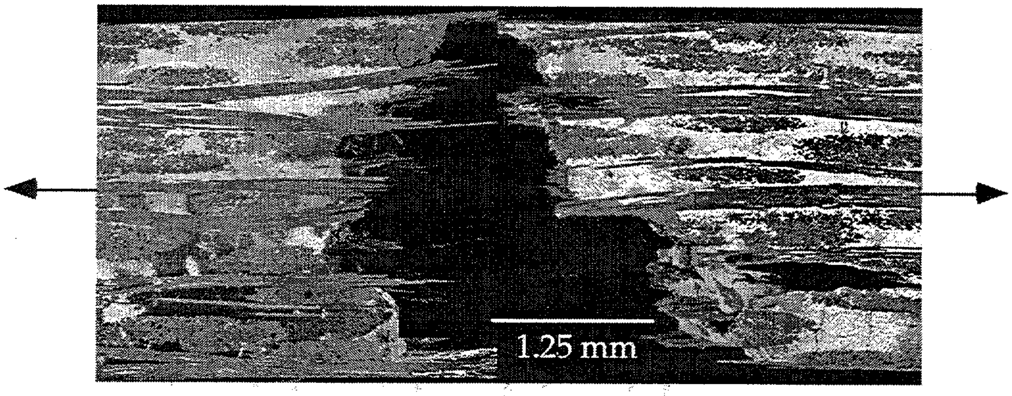


Figure 6. Tensile (bottom) surfaces of an edge-on specimen monotonically loaded at ambient temperature.

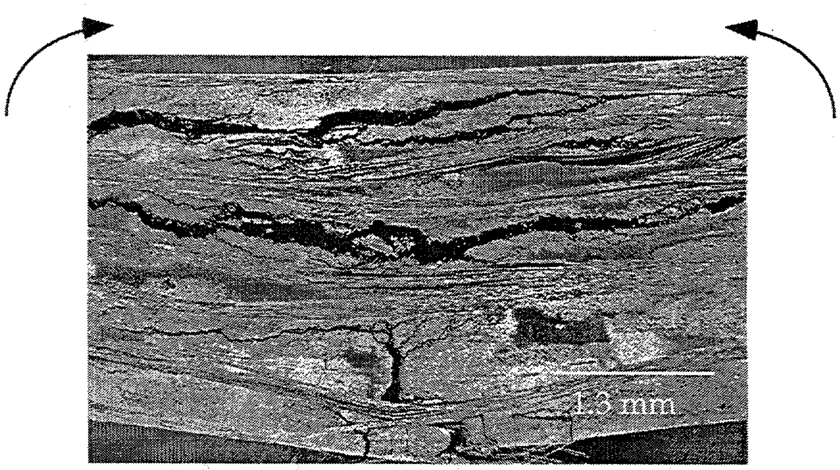
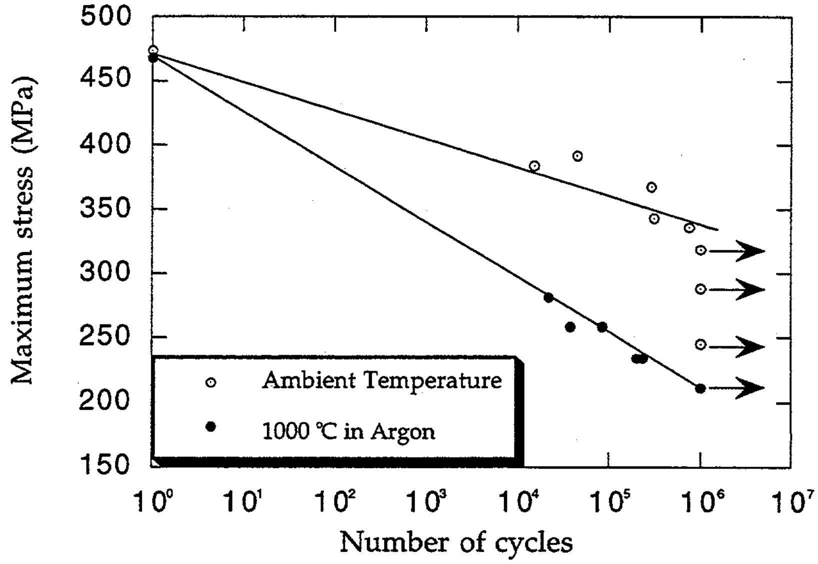
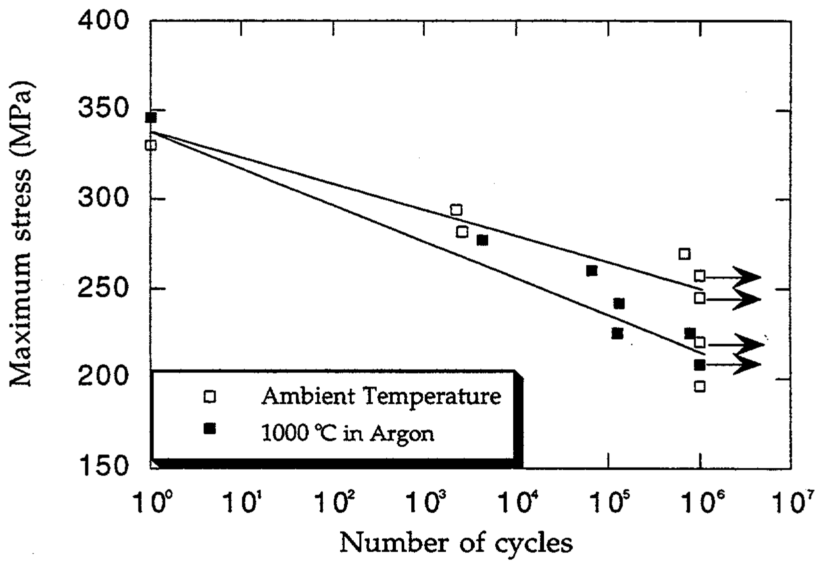


