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Frequency effects on fatigue behavior of Nextel720™/alumina at room temperature☆

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Abstract

Cycling frequency effects on the fatigue behavior of an oxide/oxide ceramic matrix composite, Nextel720™/alumina, were investigated. Tension–tension fatigue tests were conducted at three frequencies: 1, 100 and 900 Hz to establish stress versus cycles to failure (*S–N*) relationships. Cycles to failure at a stress level increased considerably with increase of the frequency from 100 to 900 Hz, but very little from 1 to 100 Hz. Fatigue behavior of Nextel720™/alumina appeared to be a combination of cycle-dependent and time-dependent phenomena. Surface temperature of specimens tested at 900 Hz increased considerably relative to that at 1 or 100 Hz (70 °C versus 5 °C increase). Damage mechanisms showed an evidence of local fiber/matrix interfacial bonding developed during cycling due to frictional heating at the highest frequency of 900 Hz. This was not observed at the two lower frequencies. This interfacial bonding may have caused an increase in fatigue life/strength of the tested CMC system at the highest frequency of 900 Hz.
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Keywords: Composites; Fatigue; Interfaces; Lifetime; Fracture; Cyclic frequency

1. Introduction

Continuous ceramic fiber-reinforced ceramic matrix composites (CMCs) are currently receiving a good deal of attention for long-term high-temperature applications in the next generation engines of aircrafts and spacecrafts due to the potential of offering increased high-temperature performance and environmental stability relative to metallic materials.[1–3] Since CMCs in these applications will be subjected to cyclic loading conditions, fatigue behavior needs to be characterized. Fatigue behavior is influenced by the cyclic frequency besides many other factors/parameters. Fatigue behavior could become complex in CMCs relative to homogeneous materials due to the presence of fiber/matrix interface. Holmes and Shuler observed that cyclic load ranging from 1 to 85 Hz frequencies caused a temperature increase in woven C/SiC CMC with increasing frequency due to the relative motion at fiber–fiber and fiber–matrix interfaces, and a reduction in cycles to failure.[4,5] A similar trend was observed in Nicalon/CAS-II CMC when cycled from 25 to 75 Hz.[6] Both of these CMCs had a relatively weak fiber/matrix interphase that caused extensive debonding between fiber–fiber and fiber–matrix interfaces during cyclic loading conditions. On the other hand, a Nicalon/SiCON CMC system with a stronger fiber–matrix interface showed no difference between 1 and 100 Hz fatigue life data.[7,8]

When combined with temperature, cyclic frequency can have a much more complex role in the fatigue performance of CMCs. In recent studies with woven C/SiC CMC, an increase in failure cycles was observed at a stress level when frequency increased from 0.1 to 375 Hz at 550 °C.[9] This trend was quite different at room temperature where cycles to failure decreased when frequency increased from 40 to 375 Hz but remained almost the same below 40 Hz.[10] Analysis of damage mechanisms showed

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that the oxidation of carbon fibers was a major difference between specimens tested at room and elevated temperatures. This caused an opposite trend in fatigue life dependence on the cyclic frequency.

Recently, several damage-tolerant oxide/oxide CMCs, including Nextel720^{TM} fibers reinforced alumina oxide matrix, have been studied for applications in high temperature (>1100 °C) environment.[11–14] A summary of mechanical properties and damage mechanisms of porous-matrix CMCs is given in a previous study.[15] However, there is no previous investigation involving the effects of high frequency on the fatigue behavior of oxide/oxide CMCs as far as the authors are aware, which is needed with respect to their applications in aeroengines. This study is a step in this direction and it involved the laboratory room temperature environment only.

2. Experiments

2.1. Materials and specimens

The oxide/oxide CMC, used in this study, was Nextel720^{TM}/alumina manufactured by COI Ceramics, San Diego, CA in a form of 2.8 mm thick plate. Composite composed of uncoated Nextel 720 fibers (manufactured by 3M) in 8HSW, 0°/90° woven layers, with a density of ∼2.78 g/cm^3 and fiber volume fraction of approximately 44%. Nextel720^{TM} fiber is a meta-stable mullite having the chemical composition of Al_2O_3: 85% (by weight) and SiO_2 of 15% (by weight). This mullite fabric preform was infiltrated with the matrix precursor of alumina by a sol–gel process. Nextel720^{TM}/alumina CMC was made by a vacuum bag process under low pressure and low temperature followed by a pressureless sintering technique. Representative micrographs of the untested as-received material are shown in Fig. 1. Fig. 1(a) shows 0° and 90° fiber tows as well as numerous matrix cracks. These were shrinkage cracks formed during processing due to mismatch of thermal expansion coefficient between matrix and fibers. Matrix porosity was ∼24% and such porosity level makes the matrix sufficiently weak and the composite damage tolerant. The porous nature of the matrix can be seen in Fig. 1(b).

