TENSION-COMPRESSION FAGITUE OF NEXTEL™720/ALUMINA COMPOSITE AT 1200°C IN AIR AND IN STEAM

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Summary: Tension-compression fatigue behavior of an oxide-oxide ceramic matrix composite was investigated at 1200°C in air and in steam. The composite is comprised of an alumina matrix reinforced with Nextel™720 alumina-mullite fibers woven in an eight harness satin weave (8HSW). The composite has no interface between the fiber and matrix, and relies on the porous matrix for flaw tolerance. Tension-compression fatigue behavior was studied for fatigue stresses ranging from 60 to 120 MPa at a frequency of 1.0 Hz. The R ratio (minimum stress to maximum stress) was -1.0. Fatigue run-out was defined as $10^5$ cycles and was achieved at 80 MPa in air and at 70 MPa in steam. Steam reduced fatigue lives by an order of magnitude. Specimens that achieved fatigue run-out were subjected to tensile tests to failure to characterize the retained tensile properties. Specimens subjected to prior fatigue in air retained 100% of their tensile strength. The steam environment severely degraded tensile properties. Tension-compression fatigue was considerably more damaging than tension-tension fatigue. Composite microstructure, as well as damage and failure mechanisms were investigated.

1 INTRODUCTION

Advanced applications such as aircraft turbine engine components, land-based turbines, hypersonic missiles and flight vehicles and, most recently, spacecraft re-entry thermal protection systems have raised the demand for structural materials that exhibit superior long-term mechanical properties and retained properties under high temperature, high pressure, and varying environmental factors. Ceramic-matrix composites (CMCs), capable of maintaining excellent strength and fracture toughness at high temperatures are prime candidate materials for such applications. Because these applications require exposure to oxidizing environments, the thermodynamic stability and oxidation resistance of CMCs are vital issues. The need for environmentally stable composites motivated the development of CMCs based on environmentally stable oxide constituents [1-4].

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1 The views expressed are those of the authors and do not reflect the official policy or position of the United States Air Force, Department of Defense or the U. S. Government.
Oxide/oxide CMCs exhibit damage tolerance combined with inherent oxidation resistance [3, 5]. Moreover, oxide-oxide CMCs have displayed excellent high-temperature mechanical properties [4, 6-9]. However, recent studies revealed dramatic degradation of mechanical performance of oxide-oxide CMCs and their constituents at elevated temperature in steam [10-17]. When a CMC is subjected to mechanical loading at elevated temperature in steam, multiple degradation and failure mechanisms may operate simultaneously. These may include environmentally assisted subcritical crack growth, grain growth and matrix densification, and loss of SiO₂ as Si(OH)₄.

Numerous recent studies investigated mechanical behavior of oxide-oxide CMCs at elevated temperature [6-20]. Porous-matrix oxide/oxide CMCs exhibit several behavior trends that are distinctly different from those exhibited by traditional dense-matrix CMCs with a fiber-matrix interface. Most SiC-fiber-containing CMCs exhibit longer life under static loading and shorter life under cyclic loading [21]. For these materials, fatigue is significantly more damaging than creep. Conversely, in the case of porous-matrix CMCs creep loading was found to be considerably more damaging than fatigue [9, 10]. Furthermore, both creep resistance and fatigue performance of N720/A composite were significantly degraded in the presence of steam [10-18, 22].

Efforts to assess the life-limiting behavior of oxide-oxide CMCs under cyclic loading focused mainly on tension-tension fatigue. Yet, in many potential applications, porous-matrix oxide/oxide CMCs may be subjected to fatigue loading with negative ratios of minimum to maximum stress. Therefore a thorough understanding of tension-compression fatigue performance of oxide-oxide CMCs in service environments is critical to their acceptance for high-temperature structural applications. This study investigates the tension-compression fatigue behavior of an oxide-oxide CMC consisting of a porous alumina matrix reinforced with Nextel™720 fibers. Tension-compression fatigue tests were conducted at 1200°C in air and in steam environments. The composite microstructure, as well as damage and failure mechanisms are discussed.

