Tensile creep and fatigue of Sylramic-iBN melt-infiltrated SiC matrix composites: Retained properties, damage development, and failure mechanisms

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ABSTRACT

An understanding of the elevated temperature tensile creep, fatigue, rupture, and retained properties of ceramic matrix composites (CMC) envisioned for use in gas turbine engine applications is essential for component design and life-prediction. In order to quantify the effect of stress, time, temperature, and oxidation for a state-of-the-art composite system, a wide variety of tensile creep, dwell fatigue, and cyclic fatigue experiments were performed in air at 1204 °C for the SiC/SiC CMC system consisting of Sylramic-iBN SiC fibers, BN fiber interphase coating, and slurry-cast melt-infiltrated (MI) SiC-based matrix. Tests were either taken to failure or interrupted. Interrupted tests were then mechanically tested at room temperature to determine the residual properties. The retained properties of most of the composites subjected to tensile creep or fatigue were usually within 20% of the as-produced strength and 10% of the as-produced elastic modulus. It was observed that during creep, residual stresses in the composite are altered to some extent which results in an increased compressive stress in the matrix upon cooling and a subsequent increased stress required to form matrix cracks. Microscopy of polished sections and the fracture surfaces of specimens which failed during stressed-oxidation or after the room-temperature retained property test was performed on some of the specimens in order to quantify the nature and extent of damage accumulation that occurred during the test. It was discovered that the distribution of stress-dependent matrix cracking at 1204 °C was similar to the as-produced composites at room temperature; however, matrix crack growth occurred over time and typically did not appear to propagate through-the-thickness except at the final failure crack. Failure of the composites was due to either oxidation-induced unbridged crack growth, which dominated the higher stress regime ($\geqslant$179 MPa) or controlled by degradation of the fibers, probably caused by intrinsic creep-induced flaw growth of the fibers or internal attack of the fibers via Si diffusion through the CVI SiC and/or microcracks at the lower stress regime ($\leqslant$165 MPa).

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1. Introduction

Silicon carbide fiber-reinforced silicon-carbide matrix composites are currently being evaluated for aircraft engine hot-section components [1–3]. Elevated temperature creep and fatigue conditions under oxidative environments are a primary concern for these types of applications. Therefore, it is essential that the effect of elevated temperature creep and fatigue conditions be well understood in order to predict useful lives for these types of composites. As part of this effort, it is critical to understand the development of damage in these materials over stress, time, and environment in order to understand the mechanisms that lead to degradation in stress-strain behavior and ultimate creep or fatigue rupture.

The effect of damage development in some 2D-woven 0/90 SiC-reinforced non-oxide reinforced composites has been determined for CVI SiC matrix systems with lower modulus SiC-based fiber-types (Nicalon™ and Hi-Nicalon™) for creep and fatigue conditions between 1100 and 1400 °C in air and argon environments [4–6]. In those composite systems, with increasing stress and time, micro cracks are formed in the 90° minicomposite bundles. With increasing stress and/or time, the cracks grow and extend through the CVI SiC into the load-bearing 0° fiber minicomposite bundles producing through-the-thickness matrix cracks. Eventually a "master crack" [6] will form that becomes the site of ultimate failure. For these composite systems, fairly high strains to failure (0.5% to

greater than 1%) are achieved. However, times to failure in air are usually less than 100 h for stresses that are in excess of the matrix cracking stress (e.g., ∼100 MPa for the Hi-Nicalon™ CVI SiC composite tested in Ref. [4] at 1300 °C).

For SiC/SiC composites reinforced with high modulus polycrystalline SiC fibers (Hi-Nicalon S) and a slurry-cast melt-infiltrated (MI) SiC matrix composite system a somewhat different damage development was observed at 1315 °C [7]. For the stress ranges tested (up to 172 MPa), only minor microcracking in the 90° minicomposites was observed. In some cases, these cracks were observed to extend to the surface (138 MPa) where 90° minicomposites were adjacent to the surface and into some 0° minicomposites at higher stresses (172 MPa) resulting in local fiber failure but not significant through-the-thickness matrix crack formation. However, for this study, creep times were usually limited to 100 h followed by determination of retained stress–strain behavior, which was usually very high since minimal damage occurred in the composites.

It is the goal of this study to further understand the development of damage in a similar polycrystalline SiC fiber-reinforced slurry-cast MI SiC matrix system, the Sylramic-iBN MI SiC system developed at NASA Glenn Research Center and referred to as N24A [8]. This material has undergone some of the most exhaustive testing to date of any high-performance SiC/SiC composite system under an US Air Force sponsored program, including tensile creep, 2 hour dwell fatigue, 1 Hz cyclic fatigue, and 30 Hz cyclic fatigue at 1204 °C in air for stresses ranging from 110 to 220 MPa for times up to 2000 h [9–10]. Specimens from this wide range of tests were acquired for this study in order to determine the damage accumulation for the wide range of stress–time conditions which will be described here. Some of the specimens had been interrupted at predetermined times. For most of those specimens, a room temperature unload-reload test to failure was performed with acoustic emission (AE) monitoring in the same way as Ref. [7] in order to determine the residual properties of the composite.