The CMC plate was cut into dog bone-shaped specimens using a water jet. Test specimens for fatigue testing had thickness, width and gage length of 2.8, 10.2 and 30 mm, respectively, and total length of 150 mm for 1 Hz tests, and 2.8, 6.4 and 20 mm with total length of 64 mm for 100 and 900 Hz tests. Specimens for the monotonic tensile test had the same dimensions as that of 1 Hz tests. Fracture surfaces of failed specimens were gold-coated and examined using a scanning electron microscope (SEM).

2.2. Test details

All tests were conducted at room temperature in air under ambient laboratory environment. Monotonic tension tests were performed under the stroke control mode with a constant displacement rate of 0.05 mm/s. Strain in the monotonic tension tests was measured using an extensometer with 7.62 mm gage length. However, it could not be used during fatigue tests due to vibration and instability at cyclic frequency of 100 Hz or higher. All test operations and data collection were computer controlled. Fiberglass tabs were attached to the grip portion of each specimen to prevent sliding of the specimen.

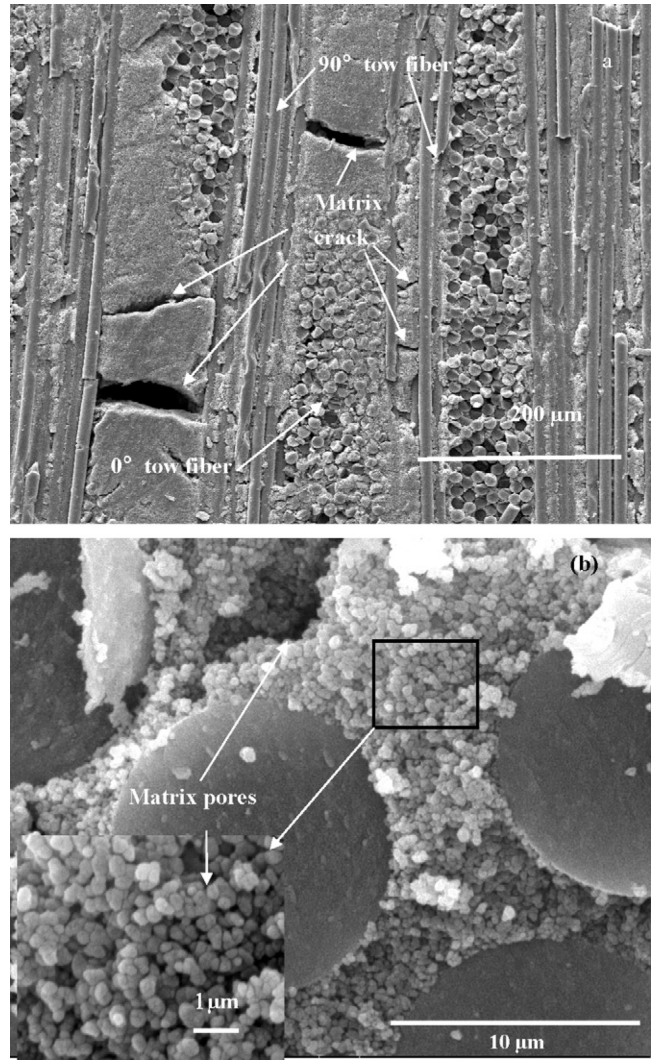

Fig. 1. Photomicrographs of cross-section of as-received Nextel720^{TM}/alumina: (a) fibers and matrix cracks and (b) porous matrix.

