

MONOTONIC AND FATIGUE LOADING BEHAVIOR OF A 2-D WOVEN CERAMICS MATRIX COMPOSITE AT ROOM AND ELEVATED TEMPERATURES

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SUMMARY: This study characterized a woven fabric reinforced ceramic matrix composite under monotonic and fatigue loading conditions at room and elevated temperatures in order: (1) to investigate monotonic tensile and compressive loading behavior at room and elevated (760°C) temperatures, (2) to establish the fatigue life diagrams (S-N curves) under tension-tension and tension-compression, low frequency (0.1Hz) loading conditions at room and elevated temperatures, and (3) to investigate damage mechanisms and failure modes under monotonic and fatigue loading conditions. The tested CMC had higher fatigue life in tension-tension cycling loading condition than in tension-compression cycling loading condition at a given maximum stress level at room temperature. However, it had the same fatigue life at the elevated temperature at a given stress level under tension-tension and tension-compression fatigue loading conditions.

KEYWORDS: ceramics matrix composites, 2-D woven, monotonic loading, fatigue loading, tension, compression, room and elevated temperatures.

INTRODUCTION

Ceramics Matrix Composites (CMCs) have the tremendous potential of application in various components in internal combustion engines, gas turbine engines, space vehicle engines and nozzles, etc. because of their ability to provide the higher damage tolerance at elevated high temperatures. While CMCs are, in general, relatively new materials, several studies have nevertheless been completed to investigate their behavior under various loading environments.

Non-woven CMCs have received a lot of attention, and their behavior has been characterized extensively and reported, e. g. see References 1-4. On the other hand, a lot less work has been reported on woven CMCs, their behavior needs to be investigated further. Furthermore, most of the research done involving mechanical characterization of woven CMCs have concentrated on tensile monotonic loading and tension-tension fatigue behavior, e. g. see References 5-7.

During application of CMCs, which would take advantage of their higher damage tolerance and temperature capabilities, there is always a possibility that these materials will be subjected to loading environments containing compressive loads. It is, therefore, necessary to investigate the mechanical behavior of CMCs under these loading environments also before they are put into use. This study, thus, investigated the mechanical behavior of a 2-D woven CMC under tensile and compressive monotonic loads as well as under tension-tension and tension-compression fatigue loads at room and elevated temperatures.

EXPERIMENTS

The material tested was NextelTM 312/BlackglasTM woven fabric ceramic matrix composite (CMC). The Nextel 312 is a polycrystalline metal oxide ceramic fiber. The reinforcement is made from *alumin-boria-silica* fibers, and has a composition of 62% Al₂O₃, 24% SiO₂, and 14% B₂O₃ w/o [8]. NextelTM is white in color, has a fiber diameter of 10-12 microns, possesses desirable density and coefficient of thermal expansion, and is known for its thermomechanical qualities of retaining its strength at high temperatures for extended periods of time. The NextelTM 312 material used in these tests had a *Boron Nitride (BN)* interface coating.

The BlackglasTM system used as the matrix material in this study is a *preceramic siloxane polymer* system. BlackglasTM has the advantages of resistance to oxidation, low density, very low viscosity, controllable thermal expansion coefficient by varying the carbon content, short cure time (3 hours), no evolution of harmful reaction gases during the cure process, and ease as well as low cost of fabrication [8].

The CMC material was received in the form of specimens which were rectangular in shape and had the nominal length, width, and thickness of 152, 12.3, and 2.3 mm, respectively. However, the specimens used for the monotonic compressive tests and tension-compression fatigue tests were cut to 114 mm length. This length was selected because it was short enough to alleviate flexure during compression part of testing. Thermocouple wires were attached on each side of the specimens to control the desired test temperature. These thermocouples were placed at 6.35 mm from the center of the specimen on both sides of the specimens, so that uniform temperature distribution was obtained throughout the cross-section of the test specimen.

