EFFECT OF ENVIRONMENT ON FATIGUE BEHAVIOR OF A NICALON^{TM}/SI-N-C CERAMIC MATRIX COMPOSITE

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ABSTRACT

The effect of environmental exposure on the fatigue life of Nicalon^{TM} /Si-N-C composite was investigated in this study. Test specimens with arrays of 1.8 mm diameter holes and two different open areas, 25 and 35%, were machined. Three environmental conditions were studied: 1) continuous fatigue cycling in air, 2) fatigue cycling in air alternating with humidity exposure, and 3) fatigue cycling in air alternating with exposure to a salt-fog environment. All fatigue testing on specimens with holes was performed with a load ratio, $R = 0.05$, and at a temperature of 910 °C. In general, fatigue lives were shortest for specimens subjected to salt-fog exposure and longest for specimens subjected to continuous fatigue cycling in air. The fatigue data generated on the specimens with holes were compared with fatigue data generated in air on specimens with no holes. Fatigue strength reduction factors for different environmental conditions and open areas investigated in the study were calculated for the Nicalon^{TM} /Si-N-C composite.

INTRODUCTION

A Nicalon^{TM} fiber-reinforced, Si-N-C matrix composite (SiC/Si-N-C) was proposed for application in the exhaust nozzle as acoustic liner's tile material for the High Speed Civil Transport gas turbine engine [1]. The acoustic liner tile design included hexagonal arrays of small holes in the composite material. In this application, the composite tiles would be exposed to various environments, including humidity and salt-containing environments. Estimation of the durability of the composite material would require determination of the fatigue life reduction due to the holes and exposure to different types of environments.

The objective of this study was to characterize the fatigue behavior of Nicalon^{TM} /Si-N-C composite with holes in different environmental conditions and to quantify the fatigue strength reduction, if any. Initially, tensile and fatigue tests were conducted in air at 910°C on specimens with no holes to establish baseline properties for the composite. Subsequently, tensile and fatigue tests were conducted at 910°C on specimens with holes in air. In addition, fatigue tests were conducted at the same temperature in air alternating with a) humidity and b) salt-fog exposures.

The fatigue results generated and the calculated fatigue strength reduction (FSR) factors are reported.

MATERIAL AND SPECIMENS

The SiC/Si-N-C composite material was manufactured by the polymer impregnation and pyrolysis (PIP) method [2]. Nicalon^{TM} fibers were woven in an eight harness satin weave configuration and the fiber mats were assembled in quasi-isotropic [0/$\pm$45/90]s lay up. A proprietary interface material was applied by the manufacturer (Dow Corning Corporation) of the composite. Typically, C and BN are used as interface materials for similar ceramic matrix composites [3-5]. The composite preform was first impregnated with a ceramic precursor polymer and then it was pyrolized to convert the polymer into ceramic. Several PIP cycles are usually employed to decrease the void content thereby increasing the density of the composite [2]. Test specimens with no holes were machined from the SiC/Si-N-C composite plates by diamond grinding (Fig. 1). Similar fatigue specimens were used in previous investigations for stress-rupture characterization [6] and high-cycle fatigue characterization [7] of other types of ceramic matrix composites. In the case of test specimens with holes, test specimen blanks were initially machined by diamond grinding and holes with a nominal diameter of 1.8 mm were drilled later by an ultrasonic method. Additional PIP cycles were performed after drilling the holes to seal the edges of the holes. The percent open area (POA) in the test section and the hole-pattern dictated the widths of the 25 and 35 POA test specimens (Fig. 1). All test specimens had a nominal thickness of 2.8 mm.

EXPERIMENTAL DETAILS

All tensile and fatigue (R=0.05) tests were conducted at 910°C. Tensile tests were conducted in stroke-control, whereas fatigue tests were conducted in load-control. Typically, each test specimen was heated in a susceptor and the test temperature was controlled and monitored with two R-type thermocouples located inside the susceptor. Additional details on the test specimen heating technique are available in Ref. 6.