Three cyclic frequencies were used: 1, 100 and 900 Hz. Fatigue tests were run for the maximum of 10^5, 10^7 and 10^8 cycles at 1, 100 and 900 Hz, respectively, if the specimen did not fail. These were thus the run-out limits of the fatigue test in this study. Fatigue tests at 1 Hz were conducted on a standard servo-hydraulic test machine. Fatigue tests at 100 and 900 Hz were conducted on a servo-hydraulic high-frequency fatigue test machine ((25 kN, 1000 Hz High-Cycle/Frequency Fatigue Test System, MTS Corp.). All fatigue tests were run under the load control mode with a stress ratio (minimum load/maximum load) of 0.05. The temperature increase on the specimen surface during the fatigue test was monitored through an infrared (IR) camera.[10]

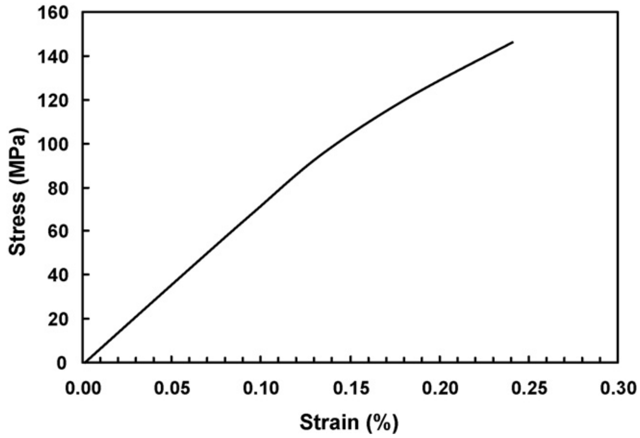

Fig. 2. Typical monotonic tensile stress–strain curve.

3. Results and discussion

3.1. Tension

A typical stress–strain curve from a monotonic tensile test at room temperature is shown in Fig. 2. This shows initially a linear region up to about 60–70 MPa, which represents the damage state before any appreciable amount of matrix crack was developed. The latter region of stress–strain curve shows a slightly decreasing slope, which indicates further increase of matrix cracks and internal flaws with increasing applied load. The average ultimate tensile strength (UTS), tensile failure strain and Young's modulus from three replicate tests were 145 MPa, 0.25%, and 70 GPa, respectively.

3.2. Fatigue

Applied maximum stress versus cycles to failure relationships (*S–N* curves) of the tested Nextel720^{TM}/alumina CMC for three frequencies of 1, 100 and 900 Hz are shown in Fig. 3. The average ultimate tensile strength is also indicated in this figure. Fig. 3 shows that there is, in general, a frequency effect in the tested composite. Cycles to failure at a stress level increased with increase in the cyclic frequency from 1 to 900 Hz. For example, cycles to failure at 1, 100 and 900 Hz were about 600, 6000 and greater than 10^8 at the maximum stress of 120 MPa, respectively. Further, it is to be noted that the slope of these *S–N* relationships is decreasing with increase of cyclic frequency from 1 to 100 Hz. In other words, the effect of frequency is diminishing at higher stress levels, and it is almost negligible at stress levels greater than ∼130 MPa between 1 and 100 Hz. On the other hand, the fatigue strengths at 10^7 cycles at 100 and 900 Hz are about 100 and 125 MPa, respectively. Overall, data in Fig. 3 indicate that cycles to failure increased considerable with increasing cyclic frequency from 100 to 900 Hz, but not much between 1 and 100 Hz.

Frequency effects of the tested CMC system can be looked into also by plotting the test data as a function of failure time (i.e. *S–T* relationship) as shown in Fig. 4. If cycles to failure had been totally independent of frequency (i.e. cycle dependent), the three lines in Fig. 4 should have been parallel and the distance between them would have been proportional to the difference in applied frequency. This is not the case in the present system. This again shows that cycling frequency had an effect on the fatigue life. In general, time to failure under fatigue condition of Nextel720^{TM}/alumina CMC system appears to increase with increasing frequency from 1 to 900 Hz. Overall, if the results from Figs. 3 and 4 are considered together, the fatigue behavior of Nextel720^{TM}/alumina CMC system appears to be a combination of cycle-dependent and time-dependent phenomena.