A servohydraulic test machine was used in the present study. The hydraulic wedge grips of the test system were cooled using cold water. The cooling water was also used to cool fixture containing the quartz heat lamps which was used to heat the specimen at 760⁰ C. Temperature controllers regulated the heat lamps to maintain the desired temperature of test section (of about 25.4 mm length) at 760⁰ C. A high temperature extensometer with gage length of 12.0 mm was used to measure the strain in the specimen.

The loading waveform in the fatigue tests was of a triangular shape with a frequency of 0.1 Hz. The ratio of minimum to maximum stress levels ($\sigma_{\min}/\sigma_{\max}$), called R-ratio, in the tension-tension (T-T) and tension-compression (T-C) fatigue tests was 0.05 & -1, respectively. σ_{\min} is the smallest positive stress for T-T tests or the magnitude of the largest negative stress for T-C tests. σ_{\max} is the largest positive for both T-T and T-C tests.

RESULTS AND DISCUSSION

Monotonic Tests

Table 1 shows the summary of all results from monotonic tension and compression tests. This table provides the average of maximum stress and maximum strain at failure, the proportional limit (PL), and the Young's modulus of elasticity, E obtained in this study. Young's moduli of elasticity, obtained from the monotonic tension and compression tests, agree with each other for both room and elevated temperatures. The room temperature value of Young's modulus from the present study is within 5% of previously reported value [7]. The stress-strain curves became non-linear beyond the proportional limit as damage took place progressively in the inter-yarn region, 90° fibers, matrix, and 0° fibers. Also, in all of these tests the fracture occurred in plane perpendicular to the applied load in the gage section.

Table 1: Monotonic Loading Test Results

Test Type	Max. Strength (MPa)	Failure Strain (%)	E (GPa)	PL (MPa)
Room Temp. Tension	69	0.13	62.8	44
Elevated Temp. Tension	65	0.25	57.6	24
Room Temp. Compression	274	0.43	62.5	274
Elevated Temp. Compression	334	0.54	60.5	334

A typical stress-strain relationship from the elevated temperature monotonic tensile test is shown in Fig. 1. It can be seen that the stress-strain relationship is linear up to about 24 MPa, which is the proportional limit, then the curve becomes non-linear and shows a lower slope. The Young's modulus of elasticity calculated from the linear region up to the proportional limit is 57.6 GPa, and the slope in the non-linear region is 23.2 GPa. This is an indication that damage had occurred in the specimen. The failure stress in this test was about 65 MPa, which was slightly lower than the room temperature value of 69 MPa. The stress-strain relationship under the room temperature monotonic test showed the similar nonlinear behavior. The failure strain was 0.25%, which was about twice as much as the room temperature test failure strain. These values of failure stress and failure strain indicate that the elevated temperature had little

effect on the maximum stress that the material can withstand before it fails, and the material can withstand higher values of strain before it fails under elevated temperature conditions.