Figure 7. Cross-section of a transverse specimen monotonically loaded at ambient temperature.



(a)



(b)

Figure 8. Stress versus life curves for the Nicalon/alumina composite at ambient temperature and 1000°C in (a) edge-on orientation and (b) transverse orientation.

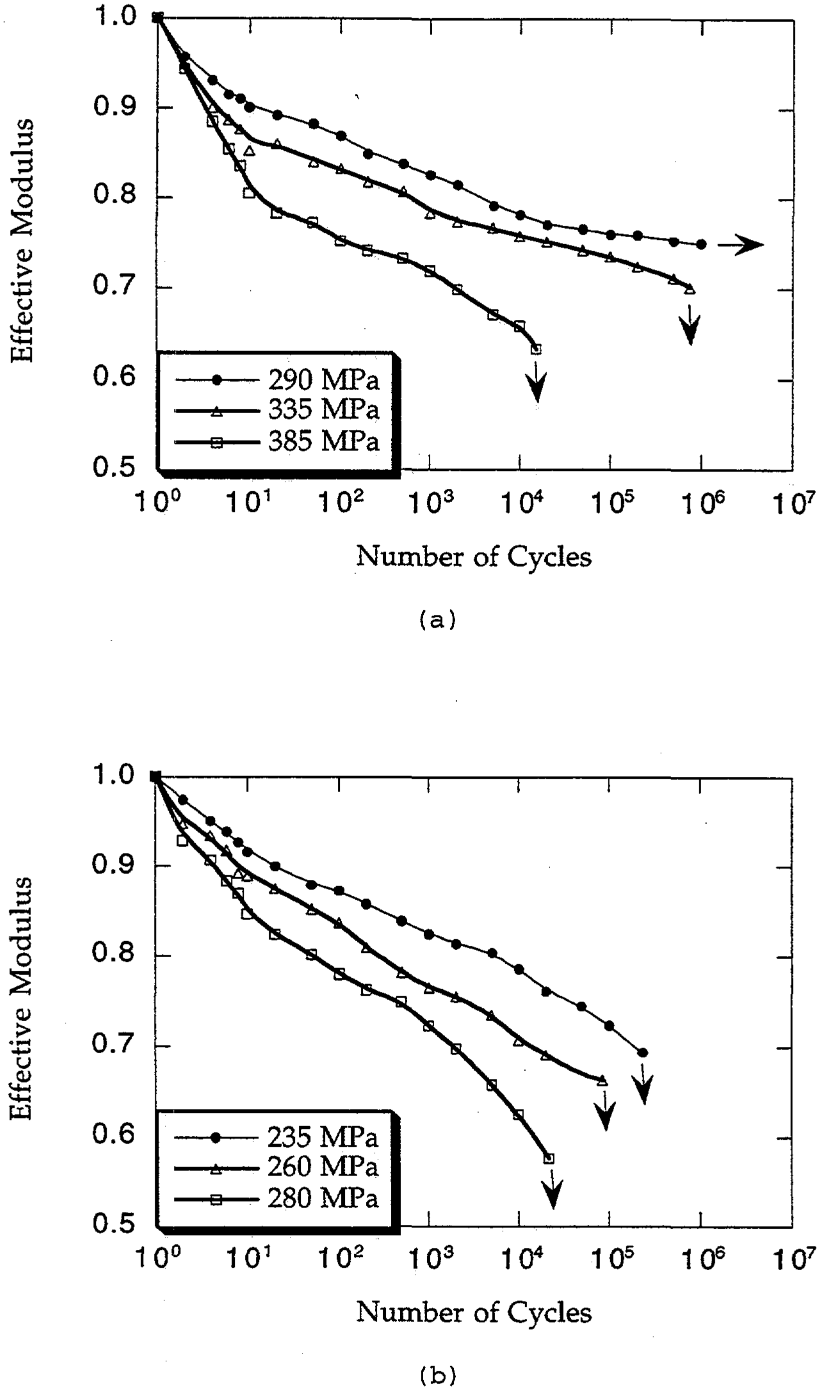


Figure 9. Reduction in effective modulus upon cyclic loading in the edge-on orientation at (a) ambient temperature and (b) 1000°C in argon atmosphere.

steeper at the high temperature, compared to that at the ambient temperature, for the edge-on orientation. Some specimens were loaded to the stress levels used in the fatigue experiments at 1000°C and held at a constant stress for about 50 hours or till the specimen failure, whichever occurred earlier [9]. It was noticed that the midspan deflection values increased significantly, particularly at the higher stress levels, in the first few hours of testing. It is also well known that the Nicalon™ fibers degrade in strength, upon prolonged exposure to elevated temperatures [10]. We believe that the creep in the material and the fiber degradation are responsible for the poor fatigue behavior of the composite at the elevated temperature, particularly in the edge-on orientation.

The effective modulus value trends for the Nicalon/alumina composite, in the edge-on orientation, at room temperature and 1000°C, respectively, are plotted in Figures 9. It can be seen from Figure 9 that the effective modulus values continued to drop after the first cycle, in both orientations, at ambient as well as elevated temperatures. The initial drop in the effective modulus values (< 1000 cycles) was observed to be due to cracking in the 90° fiber bundles, in both orientations, at ambient as well as elevated temperatures. These cracks then penetrated into 0° fiber tows in the edge-on orientation, but propagated as interlaminar cracks in the transverse orientation. For a given orientation, the failure modes at room temperature and 1000°C were similar. However, the creep of the specimens and degradation of the Nicalon fibers contributed to more precipitous drop in the effective modulus values at the high temperature.

