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Fatigue behavior of a Hi-Nicalon^{TM}/SiC–B_4C composite at 1200 °C in air and in steam☆

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ABSTRACT

Effects of steam environment on fatigue behavior of a non-oxide ceramic composite with a multilayered matrix were investigated at 1200 °C. The composite was produced via chemical vapor infiltration (CVI). The composite had an oxidation inhibited matrix, which consisted of alternating layers of silicon carbide and boron carbide and was reinforced with laminated woven Hi-Nicalon^{TM} fibers. Fiber preforms had pyrolytic carbon fiber coating with boron carbon overlay applied. Tensile stress–strain behavior and tensile properties were evaluated at 1200 °C. Tension-tension fatigue tests were conducted at 0.1 Hz and at 1.0 Hz for fatigue stresses ranging from 100 to 140 MPa in air and in steam. Fatigue run-out was defined as 10^5 cycles at 0.1 Hz and as 2×10^5 cycles at 1.0 Hz. Presence of steam had little influence on fatigue performance at 1.0 Hz, but noticeably degraded the fatigue lifetimes at 0.1 Hz. Specimens that achieved run-out were subjected to tensile tests to failure to characterize the retained tensile properties. Prior fatigue in air and in steam caused significant reduction in tensile strength and modulus. Composite microstructure, as well as damage and failure mechanisms were investigated.

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1. Introduction

Advanced aero- and space applications such as turbine engine components, hypersonic flight vehicles, and spacecraft reentry thermal protection systems require structural materials that have superior long-term mechanical properties under high temperature, high pressure, and varying environmental factors, such as moisture. Because of their low density, high strength and fracture toughness at high temperatures SiC fiber-reinforced SiC matrix composites are currently being evaluated for aircraft engine hot-section components [1–4]. In these applications the composites will be subjected to cyclic loadings at elevated temperatures in oxidizing environments. Therefore a thorough understanding of fatigue performance of SiC/SiC composites in service environments is critical to design with and life prediction for these materials.

The main advantage of CMCs over monolithic ceramics is their superior toughness, tolerance to the presence of cracks and defects, and non-catastrophic mode of failure. This key advantage is achieved though a proper design of the fiber/matrix interphase, which serves to deflect matrix cracks along the fibers allowing for crack bridging, uncorrelated fiber fracture, and frictional sliding [5–9]. Because their constituents are intrinsically oxidation-prone, the most significant problem hindering SiC/SiC composites is oxidation embrittlement [10]. Typically the embrittlement occurs once oxygen enters through the matrix cracks and reacts with the fibers and the fiber coatings [11–13]. The degradation of fibers and fiber coatings is generally accelerated in the presence of moisture [14]. Composite degradation may be further accelerated by cyclic loading, where the reaction gases are expelled from matrix cracks during unloading and oxidizing atmosphere is drawn into the composite through the matrix cracks during reloading [10]. The issue of improving the oxidation resistance of the SiC/SiC composites has been addressed through the design of innovative multilayered interphases [9,15,16] and of self-healing multilayered matrices [9,16–20]. The multilayered matrices contain phases that facilitate glass formation at high temperatures thus healing the cracks and preventing oxygen from diffusing further into the composite and reaching the oxidation-prone fibers.

Several recent studies investigated mechanical behavior of high-performance SiC/SiC composites at elevated temperature. Zhu et al. [21,22] evaluated creep and fatigue behavior of CVI derived SiC matrix composites reinforced with SiC fibers (Nicalon^{TM} and Hi-Nicalon^{TM}) at temperatures ranging from 1000 °C to 1300 °C in air and in argon. The two woven 2D 0°/90° composites were tested with maximum stresses ranging from 30 to 180 MPa and displayed

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similar creep and fatigue behaviors. At 1300 °C, creep performance of the Hi-Nicalon^{TM} reinforced CMC deteriorated in the presence of argon. This was attributed to the instability of the Hi-Nicalon^{TM} fibers at elevated temperature in argon. At 1300 °C in air for applied stresses exceeding 100 MPa, creep or fatigue failures of both CMCs usually occurred after only a few hours, thus limiting the use of these materials to lower temperatures. Chermant et al. [23] studied creep mechanisms in a CVI SiC–matrix composite reinforced with Hi-Nicalon^{TM} fibers at 1400 °C in air. It was found that microcracks initiated in the 90° bundles, which grew and extended into the load-bearing 0° bundles, producing through-thickness matrix cracks. Eventually one dominant crack formed that led to the ultimate composite failure. Morscher and Pujar [24] investigated creep of the composites consisting of Hi-Nicalon^{TM} S fibers and a melt-infiltrated SiC matrix at 1315 °C in air. These CMCs exhibited excellent creep resistance, which was attributed to the high creep resistance of the Hi-Nicalon^{TM} S fibers. Ojard et al. [25,26] and Morscher et al. [4] evaluated creep and fatigue performance of a melt-infiltrated SiC–matrix composite reinforced with Sylramic^{TM}-iBN fibers. The composite demonstrated excellent creep-rupture and fatigue properties at 1204 °C in air. Two regimes of strength degradation were identified. At higher stresses ($\geq$179 MPa) failure was due to oxidation-induced growth of matrix cracks. At lower stresses ($\leq$165 MPa), failure was controlled by degradation of the fibers. Ruggles-Wrenn and Sharma [27] performed fatigue experiments with Sylramic^{TM} and Sylramic^{TM}-iBN reinforced woven PIP-derived crystalline [SiC + Si_3N_4] matrix composites at 1300 °C in laboratory air and in steam. It was observed that the presence of steam caused noticeable degradation in the fatigue performance of both CMCs. Ruggles-Wrenn et al. [28] investigated fatigue behavior at 1200 °C in air and in steam of a CVI SiC–matrix composite reinforced with Hi-Nicalon^{TM} fibers. The fatigue performance of the CMC improved with decreasing loading frequency, but was noticeably degraded in the presence of steam. Failures of the composite in air and in steam were due to oxidation embrittlement.