For comparison, fatigue data of C/SiC CMC tested at 4, 40 and 375 Hz from a previous study[10] are plotted along with the present study's data of Nextel720^{TM}/alumina CMC in Fig. 5. This comparison is shown after normalizing the applied maximum fatigue stress by the ultimate tensile strength of each material. Fig. 5 shows the almost opposite trend of frequency on cycles to failure, i.e. cycles to failure decreases with increasing cyclic frequency for C/SiC while it increases with increasing

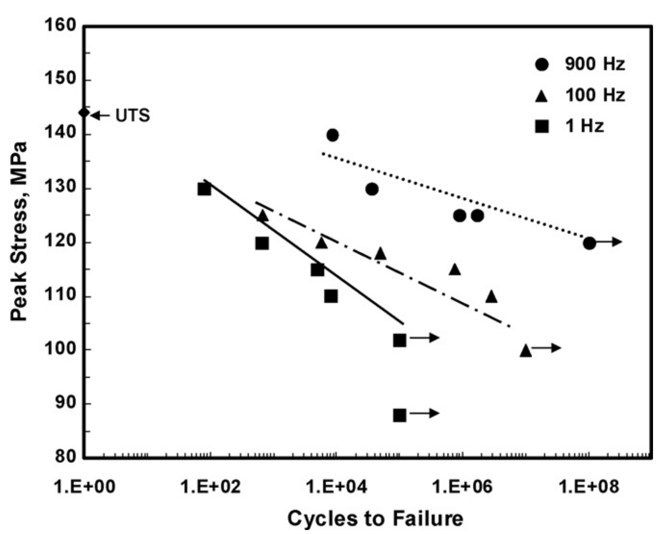

Fig. 3. Stress vs. cycles to failure (*S–N*) relationships of Nextel720^{TM}/alumina at frequencies of 1, 100, and 900 Hz.

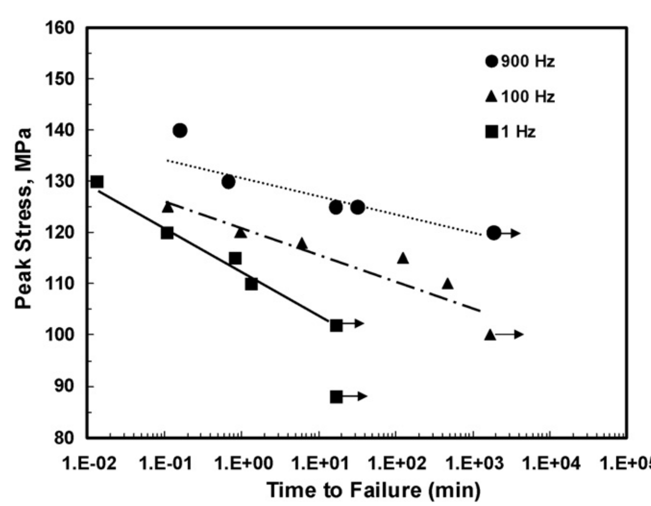

Fig. 4. Stress vs. time to failure (*S–T*) relationships of Nextel720^{TM}/alumina at frequencies of 1, 100 and 900 Hz.

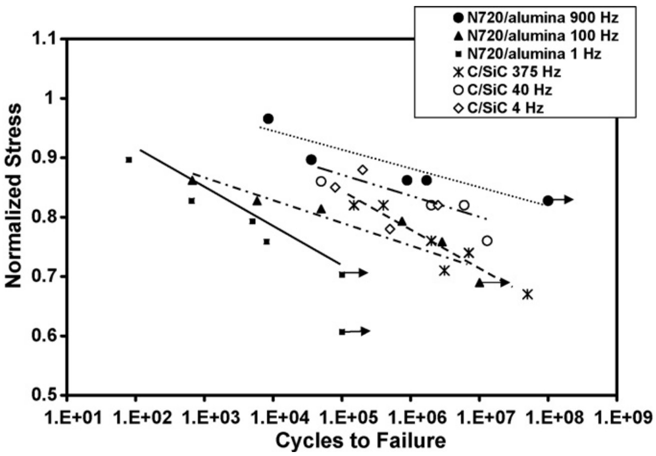

Fig. 5. Normalized stress vs. cycles to failure relationships of Nextel720TM/alumina at 1, 100, and 900 Hz and C/SiC tested at 4, 40 and 375 Hz[10] at room temperature.