2. Experimental

The composite system evaluated is described in more detail elsewhere [8]. It consists of eight plies of 2D woven five-harness satin Sylramic-iBN fabric, a CVI BN fiber interphase coating, a CVI SiC coating of initial matrix to rigidize the preform and protect the fibers, slurry-infiltrated SiC particulates, and molten Si infiltrated to fill in the remaining open porosity. The initial five-harness fabric consisted of 7.9 tow ends per cm of Sylramic™ SiC fiber balanced in the warp and weft 0/90 orthogonal directions. The fiber tows consisted of 800 fibers approximately 10 μm in diameter. The fibers were originally produced by Dow Corning, Midland, Michigan, but are now produced by ATK COI Ceramics in San Diego, CA. The precursor Sylramic fabric plies were subjected to a NASA-proprietary treatment in order to improve fiber creep-resistance and also produce a thin (∼150 nm) in situ grown BN layer on the surface of each fiber prior to composite fabrication. The composites were fabricated as 153 × 230 mm panels by ply lay-up at GE Ceramic Composite Products, LLC in Newark, DE. All plies were aligned 0/90 in plane, but were randomly stacked through-the-thickness with a degree of fiber nesting. The fiber volume fraction of composites was approximately 36–38% as measured by weight.

The tensile specimens were machined from the as-fabricated panels with a length of ∼155 mm, a grip width of ∼12 mm, and a dogboned section in the middle with a length of ∼40 mm and a width of ∼8.2 mm. One of the orthogonal fiber directions was aligned within +/−3° of the specimen length or tensile direction. Typical specimen thickness was ∼2 mm.

Some of the elevated temperature tests were performed in other studies [9–10]. All the tests were performed at 1204 °C in lab air at Southern Research Institute, Birmingham AL or Cincinnati Test Labs, Cincinnati, OH. Tensile creep tests were performed under constant load using either universal testing or lever-arm machines. The cyclic fatigue tests were all performed in hydraulic testing machines; while dwell fatigue testing was done in modified lever-arm tensile machines to control loading rates. Several fatigue loading rates were used. Dwell fatigue (DF) consisted of a 2 h hold followed by a 1 min unload-reload back to a two hour hold. High cycle fatigue (HCF) was performed at 1 or 30 Hz. All of the fatigue tests were performed for an R ratio of 0.05. Displacement for all tests was monitored using contact extensometers on the edge of the specimen with a gage length of 25 mm. Creep strain was determined after reaching full applied stress. The furnace assured uniform temperature along the gage section.

For some of the specimens which were not taken to failure, a room temperature unload-reload hysteresis tensile test was performed to failure to determine residual properties. AE sensors were placed above and below the extensometers (50–60 mm apart) and AE was monitored during the room temperature test using a Digital Wave Fracture Wave detector (Englewood CO) as described elsewhere [7,11]. After the test, the events were sorted as to location along the specimen length and only those events which occurred in the 25 mm gage section were used for AE analysis.

Analysis of the crept/fatigued specimens consisted of both optical and scanning electron microscopy. Fig. 1 shows a typical failed specimen. One part of the fracture surface was used for observation of the fracture surface in a field emission scanning electron microscope (FESEM – Hitachi 4700, Tokyo Japan). The other half of the specimen was cut (between 10 and 15 mm long) and polished along the edge (approximately 1 mm from the exposed edge) and/or face of the specimen in order to observe and quantify the number of matrix cracks along the length. Matrix cracks caused by time-dependent deformation had significant crack-openings and were easy to distinguish from matrix cracks formed during the room temperature retained strength tests, which were often not discernable as-polished due to crack closure from the high compressive stresses in the matrix. For the edge-polished specimens, matrix cracks were counted along the length for both
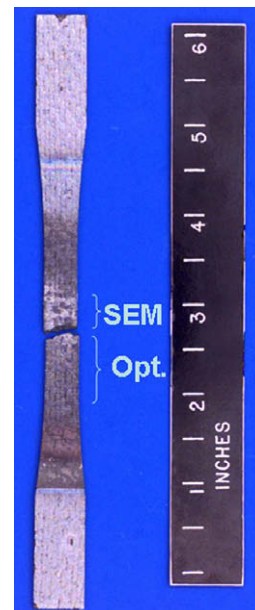

Fig. 1. Typical failed specimen after creep or fatigue.