2 MATERIAL AND EXPERIMENTAL ARRANGEMENTS

The material studied was Nextel™720/alumina (N720/A), an oxide-oxide CMC (manufactured by ATK-COIC, San Diego, CA) consisting of a porous alumina matrix reinforced with Nextel™720 fibers woven in an eight harness satin weave (8HSW). There is no fiber coating. The damage tolerance of the N720/A CMC is enabled by the porous matrix. The composite was supplied in a form of 5.76-mm thick panels comprised of 24 0°/90° woven layers, with a density of ~2.84 g/cm³, a fiber volume of ~44.2%, and matrix porosity of ~22.3%. The fiber fabric was infiltrated with the matrix in a sol-gel process. The laminate was dried with a “vacuum bag” technique under low pressure and low temperature, and then pressureless sintered [23]. The overall microstructure of the CMC is presented in Fig. 1.

All tests were performed at 1200°C using the experimental set-up detailed elsewhere [10, 22, 24]. In all tests, a specimen was heated to test temperature at 1°C/s, and held at temperature for additional 45 min prior to testing. Because compressive loading, and thus the potential for buckling failure modes, was involved in the cycle type, specimens with hourglass-shaped gage section (Fig. 2) were used in all tests. The stress concentration inherent in an hourglass specimen was assessed. Finite element analysis of the specimen shows that the axial stress at the edges in the middle of the hourglass section is only 3.5% higher than the average axial stress. The same procedures were used for testing in air and in steam. Tension-compression fatigue tests were performed in load control with an R ratio
(minimum to maximum stress) of -1.0 at 1.0 Hz. Fatigue run-out was defined as $10^5$ cycles. This cycle count represents the number of loading cycles expected in aerospace applications at that temperature. Cyclic stress-strain data were recorded throughout each test, so that modulus change as well as variations in maximum and minimum strains with fatigue cycles and/or time could be examined. All specimens that achieved run-out were tested in tension to failure at 1200°C in air to determine the retained tensile properties. Fracture surfaces of failed specimens were examined using an optical microscope (Zeiss Discovery V12) and a scanning electron microscope (SEM, Quanta 450).

Figure 1: As-received material: (a) overview, (b) porous nature of the matrix is evident.

Figure 2: Test specimen. All dimensions in mm.

3 RESULTS AND DISCUSSION

3.1 Tension-Compression Fatigue

Results of the tension-compression fatigue tests are shown in Fig. 3 as maximum stress vs. cycles to failure (S-N) curves, where results of the tension-tension fatigue tests from prior work [22] are also included. It is noteworthy that all fatigue failures occurred during the compressive portion of the fatigue cycle.

At 1200°C in air, the fatigue run-out was achieved at 80 MPa (40%UTS), suggesting that the fatigue limit is between 80 and 90 MPa. The tension-compression cycling is considerably more damaging than tension-tension fatigue. Including compression in the load cycle caused dramatic reductions in fatigue life of N720/A composite. For a given stress level, the cyclic lives obtained in tension-tension fatigue [22] were at least three orders of magnitude higher than those produced under tension-compression fatigue. The run-out stress in tension-tension fatigue was a high 170 MPa, more than twice the run-out stress of 80 MPa obtained in tension-compression fatigue. Furthermore, while in tension-tension fatigue a run-out of $10^5$ cycles was achieved at 125 MPa, tension-compression cyclic life at 120 MPa was a very poor
199 cycles. Including compression in the fatigue cycle reduced fatigue life by 99% for $\sigma_{\text{max}}$ of 120 MPa.

![Fatigue S-N curves for N720/A at 1200°C in air and in steam. Arrow indicates that failure of specimen did not occur when the test was terminated. Tension-tension fatigue data from Ruggles-Wrenn et al [22].](image)

Presence of steam causes significant degradation in fatigue performance of the N720/A composite. In steam the tension-compression fatigue run-out was reached only at 70 MPa (35%UTS). The reduction in cyclic life due to steam was 80-92% for $\sigma_{\text{max}}$ of 80 MPa. Similar reductions in cyclic lifetimes due to steam were observed under tension-tension fatigue [10, 22]. However, detrimental effect of steam on cyclic lifetimes was considerably more pronounced in the case of tension-tension fatigue than under tension-compression fatigue. The cyclic lifetimes produced in tension-compression fatigue in steam were nearly an order of magnitude lower than those produced in air. Contrastingly, in the case of tension-tension fatigue steam reduced the cyclic lifetimes by up to three orders of magnitude [10, 22].