All the fatigue tests on specimens with no holes (0 POA) and a limited number of fatigue tests on the 25 and 35 POA specimens were conducted at 0.33 Hz in air. A majority of the fatigue tests on the 25 and 35 POA specimens were performed at 1 Hz in air, in air alternating with humidity exposure, and in air alternating with exposure to a salt-fog environment. In all the fatigue tests, failure was defined as separation of the test specimen into two pieces. For tests conducted in air, if a specimen did not fail by 10^6 cycles it was considered a runout. Fatigue tests conducted in air alternating with a) humidity and b) salt-fog exposures on 25 and 35 POA specimens were interrupted fatigue tests. In the case of specimens exposed to humidity, initially the test specimen was loaded in the test rig and cycled in air at 910°C for 10,000 cycles and then it was removed from the test rig and placed in a humidity chamber (maintained at 35°C and 95% relative humidity) for a period of 16 hours. This sequence was repeated on the test specimen until it failed. A similar approach was followed for the specimens exposed to salt-fog. The chamber was maintained at 32°C and 95-98% relative humidity for salt-fog exposure. A solution of 0.05 wt% NaCl was used to generate the salt-fog in the chamber. For tests conducted in air/humidity and air/salt-fog the runout life was defined as 340,000 cycles. In the 25 and 35 POA specimens final failure typically occurred across the hole pattern essentially by linking up the existing holes.

RESULTS

Tensile Behavior

Duplicate tensile tests were conducted on the 0, 25 and 35 POA test specimens. The average values of tensile properties obtained from the 0 POA (no holes) test specimens were as follows: Elastic modulus, 104 GPa; tensile strength, 222 MPa; and yield strength (0.05% offset), 130 MPa.

The average values of the tensile strengths obtained from the 25 and 35 POA were 201 and 163 MPa, respectively. A comparison of the average tensile strengths (calculated on the basis of net section area for 25 and 35 POA specimens) is shown in Fig. 2. The error bar for 0 POA specimens indicates the range of the data. No error bars are visible for 25 and 35 POA specimens because in both cases the duplicate tests yielded nearly identical values. As expected, net section tensile strength decreased as POA increased due to loss of integrity of the composite resulting from the introduction of the holes.

Fatigue Behavior

Fatigue data generated on the 0 POA (no holes) specimens in air at 0.33 Hz are shown in Fig. 3. Note the scatter exhibited by the baseline fatigue data. A power-law type life relationship based on maximum stress (Eq. 1) was used to characterize the baseline fatigue behavior.

$$\sigma_{max} = B(N_f)^b \tag{1}$$

The values of coefficient, B and exponent, b determined with least squares analysis were 612 MPa and –0.149, respectively, for the baseline fatigue data.

Fatigue data generated on 25 and 35 POA specimens under three different environmental conditions are shown in Figs. 4 and 5, respectively. Once again, net cross-sectional areas were used to calculate the stresses in these specimens. In these figures, filled symbols denote fatigue tests conducted at 0.33 Hz and open symbols denote tests conducted at 1 Hz. Data symbols with an arrow indicate runout tests. All the fatigue data generated on 25 and 35 POA test specimens were lower than the baseline fatigue data on 0 POA specimens. No significant differences were observed in the fatigue lives of the specimens tested in air at 0.33 Hz and 1 Hz for 25 and 35 POA specimens. As a result, for subsequent determination of the fatigue life relationships the data generated at these two frequencies were combined. In general, scatter in the fatigue data for 35 POA specimens, was greater than that exhibited by the 25 POA specimens. Potential reason for this scatter is discussed later in the paper. In the case of 25 POA specimens, salt-fog was more detrimental to fatigue life than humidity and both were more damaging to the composite than laboratory air environment. Such clear trends were not observed in the 35 POA fatigue data. In this case, the additional damage caused by both salt-fog and humidity conditions was about the same compared to the laboratory air environment.

Fatigue life relationships (Eq. 1) were computed with least squares analyses for the three environmental conditions for the 25 and 35 POA data. The values of coefficients and exponents are listed in Table I. In computing these life relationships, all the runout data were omitted from the analyses. The fatigue life relationships together with the fatigue data are depicted in Figs. 6 to 8 for the three environmental conditions investigated.

Table I. Computed constants for the fatigue life relationships of 25 and 35 POA specimens

Specimen Type	Environmental Condition	Coefficient, B [MPa]	Exponent, b
25 POA	Air	367	-0.123
25 POA	Humidity	1470	-0.272
25 POA	Salt-fog	2910	-0.353
35 POA	Air	472	-0.167
35 POA	Humidity	2990	-0.370
35 POA	Salt-Fog	501	-0.192

For all the environmental conditions investigated, the 25 and 35 POA fatigue life relationships were considerably lower than the baseline 0 POA fatigue life relationship (Figs. 6 to 8). Moreover, the 35 POA fatigue life relationships were lower than the corresponding 25 POA life relationships both in air and humidity environments. In the case of salt-fog environmental condition, the 35 POA life relationship was lower than the 25 POA life relationship at high applied maximum stresses and a opposite trend was observed at lower applied maximum stresses. Similar life relationships were utilized to calculate the FSR factors later in the paper.