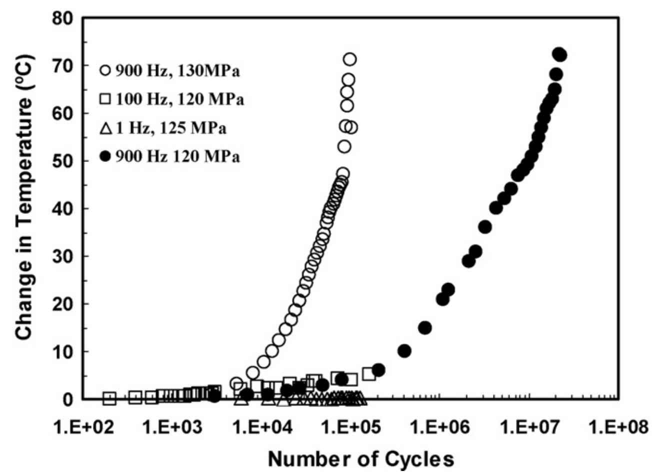

Fig. 6. Specimen surface temperature change vs. number of cycles.

cyclic frequency for Nextel720TM/alumina. However, there are some differences, i.e. this effect is not present or relatively small at the low frequencies.

Mechanisms responsible for the reduction in fatigue life due to increase in frequency for C/SiC were investigated in the previous study.[10] Specimen surface temperature increased during fatigue due to the internal heat generation from sliding friction between constituents of the composite. The increase was directly related to frequency. There was a difference in the interfacial damage of fibers between 4 or 40 and 375 Hz. There was clear evidence of oxidation on fiber surfaces which occurred during fatigue at the highest frequency of 375 Hz which possibly occurred due to the higher temperature inside the specimen. This oxidation caused weakening of interfacial strength resulting in reduction of cycles to failure at 375 Hz. It should be mentioned that the increase in specimen surface temperature was in the range 100–170 °C at 375 Hz while it was less than 20 °C at 4 or 40 Hz in the C/SiC CMC.[10] Therefore, mechanisms for the frequency effects in the present CMC system, Nextel720TM/alumina were investigated including the temperature change due to cyclic frequency. These are discussed next.

3.3. Temperature

An increase in temperature of the specimen/component is expected when subjected to fatigue at high frequency. This is especially true in materials with internal damage which can be present initially or could develop during the application of fatigue load. Fig. 6 shows typical specimen surface temperature changes as a function of number of applied cycles for the three test frequencies: 1, 100 and 900 Hz. There was an increase in temperature on the specimen surface during the fatigue in the present CMC system, which has also been observed in other CMC systems in the previous studies.[4,6,10] Increase in temperature at 1 Hz with applied stress of 125 MPa and at 100 Hz with applied stress of 115 MPa was almost negligible (i.e. about 5 °C) during the fatigue test. However, the surface temperature at 900 Hz increased with accumulating fatigue cycles, and this increase was more than 70 °C before failure. The surface temperature increase at 900 Hz for the two stress levels of 120 and 130 MPa show slightly different trends. The rate of temperature increase at 130 MPa was faster than at 120 MPa, which indicates that there was faster damage growth at higher stress level as expected. In general, temperature increase in CMCs during fatigue is attributed to the internal friction caused by the relative motion between constituents of the composite.[10] Since the frictional heating in the composite is generally dependent on the matrix crack density, and the number and length of debonded fiber/matrix interfaces, the present results of temperature evolution suggest that the tested system had similar damage and damage evolution during cycling at the two lower frequencies of 1 and 100 Hz while it was different at the highest frequency of 900 Hz.

3.4. Damage mechanisms

Fig. 7 shows a typical fractured portion of specimen which was similar in all three frequencies. It shows a brushy appearance of failure which is typical feature of brittle fracture commonly seen with woven (fabric) ceramic matrix composites. However, there were differences in the damage mechanisms between

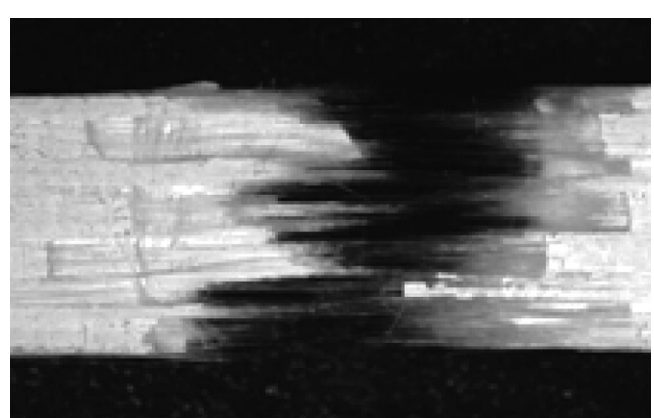

Fig. 7. Typical fractured specimen.

