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Tension-compression fatigue of an oxide/oxide ceramic composite at elevated temperature ☆

M.B. Ruggles-Wrenn *, R.L. Lanser

Air Force Institute of Technology, Wright-Patterson Air Force Base, Ohio 45433-7765, USA

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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.

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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].

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 [10–21]. 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 SiO_2 as $Si(OH)_4$.

Numerous recent studies investigated mechanical behavior of oxide-oxide CMCs at elevated temperature [6–18,22–24]. 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 [25]. 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 Nextel™720/alumina composite were significantly degraded in the presence of steam [10–18].

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

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* Corresponding author.
E-mail address: marina.ruggles-wrenn@afit.edu (M.B. Ruggles-Wrenn).

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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/cm^3, 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 [26]. 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 [10,18,28]. Prior to testing, extreme care was taken to align the mechanical testing system using the MTS alignment fixture and the alignment specimen instrumented with eight strain gages. In all tests, the misalignment was limited to 0.015% of bending. Note that the N720/A composite exhibits no loss of stiffness with increasing temperature in the 23–1200 °C range [11,27]. Hence the possibility of macroscopic bending during tests due to loss of stiffness with increasing temperature is unlikely. 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.

Deionized water was used to generate steam for testing in steam. Chemical analysis of water entering the steam generator revealed trace amounts (below 10 ppb) of Al, B, Fe, and Zn. Chemical analysis of condensed water exiting the steam generator revealed trace amounts (10–30 ppb) of Al, B, and Fe, and slightly higher but still negligible amounts (55–80 ppb) of Zn. We believe that these levels of impurities are too low to cause contamination of the test specimens and to influence the mechanical performance of the N720/A composite. 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. 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).

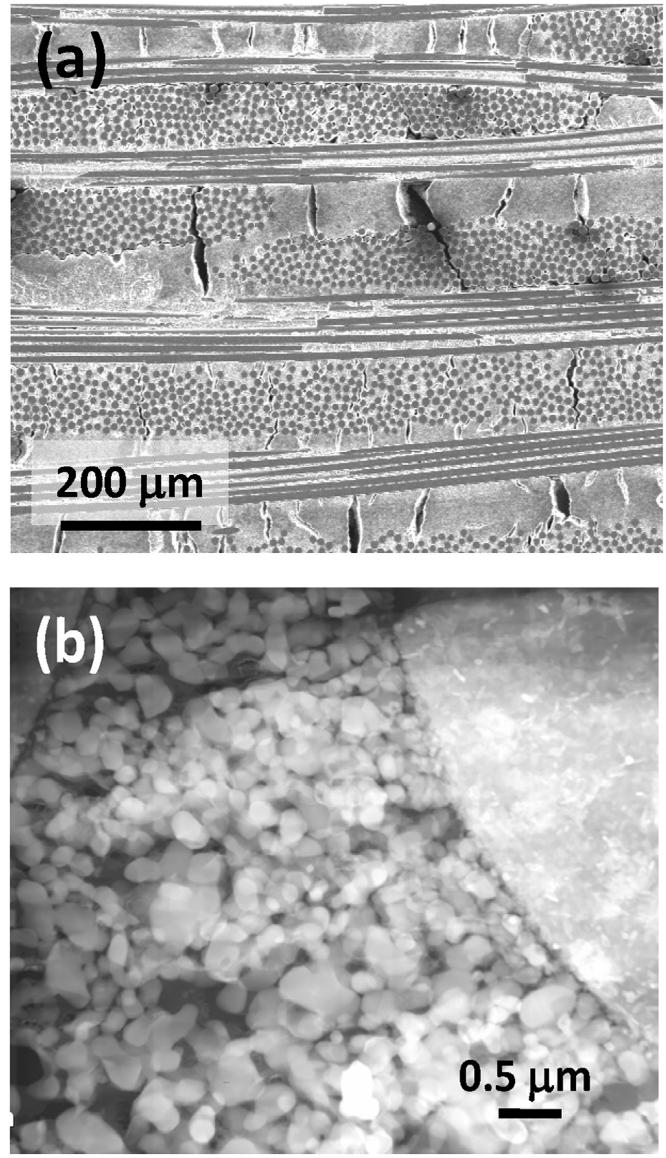

Fig. 1. As-received material: (a) overview, (b) porous nature of the matrix is evident.

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 [18] 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-compression fatigue can be three orders of magnitude lower than those produced under tension-tension fatigue [18]. 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-