DISCUSSION

As expected, drilling holes in the SiC/Si-N-C composite reduced both the net section tensile strength and the fatigue strength. In general, larger reductions in net section tensile strength (Fig. 1) and fatigue strength (Figs. 6 to 8) were observed as the POA increased in the specimens. The observed reduction in the net section tensile strength, which accounts for the reduction in the cross sectional area of the composite due to holes, indicates existence of a stress concentration effect when holes are present. Moreover, the stress concentration effect appeared to increase as the POA of the specimens increased. This observation could be explained by the loss of integrity of the SiC/Si-N-C composite at higher POA. For example, a 35 POA specimen is likely contain a higher percentage of damaged and discontinuous fiber bundles in the test section than a 25 POA specimen. As a result, the 35 POA specimen is more likely to exhibit lower tensile strength and larger variation in fatigue durability than a 25 POA specimen.

In the present investigation, only a limited number of specimens with holes were tested in fatigue under each environmental condition. Therefore, in order to compare the influences of different environmental conditions on the fatigue durability of SiC/Si-N-C composite and to calculate the FSR factors for this material under various conditions, a common exponent, $b = -0.24$ was calculated by combining the 25 and 35 POA fatigue data generated under all the environmental conditions. The calculated value of $b = -0.24$, is nearly equal to the average of the values listed in Table I. The coefficients computed for SiC/Si-N-C composite with a common exponent of -0.24 are listed in Table II. By comparing the coefficients of the life relationships the following can be surmised: 1) Humidity and salt-fog are more damaging than air for both 25 and 35 POA specimens, 2) salt-fog is more damaging than humidity for the 25 POA specimens and vice versa for the 35 POA specimens.

Table II. Fatigue life relationships of 25 and 35 POA specimens with a common exponent

Specimen Type	Environmental Condition	Coefficient, B [MPa]	Exponent, b
25 POA	Air	1220	-0.24
25 POA	Humidity	1040	-0.24
25 POA	Salt-fog	925	-0.24
35 POA	Air	993	-0.24
35 POA	Humidity	741	-0.24
35 POA	Salt-Fog	827	-0.24

FSR factors were computed at fatigue lives of $N = 10^4$ and 10^5 cycles for the SiC/Si-N-C composite investigated by using Eq.1 for the 0 POA (no holes) as the baseline and the constants listed in Table II for the 25 and 35 POA specimens. The calculated FSR factors (Table III) are useful to determine the detrimental effect due to different POA values and environmental conditions for the SiC/Si-N-C composite. For example, with 25 POA in salt-fog, the maximum stress the composite can sustain to achieve a fatigue life of 10^5 cycles is about 53% of the corresponding value for 0 POA (no holes) in air. Similar FSR factors can be calculated at other cyclic lives for 25 and 35 POA values of the composite with the procedure presented in this paper.

SUMMARY

The fatigue behavior of a woven, Nicalon™ /Si-N-C composite containing arrays of holes with 25 and 35% open areas was investigated at 910° in air, humidity, and salt-fog type of environmental conditions. Fatigue database generated on specimens with no holes was used to establish the baseline behavior for the composite. As expected, presence of the holes reduced the fatigue strength of the composite and the extent of reduction in fatigue strength increased with the percentage of open area in the specimen. Both humidity and salt-fog were detrimental to the fatigue strength of the composite with holes. Fatigue strength reduction factors were calculated to account for the damaging effects of 1) open areas (holes) in the composite and 2) the environmental conditions.

Table III. Fatigue strength reduction factors for 25 and 35 POA specimens at two fatigue lives

Specimen Type	Environmental Condition	N = 10, 000 [Cycles]	N = 100, 000 [Cycles]
0 POA	Air	1.00	1.00
25 POA	Air	0.86	0.70
25 POA	Humidity	0.74	0.60
25 POA	Salt-fog	0.65	0.53
35 POA	Air	0.70	0.57
35 POA	Humidity	0.52	0.43
35 POA	Salt-Fog	0.59	0.47