Figure 3 compares the S-N curves for tension-compression and tension-tension cycle types on the basis of maximum stress, $\sigma_{\text{max}}$, reached in the cycle. It is seen in that $\sigma_{\text{max}}$ correlates the results obtained in steam for different types of load cycles fairly well. The slopes of the tension-compression and the tension-tension S-N curves produced in steam are close. In steam as in air, the load cycle that involves compression is more damaging. As in air, including compression in the load cycle causes a nearly 100-fold reduction in cyclic life in steam.

Evolution of hysteresis stress-strain response of N720/A composite with cycles in air and in steam is typified in Fig. 4. It is seen that the evolution of the hysteresis response observed at 1200°C in steam is qualitatively similar to that at 1200°C in air. The hysteresis loops produced in tension-compression tests performed in air and in steam are nearly symmetric about the origin. Such symmetry is maintained for the duration of the test. In all tests, the tension modulus (the slope of the tensile portion of the hysteresis loop) was approximately the same as the compression modulus (the slope of the compressive portion of the hysteresis loop) for a given cycle. In all tests, the slope of the tensile portion of the hysteresis loops decreases with fatigue cycling, indicating progressive decrease in the tensile modulus. The slope of the compression portion of the hysteresis loops likewise decreases with cycles, indicating decrease in compressive modulus. The decrease in tensile (compressive) modulus is accompanied by an increase in cyclic tensile (compressive) strain.
Figure 4: Typical evolution of stress-strain hysteresis response of N720/A composite with fatigue cycles at 1200°C (a) in air and (b) in steam. $\sigma_{\text{max}} = 90$ MPa.

Figure 5 shows maximum and minimum strains vs. fatigue cycles for tests conducted at 1200°C in air and in steam. In all tests performed in this work the evolution of minimum strain with cycles mirrors the evolution of maximum strain. Notably, lower maximum strains were accumulated in tests performed with higher levels of maximum stress. Generally, lower strain accumulation with cycling indicates that less damage has occurred, and that it is mostly limited to some additional matrix cracking. However, in this case, low accumulated strains are more likely due to early bundle failures leading to specimen failure. Similar conclusion was reached in the study of tension-tension fatigue of N720/A at 1200°C [22]. It is noteworthy that the maximum strains measured during tension-compression fatigue are lower than the strains accumulated during tension-tension cycling. For example the tensile strains attained in tension-compression fatigue tests with $\sigma_{\text{max}}$ of 95 and 110 MPa do not exceed 0.36%, while tensile strains accumulated in tension-tension fatigue tests with $\sigma_{\text{max}}$ of 100 MPa performed in prior work [22] reached 0.6%. Results in Fig. 5 also reveal that strain accumulation is accelerated in the presence of steam.

Of importance in cyclic fatigue is the reduction in stiffness (hysteresis modulus determined from the maximum and minimum stress-strain data points during a load cycle), reflecting the damage development during fatigue cycling.
The change in normalized modulus (i.e. modulus normalized by the modulus obtained in the first cycle) with fatigue cycles at 1200°C is shown in Fig. 6. The rate of modulus decay and thus the rate of damage accumulation accelerates slightly with increasing maximum stress. It is noteworthy that although some specimens tested in air achieved fatigue run-out, a decrease in normalized modulus with cycling was still observed. Decay in normalized modulus is accelerated in the presence of steam, suggesting an increase in the rate of microstructural damage accumulation in steam. The degrading effect of steam on the evolution of the normalized modulus is observed for all maximum stress levels. This result is consistent with the decreased number of cycles to failure produced in steam.