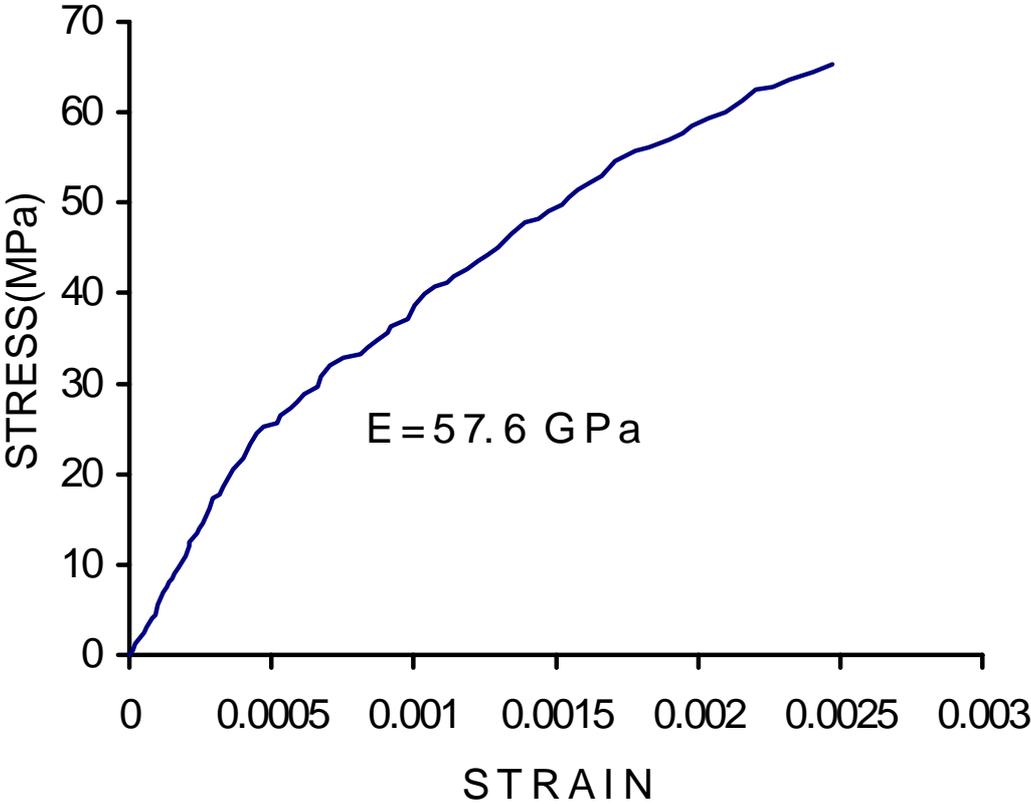


Fig. 1 Elevated Temperature Monotonic Loading Stress-Strain Relationship

On the other hand, the typical stress-strain relationships under the compression monotonic test at both room and elevated temperatures were linear up to failure, as shown typically in Fig. 2 for the elevated temperature. The maximum compressive stress at failure was 334 MPa, and the failure strain was 0.55%. The Young’s modulus of elasticity obtained from this test is 60.5 GPa. The comparison between the room and elevated temperatures tests showed that the failure stress at room temperature was about 18% lower than that at the elevated temperature, and the failure strain at room temperature is about 21% lower than that at the elevated temperature.

Fatigue Tests

Figure 3 shows the S-N curves from all fatigue tests combined on one graph for comparison purposes. These curves include the monotonic tensile strengths at room and elevated temperatures as data points corresponding to one cycle to failure. The S-N curves clearly

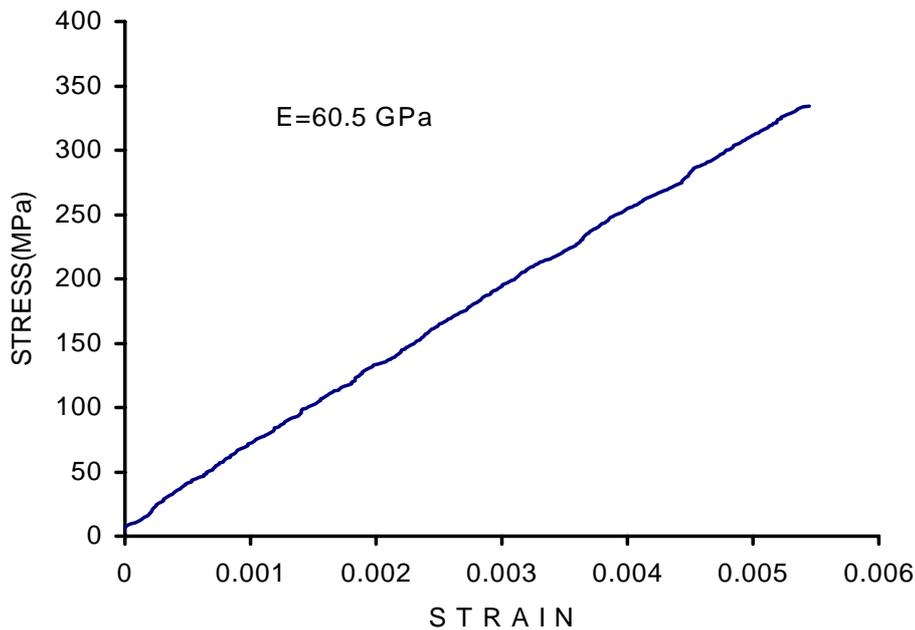


Fig. 2 Elevated Temperature Monotonic Compression Loading Stress-Strain Relationship