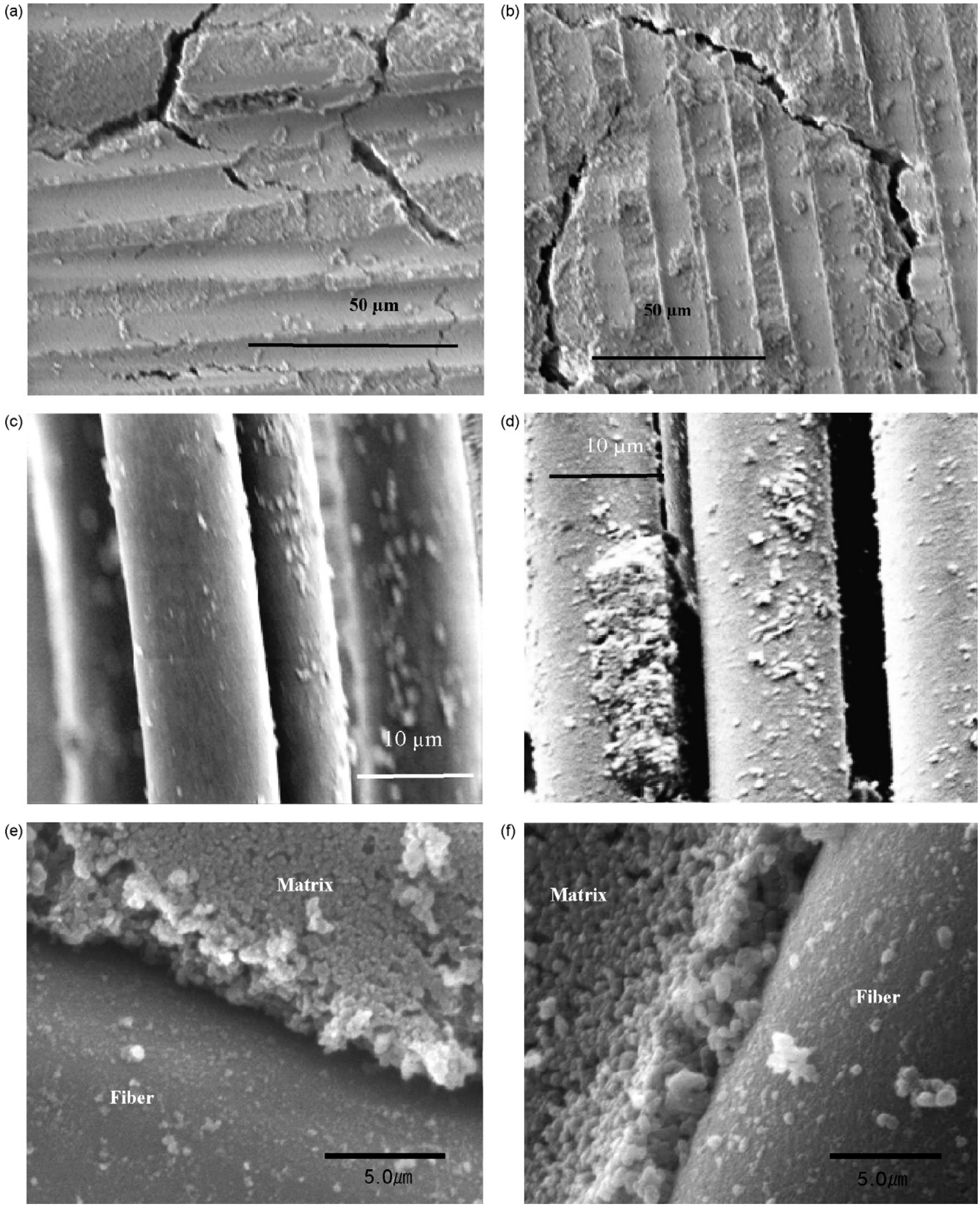

Fig. 8. Photomicrographs of Nextel720^{TM}/alumina at two frequencies: (a) matrix region for 1 Hz, (b) matrix region for 900 Hz, (c) fiber region for 1 Hz, (d) fiber region for 900 Hz, (e) interface between fiber and matrix for 1 Hz and (f) interface between fiber and matrix for 900 Hz.