Retained strength and stiffness of the specimen that achieved fatigue run-out were evaluated in tensile tests performed at 1200°C. The specimens subjected to $10^5$ cycles of prior tension-compression fatigue at 1200°C in air exhibited no loss of tensile strength. However, a modulus loss of 45% was observed. In contrast, prior tension-compression fatigue in steam caused significant degradation of tensile strength. Specimens subjected to $10^5$ fatigue cycles in steam retained only 62% - 83% of their tensile strength and less than 50% of their modulus. The considerable loss of tensile strength suggests that prior tension-compression fatigue in steam may have caused significant degradation of the N720 fibers. Prior tension-tension fatigue in steam also causes degradation of tensile strength and stiffness [22]. However, the strength and modulus loss were greater in the case of prior tension-
compression fatigue. This result indicates that tension-compression fatigue is more damaging than tension-tension fatigue for N720/A composite.

![Graph](image.png)

Figure 6: Normalized tensile modulus vs. fatigue cycles at 1200°C: (a) in air, (b) in air and in steam.

A recent study by Wannaparhun et al. [25] concluded that at 1100°C in water-vapor environment, SiO₂ could be leached from Nextel™720 (N720) fiber. Wannaparhun et al. [25] proposed that H₂O reacted with the SiO₂ in the mullite phase of N720 fibers exposed to water-vapor. The reaction product, Si(OH)₄ then dispersed into the alumina matrix. Ruggles-Wrenn et al. [22] performed energy dispersive x-ray spectroscopy (EDS) of several N720/A specimens subjected to tension-tension fatigue in air and in steam at 1200°C. The results confirmed that in the presence of steam, silicon migrated from the fibers into the adjacent alumina matrix. It was proposed that the depletion of the mullite phase from the N720 fibers caused reduced fatigue performance of the N720/A in steam [22]. These findings were supported by Armani et al. [26], who tested N720 fiber tows in creep at 1200°C in air and in steam. Armani and co-workers reported that a porous alumina layer (void of mullite) of ~2.2-μm thickness formed on the exterior of the fibers tested in steam. Conversely, fibers tested in air did not exhibit such degradation. Formation of a ~2.2-μm thick porous alumina layer that no longer contributed to the load-bearing capacity of the fiber was significant considering the 10-12 μm diameter of N720 fiber.
3.2 Composite Microstructure

Optical micrographs of fracture surfaces obtained in tension-compression fatigue tests conducted at 1200°C in air and in steam are shown in Fig. 7. The optical micrograph of the fracture surface obtained in compression to failure test performed at 1200°C in air with the stress rate of 25 MPa/s is included in Fig. 7 for comparison.

Brushy fracture surface and a fairly long damage zone (~31 mm) indicative of fibrous fracture are produced in compression to failure tests (Fig. 7a). The fracture surface topography of the N720/A specimens failed in fatigue tests performed with $\sigma_{\text{max}}$ of 120 MPa in air (Fig. 7b) and with $\sigma_{\text{max}}$ of 100 MPa in steam (Fig. 7c) is less serrated. Shorter damage zones (~22 mm) are also observed. Not surprisingly, the effects of steam on fracture surface appearance are minimal. These specimens produced the shortest fatigue lives in their respective test environments; hence these fatigue tests were of a fairly short duration (< 8 min). Consider fracture surfaces produced in tension-compression fatigue tests of longer duration (> 24 h) shown in Figs. 7d and 7e. Fracture surface obtained in air with $\sigma_{\text{max}}$ of 80 MPa (Fig. 7d) is similar to that obtained in the fatigue test of shorter duration performed with $\sigma_{\text{max}}$ of 120 MPa in air (Fig. 7b). Uncorrelated fiber fracture and a shorter damage zone are observed. In contrast, the fracture surface of the specimen tested in fatigue with $\sigma_{\text{max}}$ of 75 MPa in steam (Fig. 7e) is dominated by coordinated fiber failure and has a significantly shorter damage zone. At 1200 °C in air and in steam, the length of the damage zone can be
correlated with fatigue lifetime. A short damage zone corresponds to a short cyclic life, while a longer damage zone indicates longer life.

