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Effects of steam environment on compressive creep behavior of Nextel™720/Alumina ceramic composite at 1200 °C ☆

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ABSTRACT

The compressive creep behavior of an oxide–oxide ceramic-matrix composite (CMC) was investigated at 1200 °C in laboratory air and in steam. The composite consists of a porous alumina matrix reinforced with laminated, woven mullite/alumina (Nextel™720) fibers, has no interface between the fiber and matrix, and relies on the porous-matrix for flaw tolerance. The compressive stress–strain behavior was investigated and the compressive properties measured. The influence of the loading rate on compressive stress–strain response and on compressive properties was also explored. A change in loading rate by four orders or magnitude had a profound effect on compressive properties in air and especially in steam. Compressive creep behavior was examined for creep stresses in the −40 to −100 MPa range. Minimum creep rate was reached in all tests. In air, compressive creep strain magnitudes remained <0.4% and compressive creep strain rates approached $-3.5 \times 10^{-7}\ s^{-1}$. Creep run-out defined as 100 h at creep stress was achieved in all tests conducted in air. The presence of steam accelerated creep rates and significantly reduced creep lifetimes. In steam, compressive creep strains approached −1.6%, and compressive creep strain rates, $-1.9 \times 10^{-2}\ s^{-1}$. In steam, maximum time to rupture was only 3.9 h. Composite microstructure, as well as damage and failure mechanisms were investigated.

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1. Introduction

Advances in power generation systems for aircraft engines, land-based turbines, rockets, and, most recently, hypersonic missiles and flight vehicles have raised the demand for structural materials that have superior long-term mechanical properties and retained properties under high-temperature, high pressure, and varying environmental factors, such as moisture [1]. Ceramic-matrix composites, capable of maintaining excellent strength and fracture toughness at high-temperatures are prime candidate materials for such applications. Additionally, lower densities of CMCs and their higher use temperatures, together with a reduced need for cooling air, allow for improved high-temperature performance when compared to conventional nickel-based superalloys [2]. Advanced aerospace turbine engines will likely incorporate fiber-reinforced CMCs in critical components, such as combustor walls [3–5]. 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 [6–11].

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. It is widely accepted that in order to avoid brittle fracture behavior in CMCs and improve the damage tolerance, a weak fiber/matrix interface is needed, which serves to deflect matrix cracks and to allow subsequent fiber pull-out [12–14]. It has been demonstrated that similar crack-deflecting behavior can also be achieved by means of a finely distributed porosity in the matrix instead of a separate interface between matrix and fibers [15]. This microstructural design philosophy implicitly accepts the strong fiber/matrix interface. The concept has been successfully demonstrated for oxide–oxide composites [6,9,11,16,17]. Resulting oxide/oxide CMCs exhibit damage tolerance combined with inherent oxidation resistance. An extensive review of the mechanisms and mechanical properties of porous-matrix CMCs is given in [18,19].

Porous-matrix oxide/oxide CMCs exhibit several behavior trends that are distinctly different from those exhibited by traditional non-oxide CMCs with a fiber-matrix interface. For the non-oxide CMCs, fatigue is significantly more damaging than creep. Contrastingly, Zawada et al. [20] examined the high-temperature mechanical behavior of a porous-matrix Nextel610/Aluminosilicate composite. Results revealed excellent fatigue performance at 1000 °C. Conversely, creep lives were short, indicating low creep

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resistance and limiting the use of that CMC to temperatures below 1000 °C. Ruggles-Wrenn et al. [21] showed that Nextel™720/Alumina (N720/A) composite exhibits excellent fatigue resistance in laboratory air at 1200 °C. The fatigue limit (based on a run-out condition of 10^5 cycles) was 170 MPa (88% UTS at 1200 °C). Furthermore, the composite retained 100% of its tensile strength. However, creep loading was found to be considerably more damaging. Creep run-out (defined as 100 h at creep stress) was achieved only at stress levels below 50% UTS. Mehrman et al. [22] demonstrated that introduction of a short hold period at the maximum stress into the fatigue cycle significantly degraded the fatigue performance of N720/A composite at 1200 °C in air. In steam, superposition of a hold time onto a fatigue cycle resulted in an even more dramatic deterioration of fatigue life, reducing it to a much shorter creep life at a given applied stress.

Because creep was shown to be considerably more damaging than cyclic loading to oxide–oxide CMCs with porous-matrix [20–22], high-temperature creep resistance remains among the key issues that must be addressed before using these materials in advanced aerospace applications. This study investigates monotonic compression and compressive creep behaviors of the Nextel™720/alumina (N720/A), an oxide–oxide CMC with a porous-matrix, at 1200 °C in air and in steam environment. Results reveal that the presence of steam dramatically degrades compressive creep lifetimes.