illustrate the dependence of fatigue life on the maximum applied stress level. In the room temperature T-T fatigue curve, there is a sharp decrease in the number of cycles to failure at stress levels above 54 MPa (78% of failure stress), and the number of cycles to failure becomes almost independent of stress levels below 50 MPa (72% of failure stress). The sharp decrease in the number of cycles to failure at high stress levels is also evident in the T-T elevated temperature fatigue test. The fatigue life decreases noticeably up to stress levels of 36 MPa (55% of failure stress) and then gradually until 24 MPa (38% of failure stress). The fatigue strengths for 40,000 cycles at room and elevated temperatures under tension-tension fatigue are 48 MPa and 24 MPa, respectively.

In the room temperature T-C tests, the sharp decrease in fatigue life is evident up to about 45 MPa (64% of failure stress). At stress levels below 45 MPa, the fatigue life increases as the stress level drops down to 36 MPa below which the fatigue life becomes very long. In the elevated temperature T-C fatigue tests, the sharp decrease in fatigue life is noticed at stress levels above 30 MPa (46% of failure stress). Below 30 MPa stress level the fatigue life increases with lowering of stress until 25 MPa stress level, below which the fatigue life becomes again very long.

At room temperature, there is a significant difference between T-T and T-C fatigue life of the material under investigation. The number of cycles to failure at a given applied stress level in T-T is higher than in T-C, i. e. the material can withstand higher maximum stress levels in T-T than in T-C cycling condition. At the elevated temperature, it can be seen that there is

practically no difference between T-T and T-C fatigue tests, and the material behaved almost exactly the same. In both T-T and T-C fatigue tests, the material can withstand higher maximum stress levels at room temperature than at the elevated temperature, indicating that more damage occurred in the material due to the elevated temperature.