Further insight into the influence of steam on the fracture surface topography and the microstructure of the specimens tested in tension-compression fatigue can be gained by examining the fracture surfaces with an SEM. The SEM micrographs of the fracture surfaces produced in tension-compression fatigue tests performed in air with $\sigma_{\text{max}}$ of 80 MPa and in steam with $\sigma_{\text{max}}$ of 75 MPa are shown in Figs. 8 and 9, respectively. The test environment has a dramatic effect on the fracture surface appearance. The fracture surface produced in air (Fig. 8) is fibrous with considerable regions of fiber pull-out where individual fibers are clearly discernible. A higher magnification image in Fig. 8c reveals only small amounts of matrix material bonded to the fiber surfaces. In contrast, the fracture surface produced in steam (Fig. 9) is dominated by planar regions of brittle failure. A higher magnification image in Fig. 9c reveals increased fiber/matrix bonding as well as the increase in the spatial correlation in the fiber failure locations are among the main manifestations of the matrix densification [27, 28]. The near-planar fracture surface obtained at 1200°C in steam indicates the loss of matrix porosity and subsequent matrix densification due to additional sintering. As a result, the N720/A composite exhibits decreased damage tolerance and reduced fatigue lifetimes at 1200°C.

Figure 8: Fracture surface of N720/A specimen tested in tension-compression fatigue at 1200°C in air ($\sigma_{\text{max}} = 80$ MPa, $N_f = 113382$ cycles)

Figure 9: Fracture surface of N720/A specimen tested in tension-compression fatigue at 1200°C in steam ($\sigma_{\text{max}} = 75$ MPa, $N_f = 86548$ cycles)

The fracture surfaces shown in Figs. 10 and 11 were obtained in tensile tests of the N720/A specimens subjected to $10^5$ cycles of prior fatigue at 1200°C in air and in steam,
respectively. Whereas creep run-out was achieved in both environments, the retained properties of the two specimens were very different. The fracture surface of the run-out specimen tested in air (Fig. 10) is brushy. This observation suggests that matrix changes and, consequently, loss of toughness and degradation of retained properties were limited. Recall that the specimen subjected to $10^5$ cycles of prior fatigue in air retained 100% of its tensile strength. In contrast, the fracture surface of the run-out specimen tested in steam (Fig. 11) is dominated by regions of coordinated fiber failure and increased fiber-matrix bonding. The planar fracture surface in Fig. 11 is indicative of matrix densification and loss of matrix porosity. Prior tension-compression fatigue at 1200°C in steam caused changes in the matrix, which lead to loss of damage tolerance and result in a lower retained tensile strength.

Figure 20: Fracture surface obtained in tensile test of N720/A specimen subjected to $10^5$ cycles of prior tension-compression fatigue with $\sigma_{\text{max}} = 80$ MPa at 1200°C in air.

Figure 31: Fracture surface obtained in tensile test of N720/A specimen subjected to $10^5$ cycles of prior tension-compression fatigue with $\sigma_{\text{max}} = 75$ MPa at 1200°C in steam.

4 CONCLUSIONS

Tension-compression fatigue behavior of the N20/A composite was studied at 1.0 Hz at 1200°C in air and in steam. Fatigue stress levels ranged from 60 to 120 MPa. The fatigue run-out was achieved at 80 MPa (40%UTS) in air and at 70 MPa (35%UTS) in steam.
Presence of steam noticeably degrades tension-compression fatigue performance of N720/A. Steam decreases tension-compression fatigue lives by nearly an order of magnitude. Prior fatigue in air causes no reduction in tensile strength, suggesting that no damage occurred to the fibers. In contrast, prior fatigue in steam can reduce tensile strength by nearly 40%. Tension-compression cycling is considerably more damaging than tension-tension fatigue. Including compression in the load cycle severely degraded fatigue lifetimes.

The damage and failure of the composite at 1200°C in both air and steam are due to loss of matrix porosity and increased fiber/matrix bonding. Introducing compression into the load cycle promotes matrix densification and strengthening of fiber/matrix bond. Matrix densification and fiber/matrix bonding are accelerated in the presence of steam. Moreover, steam degrades N720 fibers by causing depletion of mullite phase from the fibers. These phenomena are behind the reduced fatigue performance in steam.

REFERENCES