specimens tested at 900 Hz and 1 or 100 Hz at the microscopic levels in contrast to the macro-level, shown in Fig. 7. Microphotographs of fractured surfaces for composites tested at the lowest and highest frequencies, 1 and 900 Hz, are compared in Fig. 8. Similar photomicrographs from 100 Hz tests are not shown because they showed similar features to those of 1 Hz tests. Fig. 8 shows that there were distinctive microstructural features on the fracture surface, which can be attributed to frequency effects. Fig. 8(a) and (b) shows a typical comparison between fracture surfaces in the matrix region of specimens tested at 1 and 900 Hz, respectively. This comparison showed a relatively smooth matrix region with little debris in the case of 1 Hz tests while 900 Hz tests showed relatively rough fracture surfaces with more attached debris.

The present oxide/oxide CMC system had low fiber/matrix interfacial strength. This was obvious at the low frequencies of 1 or 100 Hz as shown in Fig. 8(a). This is also evident on the fibers which were separated from the matrix as shown in Fig. 8(c). Fibers from specimen tested at the low frequencies of 1 and 100 Hz had relatively smooth surface and they were separated individually in clean manner, Fig. 8(c). On the other hand, fiber surfaces were relatively rough with attached matrix debris in the case of the high frequency of 900 Hz, Fig. 8(d). Therefore, some mechanism responsible for the enhancement of fatigue life/strength at high frequency possibly developed during the cycling in this case. Fig. 8(e) and (f) shows photomicrographs of the fiber/matrix region where fiber and matrix are still together from specimens tested at 1 and 900 Hz, respectively. The fiber in this region, in a specimen tested at the low frequency of 1 Hz, was relatively smooth and fibers and matrix were cleanly separated from each other. These are typical features of a weak fiber/matrix interface. On the other hand, this region in high frequency samples showed some local bonding of the fiber/matrix interface. This suggests that interfacial bonding strength was greater in the high frequency samples than in the lower frequencies tests of 1 and 100 Hz. This bonding between matrix and fiber must have developed during cycling at the high frequency of 900 Hz.

One possible explanation for these aforementioned differences (i.e. rougher fiber and matrix surfaces along with some local fiber/matrix interfacial bonding in 900 Hz samples) is that there was relatively faster internal sliding between fibers and matrix or among fibers at 900 Hz, which was also evident from the surface temperature measurements. This could have generated much higher temperature inside the material especially in the sliding contact regions at 900 Hz. Halbig et al. have suggested that microcracks in C/SiC CMC system could generate internal temperature close to 1100 °C due to the internal friction.[16] The intense local increase in temperature possibly caused some reaction in the fiber/matrix interfacial region. Wannaparhun et al. have reported a possibility of interfacial reaction of a Nextel720™/alumina composite in water vapor at 1100 °C using X-ray photoelectron spectroscopy.[17]

To investigate the possibility of an interfacial phenomenon, the Nextel720™/alumina composite was also tested at 1200 °C at a frequency of 1 Hz and compared with the samples tested at room temperature with the same frequency. Fig. 9 shows the fracture surfaces of the composite tested at 1200 °C. This figure again shows a relatively rough fracture surface in both matrix and fiber regions with more attached debris, similar features to those seen in 900 Hz tests at room temperature, Fig. 8(b), (d) and (f). In addition, several fiber surfaces in specimens tested at 1200 °C also showed local bonding due to some reaction between matrix and fibers at the elevated temperature, Fig. 9(b), a similar feature present in 900 Hz tests at room temperature. It is also interesting to note that the fatigue run-out limit for 10^5 cycles at 1200 °C and 1 Hz was about 78% of the ultimate tensile strength, which was greater than its counterpart at room temperature (∼72%). Thus it appears that high cyclic frequency and high temperature have similar effects in the Nextel720™/alumina.