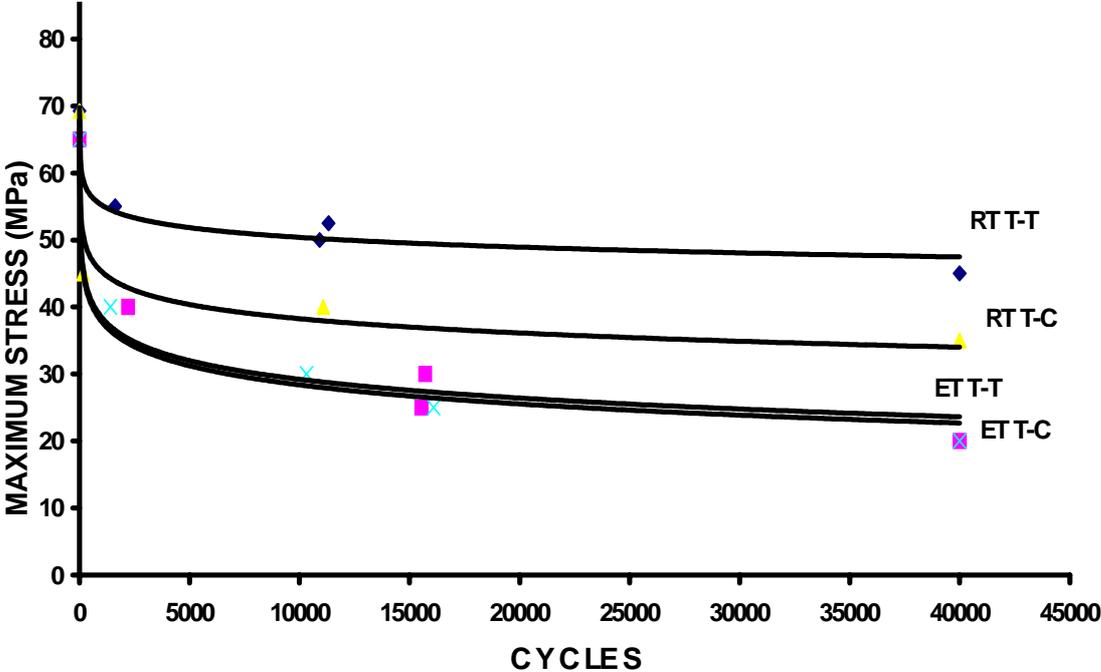


Fig. 3 Fatigue Life Diagram for all Loading Conditions

The fatigue strength for 40,000 cycles at room temperature for the T-T fatigue tests is about 48 MPa compared to 36 MPa for the T-C fatigue tests. The corresponding value at the elevated temperature is about 24 MPa for both T-T and T-C fatigue tests. So, the fatigue strength for 40,000 cycles is highest for the room temperature T-T fatigue tests, followed by the room temperature T-C fatigue tests, and lowest for the elevated temperature T-T and T-C tests.

Maximum and minimum strains were monitored during fatigue tests. There was practically no variation in both minimum and maximum strain values from the onset of cycling until failure in the room temperature tension-tension fatigue tests. This suggested that either no or a very little damage occurred to the material in this case. Figure 4 shows the typical variation of minimum and maximum strains in the elevated temperature tension-tension fatigue test. In the shown case, the minimum and maximum strains started at about 0.03% and 0.06%, respectively; and then both of these strains increased as the number of cycles progressed, up to failure. However, the difference between the minimum and maximum strains remained