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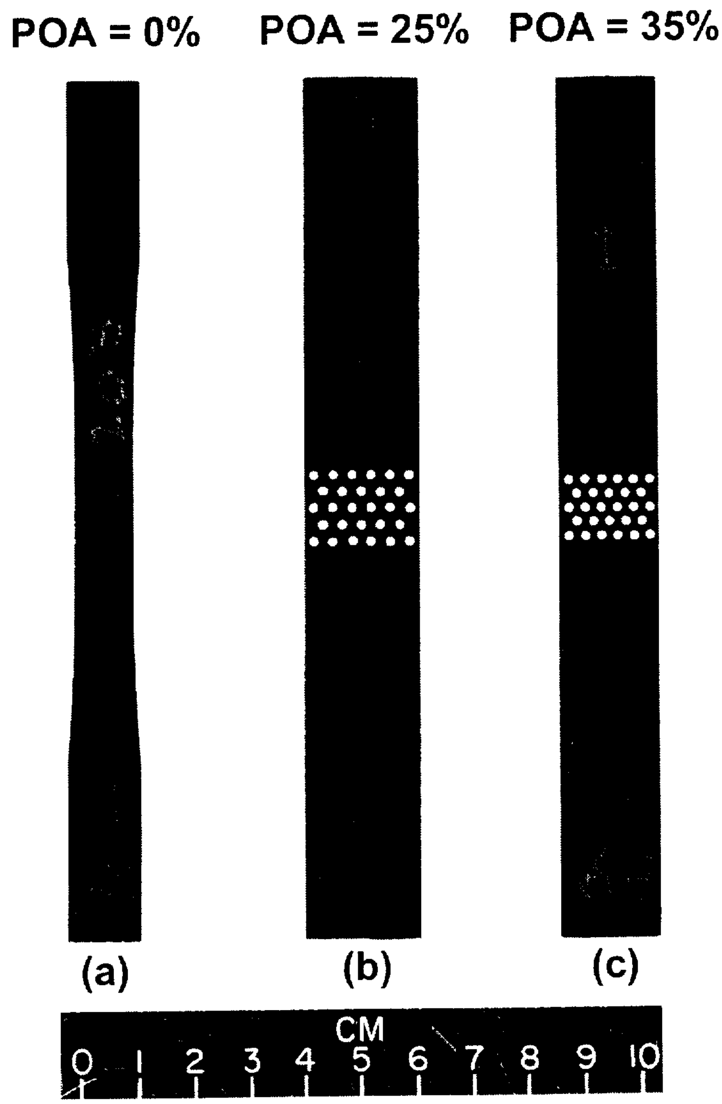

Figure 1: SiC/Si-N-C composite test specimens. (a) 0 (no holes), (b) 25, and (c) 35 POA.

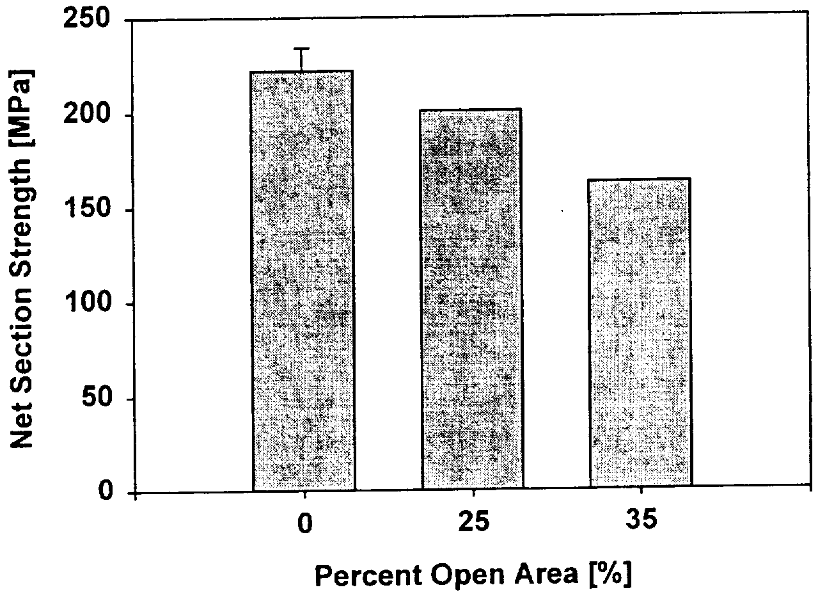

Figure 2: Comparison of average net section tensile strengths for 0, 25, and 35 POA specimens.