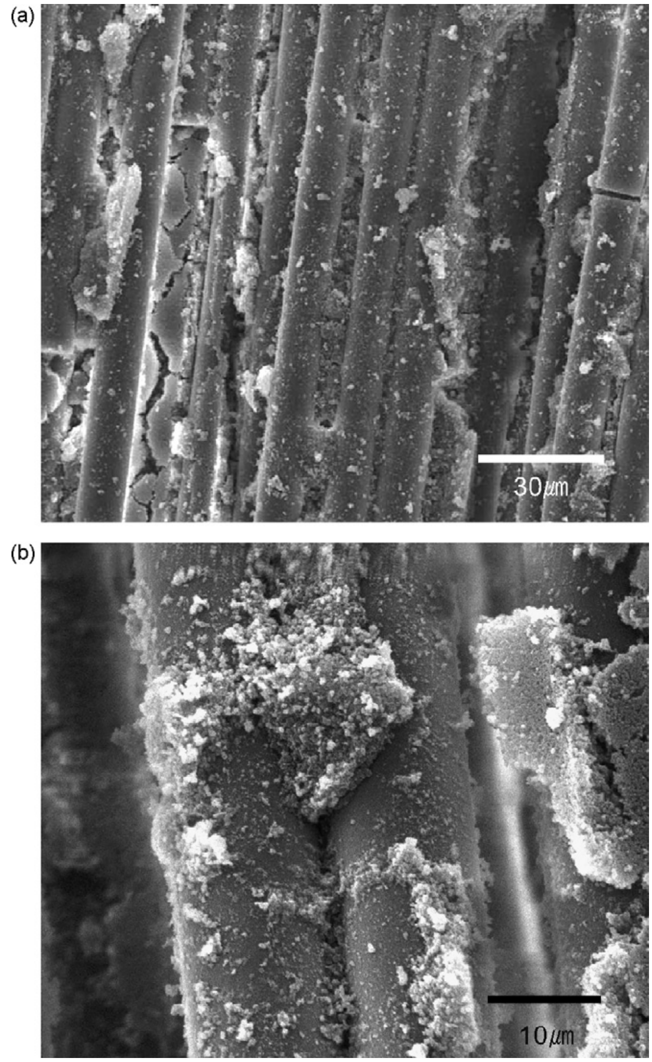

Fig. 9. Photomicrographs of Nextel720™/alumina tested at 1200 °C and 1 Hz: (a) fracture surface and (b) fiber region.

Thus damage and failure mechanisms in the Nextel720™/alumina CMC system with weak interface under cycling loading conditions as affected by frequency can be summarized as

I. In the initial stage: when the composite was subjected to fatigue, at low or high frequency, fibers were easily separated from matrix and sliding between fibers and matrix as well among fibers at their interface occurred.

II. In the intermediate stage: in 900 Hz tests frictional temperature rise accelerated causing interfacial reaction between fiber and alumina matrix leading to local bonding. As a result, damage progression was inhibited/decelerated temporarily or for some period causing the enhancement of fatigue life/strength. This intermediate stage occurred above a certain temperature which was attained in the 900 Hz tests. This did not happen at the two lower frequencies of 1 and 100 Hz in the present study. Further, the increase in the fiber/matrix interfacial strength due to local reaction between fibers and matrix at 900 Hz was insufficient to cause any embrittlement which could have resulted in a reduction of fatigue life/strength with planar fracture surfaces.[18]

III. In the final stage: bundles of fiber initially failed and then eventually the composite failed irrespective of low or high cyclic frequency, and this failure was of typical brittle nature as commonly seen in CMCs.

4. Conclusions

The influence of cycling frequency on the tension–tension fatigue behavior of Nextel720^{TM}/alumina, an oxide/oxide CMC, was investigated at room temperature by conducting tests at 1, 100 and 900 Hz. The fatigue life of the composite increased significantly with increase of frequency from 100 to 900 Hz. On the other hand there was relatively very little increase in the fatigue life with increase in cyclic frequency from 1 to 100 Hz at lower stress levels and practically none at higher stress levels (i.e. greater than 130 MPa). The surface temperature of specimens increased by about 70 °C at the highest frequency of 900 Hz, but only slightly (~5 °C) at the two lower frequencies of 1 and 100 Hz.

The possible cause of the increase in fatigue life/strength at the highest frequency was explored by analyzing the failure and damage mechanisms, and by comparing fatigue behavior at room and elevated (1200 °C) temperatures at a low frequency. There was no difference in the failure and damage mechanisms with the change of frequency from 1 to 100 Hz. However, there was development of fiber/matrix interfacial strength due to local bonding from the frictional heating during fatigue at the high frequency of 900 Hz. This interfacial strengthening phenomenon was similar to that observed in specimens tested at 1200 °C. This suggests that interfacial reaction between matrix and fibers due to frictional heating may have inhibited/decelerated the damage progression temporarily leading to an increase in the fatigue life/strength of Nextel720^{TM}/alumina CMC system at the highest frequency of 900 Hz.

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