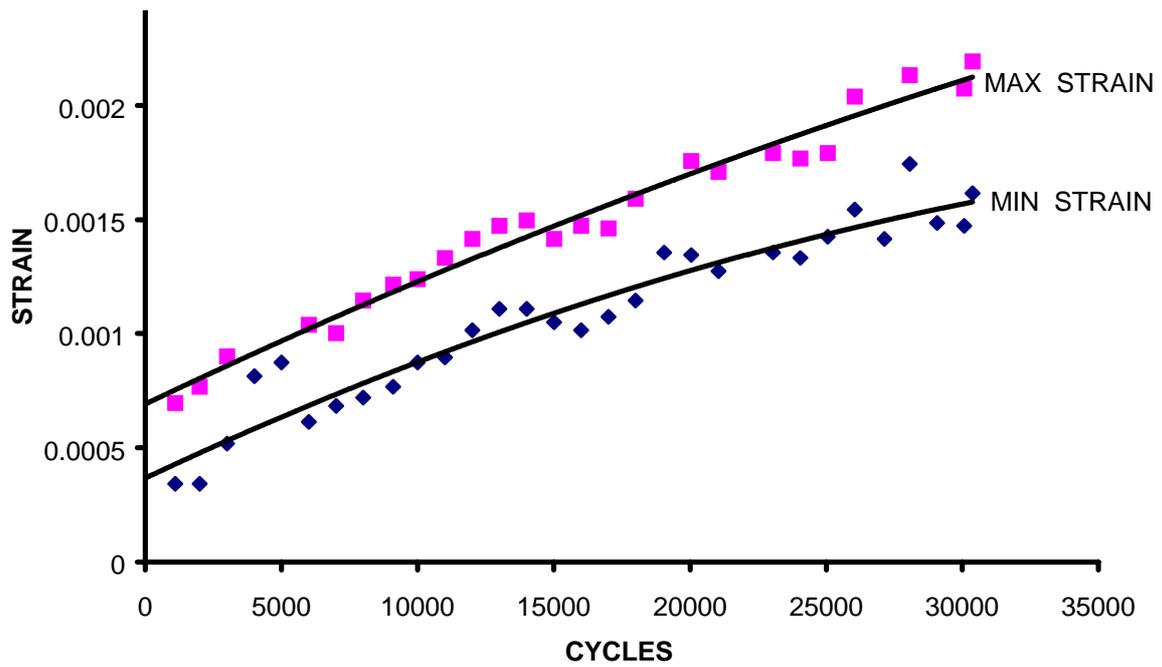


Fig. 4 Elevated Temp. Tension-Tension Fatigue Test Min. and Max. Strains; 20 MPa

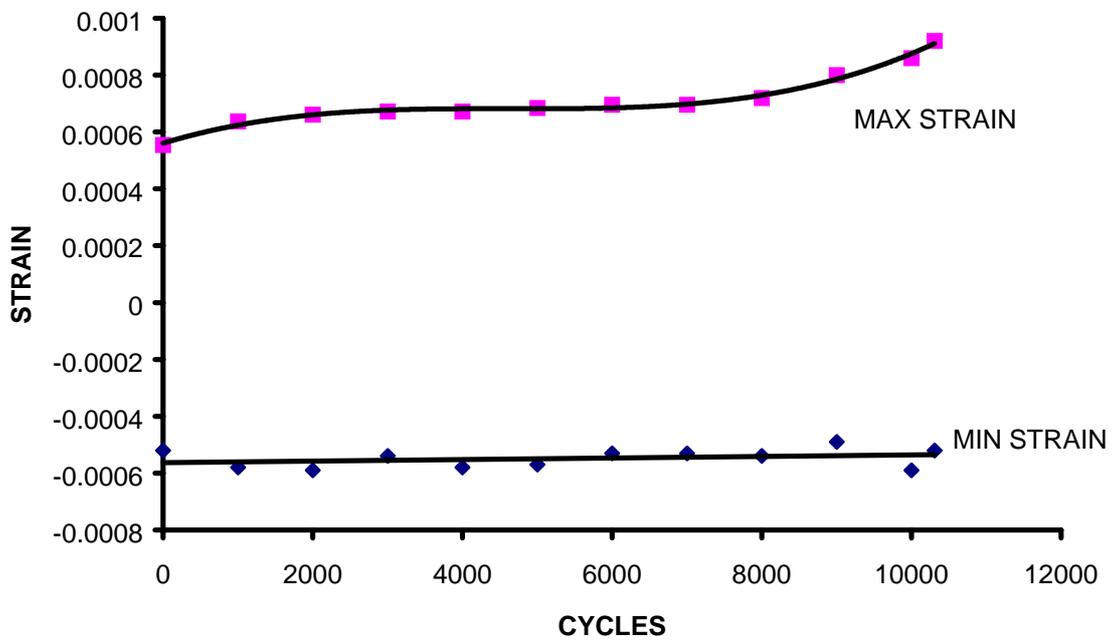


Fig. 5 Elevated Temp. Tension-Compression Fatigue Test Min. and Max. Strain; 30 MPa

almost constant until near failure, where the difference increased slightly. This accumulation of strain indicates that the deformation mechanism seen by the specimen during most of cycling is predominantly from creep deformation. More stiffness reduction was also seen at higher applied stress levels in the elevated temperature tension-tension fatigue tests where a difference between minimum and maximum strain was noticed along with their increase with the cycling.

In the room temperature tension-compression fatigue tests, the maximum and minimum strains remained almost constant from the beginning of the test up to failure. This indicates that minimum damage occurred during the cycling except near the failure. Figure 5 shows the typical variation in minimum and maximum strains during an elevated temperature tension-compression fatigue test. As shown, the minimum strain did not vary much but remained almost constant. The maximum strain also remained constant for the most of the fatigue life (about 75% of the fatigue life in this test), after which it increased with number of cycles until failure. This shows that the fatigue damage occurred in this case, but no creep was present. So, the elevated temperature had no significant effect on the strain behavior of the material under investigation when it was subjected to tension-compression fatigue loads. On the other hand, a noticeable difference was seen in the strain behavior of the material when it was subjected to tension-tension fatigue loads at room and elevated temperatures. Under room temperature tension-tension fatigue condition, both minimum and maximum strains remained almost constant up to failure; but these strains, under elevated temperature, increased at the same rate with the cycling with the difference between them remaining almost constant, indicating that creep occurred in elevated temperature tension-tension fatigue loading condition.