CONCLUSIONS

The monotonic and fatigue behavior of the Nicalon/SiC composite at ambient temperature appears to be unaffected by the orientation of fabric plies to the applied loads. There was a large scatter in the flexural strength values, owing to the variation in porosity content and distribution. Upon cyclic-fatigue loading, the effective modulus of the composite decreased after the first loading cycle in both orientations. After a relatively steep decrease in the modulus values in the first ten cycles, the modulus values decreased more gradually upon further cycling. The specimen failure after monotonic and cyclic-fatigue loadings, in both orientations, occurred due to matrix cracking in the transverse fiber tows, interlaminar cracking, and fiber breakage.

The flexural strength of the Nicalon/alumina composite was significantly higher in the edge-on orientation, compared to the transverse orientation, at ambient and elevated temperatures. While the monotonic behavior was unaffected by the exposure to 1000°C, there was a significant deterioration in the fatigue behavior of the material, particularly in the edge-on orientation, at the elevated temperature, compared to the behavior at the ambient temperature. The edge-on specimens failed by cracking in the 90° fiber tows, followed by the failure of the 0° fibers. In contrast, the cracking in the 90° bundles was followed by interlaminar cracking in the transverse orientation. The creep in the material, and the degradation of the Nicalon fibers, further contributed to the poor fatigue behavior at 1000°C.

FUTURE WORK

Monotonic and fatigue tests will be performed on the Nicalon/SiC composite at 1000°C in argon atmosphere. In addition, the influence of span-to-thickness ratio, on the monotonic and fatigue behavior of both composites, will be evaluated. Finally finite element method (FEM) analysis will be performed to help explain some of the experimental results.

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