Various authors have studied the high-temperature mechanical behavior of the SiC/Si–B–C composites with self/healing matrix. Carrere and Lamon [29] evaluated high-temperature fatigue behavior of a Nicalon^{TM} reinforced CVI composite with a self healing matrix consisting of alternating layers of SiC and SiBC. Crack healing, limited oxidation damage and fiber creep were observed at 1200 °C. Reynaud et al. [30] investigated cyclic fatigue of a CVI 2.5D Hi-Nicalon^{TM}/SiBC composite with a self healing matrix at 600 °C and at 1200 °C in air. It was found that at 600 °C the fatigue lifetime was controlled by slow crack growth in the fibers, while at 1200 °C the fatigue lifetime was controlled by fiber creep. Darzens et al. [31] studied creep damage mechanisms of the CVI composites consisting of Nicalon^{TM} fibers and SiC-based self-healing matrices. It was found that at or above 1200 °C, the creep deformation of the composite was governed by creep of the fibers. Carrere and Lamon [32] examined creep behavior of a CVI Nicalon^{TM}/Si–B–C composite with a multilayered self-healing matrix subjected to static and cyclic loading at 1200 °C. Results revealed that the creep rate of the composite was controlled by creep of fibers and interfacial debonding. No significant creep induced matrix cracking was observed.

This study investigates the fatigue behavior at 1200 °C of a CVI ceramic composite comprised of Hi-Nicalon^{TM} fibers, pyrolytic carbon fiber coating with boron carbide overlay and a SiC-based multilayered matrix. The oxidation-inhibited matrix consists of alternating layers of SiC and B_4C. Fatigue tests were conducted at 1200 °C in air and in steam at frequencies of 0.1 Hz and 1.0 Hz for stress levels ranging from 100 to 140 MPa. Resulting fatigue performance imposes limitations on the use of this material in high-temperature applications. The composite microstructure, as well as damage and failure mechanisms are discussed. The investigation of environmental and frequency effects on the fatigue behavior of the CMC of this type is new and is much needed by the end users of these materials.

2. Material and experimental arrangements

The material studied was Hi-Nicalon^{TM}/SiC–B_4C (Hi-N/SiC–B_4C) ceramic composite manufactured by Hyper-Therm High-Temperature Composites, Inc. (Huntington Beach, CA). The composite was reinforced with Hi-Nicalon^{TM} fibers woven in an eight-harness satin weave, and was processed by CVI. The oxidation inhibited matrix consists of alternating layers of silicon carbide and boron carbide. Laminated fiber preforms were produced from eight plies of woven fabric in a 0°/90° layup symmetric about mid-plane with warp and fill plies alternated. Before the infiltration, the preforms were coated with pyrolytic carbon fiber coating ($\sim$0.40 μm thick) with boron carbide overlay ($\sim$1.0 μm thick) to decrease bonding between the fibers and the matrix. The composite had a finished fiber volume of approximately 34.8% and a density of $\sim$2.56 g/cm^3. The overall microstructure of the CMC is presented in Fig. 1, which shows the oxidation inhibited matrix consisting of alternating layers of SiC and B_4C as well as 0° fibers, PyC fiber coating and B_4C overlay.

All mechanical tests were performed with an MTS servo-controlled testing machine (model 810, MTS, Eden Prairie, MN) equipped with hydraulic water-cooled wedge grips, a compact two-zone resistance-heated furnace, and two temperature controllers. An MTS Flex Test 40 digital controller was used for input signal generation and data collection. Strain was measured with an MTS high-temperature uniaxial extensometer of 12.5-mm gage length (model 632.53 E-14). Tests in steam environment employed an alumina susceptor (tube with end caps), which fits inside the furnace. The specimen gage section is located inside the susceptor, with the ends of the specimen passing through slots in the susceptor. Steam enters the susceptor through a feeding tube in a continuous stream with a slightly positive pressure, expelling the dry air and creating a near 100% steam environment inside the susceptor. For testing at high temperature, thermocouples were bonded to the specimen with Omega CC high-temperature ceramic cement (Omega Engineering Inc., Stamford, CT) to calibrate the furnace on a periodic basis. The furnace controllers (using non-contacting S-type thermocouples exposed to the ambient environment near the test specimen) were adjusted to determine the settings needed to achieve the desired temperature of the test specimen. The determined settings were then used in actual tests. The power settings for testing in steam were determined by placing the specimen instrumented with thermocouples in steam and repeating the furnace calibration procedure. Fracture surfaces of failed specimens were examined with an SEM (FEI Quanta 200 HV) and with an optical microscope (Zeiss Discovery V12).

All tests were performed at 1200 °C. Dog bone shaped specimens of 152 mm total length with a 10-mm-wide gage section were used in all tests. The test specimens were sealed after machining. In all tests, a specimen was heated to test temperature at 1 °C/min, and held at temperature for additional 25 min prior to testing. The same procedures were used for testing in air and in steam. Tensile tests were performed in displacement control with a constant rate of 0.05 mm/s. Tension–tension fatigue tests were performed in load control with an R ratio (minimum to maximum stress) of 0.05 at 0.1 Hz and 1.0 Hz. Fatigue run-out was defined as 10^5 cycles at 0.1 Hz. The cycle count of 10^5 represents the number of loading cycles expected in aerospace applications at that temperature. The fatigue run-out was defined as 2×10^5 cycles at 1.0 Hz because longer run-out time could be accommodated at this frequency. Cyclic stress–strain data were recorded throughout each test, so