2. Material and experimental arrangements

The material studied was Nextel™720/Alumina (N720/A), an oxide–oxide CMC (manufactured by COI Ceramics, San Diego, CA) consisting of a porous alumina matrix reinforced with Nextel™720 fibers. There is no fiber coating. The damage tolerance of N720/A is enabled by the porous-matrix. The composite was supplied in a form of 5.2-mm thick plates comprised of 240°/90° woven layers, with a density of ~2.77 g/cm^3 and a fiber volume of ~44%. Composite porosity was ~22%. 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, then pressureless sintered [23]. Representative micrograph of the untested material is presented in Fig. 1a, which shows 0° and 90° fiber tows as well as numerous matrix cracks. In the case of the as-processed material, most are shrinkage cracks formed during processing rather than matrix cracks generated during loading. Porous nature of the matrix is seen in Fig. 1b.

A servocontrolled MTS mechanical testing machine equipped with hydraulic water-cooled collet grips, a compact two-zone resistance-heated furnace, and two temperature controllers was used in all tests. An MTS TestStar II digital controller was employed for input signal generation and data acquisition. Strain measurement was accomplished with an MTS high-temperature air-cooled uniaxial extensometer of 12.5–mm gage length. For elevated temperature testing, thermocouples were bonded to the specimens using alumina cement (Zircar) to calibrate the furnace on a periodic basis. The furnace controllers (using non-contacting thermocouples exposed to the ambient environment near the test specimen) were adjusted to determine the power setting needed to achieve the desired temperature of the test specimen. The determined power settings were then used in actual tests. 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 is introduced into 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. The power settings for testing in steam were determined by placing the specimen instrumented with thermocouples in steam environment and repeating the furnace calibration procedure. Thermocouples were not bonded to the test specimens after the furnace was calibrated. Fracture surfaces of failed specimens were examined using an optical microscope (Zeiss Discovery V12).

All tests were conducted at 1200 °C. In all tests, a specimen was heated to the test temperature at a rate of 1 °C/s, and held at temperature for additional 30 min prior to testing. Straight-sided 18 mm × 150 mm specimens were used in all compression tests. All N720/A test specimens used in this study were cut from a single plate. Monotonic compression tests were performed in stress control with constant stress-rate magnitudes of 0.0025 and 25 MPa/s in laboratory air and in steam environment. In all compression tests the modulus of elasticity was calculated in accordance with the procedure in ASTM standard C 1358 as the slope of the compressive stress–strain curve within the linear region. In compressive creep-rupture tests specimens were loaded to the creep stress level at the stress-rate magnitude of 25 MPa/s. Creep run-out was defined as 100 h at a given creep stress. In each test, stress–strain data were recorded during the loading to the creep stress level and the actual creep period. Thus both total strain and creep strain could be calculated and examined. To determine the retained tensile (compressive) strength and modulus, specimens that achieved creep run-out were subjected to tensile (compressive) tests to failure at 1200 °C. It is worthy of note that in all tests reported below, the failure occurred within the gage section

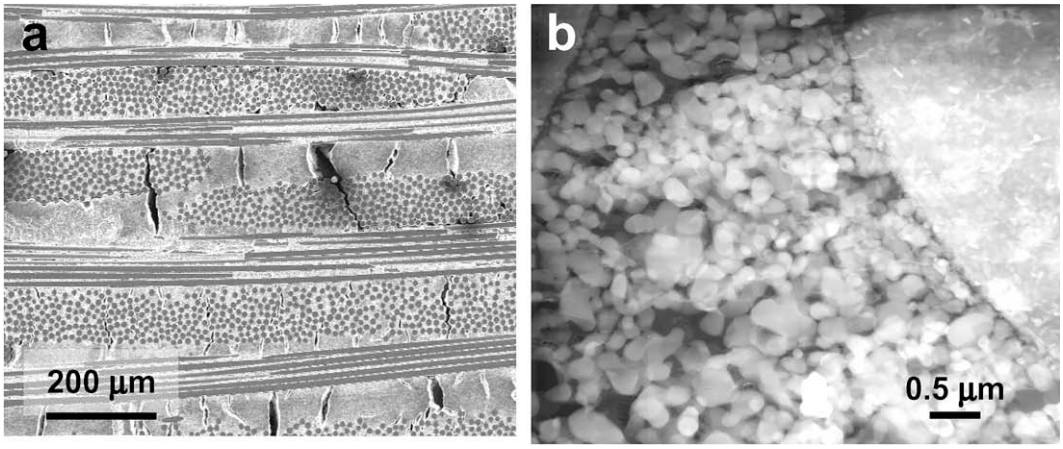

Fig. 1. As-processed material: (a) overview showing shrinkage cracks, (b) porous nature of the matrix is evident.