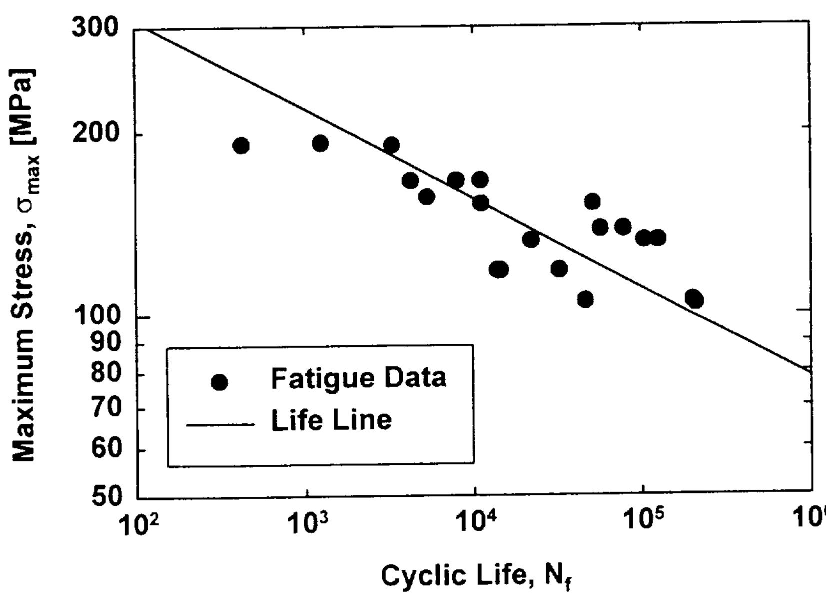

Figure 3: Fatigue Behavior of SiC/Si-N-C Composite in air at 910°C and 0.33 Hz.

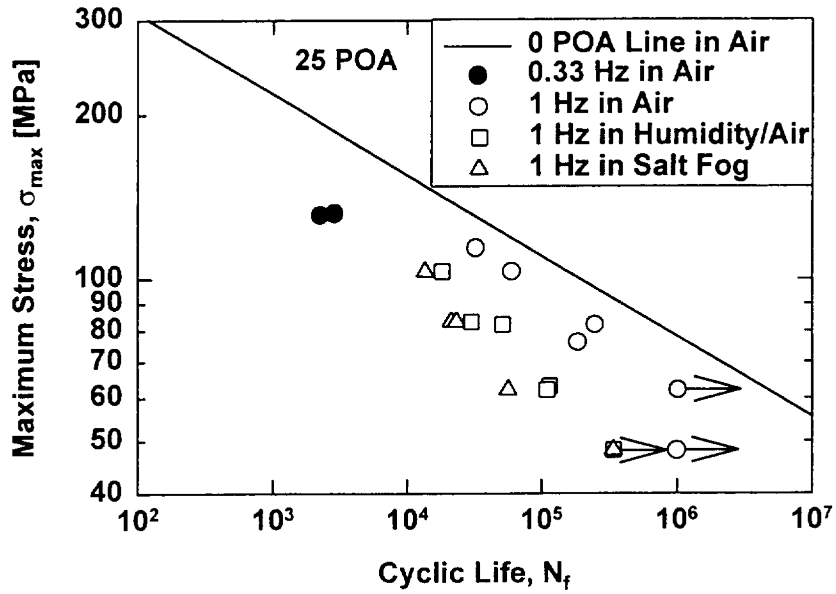

Figure 4: Fatigue data generated with 25 POA specimens under three environmental conditions.

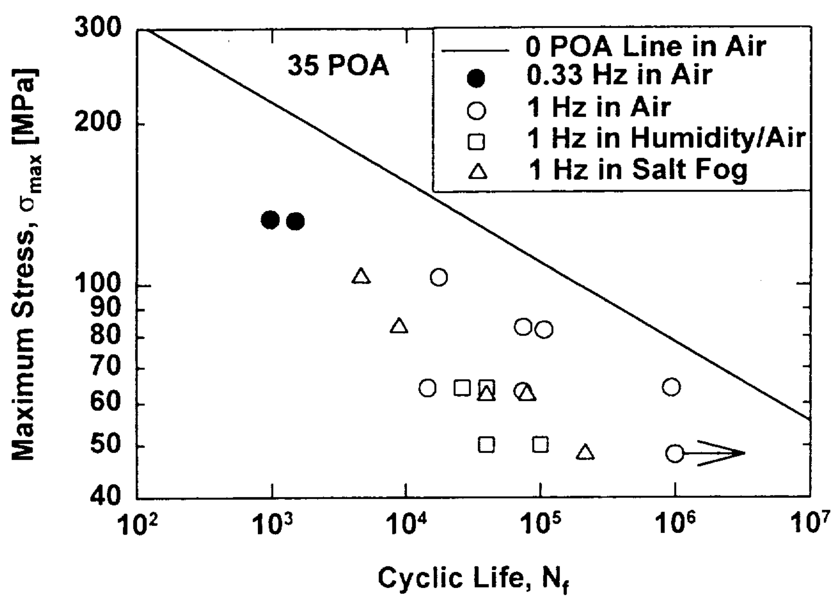

Figure 5: Fatigue data generated with 35 POA specimens under three environmental conditions.