SUMMARY

This study investigated the monotonic tension and compression, and the tension-tension and tension-compression fatigue behavior of a 2-D woven ceramics matrix composite at room temperature and at 760⁰ C. The ultimate strength was lower at the elevated temperature than that in the room temperature under monotonic tensile loading, and it was higher in the compression monotonic loading than its counterpart in the tensile loading. There was no effect on the Young's modulus due to temperature change and loading mode. The tested material had higher fatigue life in tension-tension than in tension-compression at a given maximum stress level at room temperature, but it had the same fatigue life at the elevated temperature under tension-tension and under tension-compression fatigue loading.

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REFERENCES

1. Mall, S. and Kim, R. Y., "Failure Mechanisms in Laminates of Silicon Carbide/Calcium Alumino-Silicate Ceramic Composite," *Composites*, Vol. 23, No. 4, July 1992, pp. 215-222.
2. Opalski, F.A. and Mall, S., "Tension-Compression Fatigue Behavior of a Silicon Carbide Calcium-Alumino-Silicate Ceramic Matrix Composites," *Journal of Reinforced Plastics and Composites*, Vol. 13, May 1994, pp. 420-438.
3. Rodrigues, A., Rosa, G. A. and Steen, M., "Fatigue Behavior of A Ceramic Matrix Composite, 2D C_{fiber}/SiC_{matrix}," *The American Ceramic Society*, Vol. 57, 1995, pp. 351-356.
4. Shuler, S. F., Holmes, J. and Wu, X., "Influence of Loading Frequency on the Room-Temperature Fatigue of a Carbon-Fiber/SiC-Matrix Composite," *Journal of American Ceramic Society* Vol. 76 No. 9, 1993, pp. 2327-2336.
3. Camus, G., Guillaumat, L. and Baste, S., "Development of Damage in a 2D woven C/SiC Composite under Mechanical Loading: I. Mechanical Characterization," *Composites Science and Technology*, Vol. 56, 1996, pp. 1363-1372.
4. Campbel, S. S. and Gonczy, S. T., "In-Situ Formation of Boron Nitride Interfaces on Nextel 312TM/ BlackGlasTM Continuous Ceramic Fiber I: Nitriding Process and BlackGlasTM Ceramic Matrix Composite Properties," *Ceramic Engineering and Science Proceedings*, Vol. 15, No. 4, 1994, pp. 327-336.
5. Campbel, S. S. and Gonczy, S. T., "In-Situ Formation of Boron Nitride Interfaces on Nextel 312TM/ BlackGlasTM Continuous Ceramic Fiber I: Oxidation of BlackGlasTM Ceramic Matrix Composite," *Ceramic Engineering and Science Proceedings*, Vol. 15, No. 4, 1994, pp. 337-343.
6. Vaidyanathan, K. R., Cannon, W. R., Danforth, S., Tobin, A. G. and Holmes, J., "Effect of Oxidation on the Mechanical Properties of Nextel 312TM/BN/ BlackGlasTM," *Materials Research Society Symposium Proceedings*, Vol. 365, 1995, pp. 429-434.
7. Vaidyanathan, K. R., Sankar, J. and Kelkar, A. D., "Mechanical Properties of Nextel 312TM Fiber-reinforced SiC Matrix Composites in Tension," *Ceramic Engineering and Science Proceedings*, Vol. 15, No. 4, 1994, pp. 251-261.
8. Nijhad, M. N. G. and Bayliss, J. K., "Processing and Performance of Continuous Fiber Ceramic Composites Using Vacuum Assisted Resin Transfer Molding and BlackGlasTM Pre ceramic Polymer Pyrolysis," *Ceramic Engineering and Science Proceedings*, Vol. 18, No. 3, 1997, pp. 391-399.