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Degradation mechanisms of a SiC fiber reinforced self-sealing matrix composite in simulated combustor environments

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Abstract

Non-oxide ceramic matrix composites are potential candidates to replace the current nickel-based alloys for a variety of high temperature applications in the aerospace field. The durability of a $SiC_{(f)}/PyC_{(i)}/[Si, C, B]_{(m)}$ composite with a multi-layered self-sealing matrix and Hi-Nicalon fibers was investigated at 1200 °C for exposure durations up to 600 h. The specimens are aged in a variety of slow-flowing air/steam gas mixtures and total pressures, ranging from atmospheric pressure with a 10–50% water content to 1 MPa with 10–20% water content. The degradation of the composite was determined from the measurement of residual strength and strain to failure on post-exposure specimens and correlated with microstructural observation of the damaged tows. The most severe degradation of the composite occurred at 1 MPa in an air/steam (80/20) gas mixture. Correlation between this degradation and the dissolving of the SiC fibers in the generated boria-containing glass, is discussed.
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1. Introduction

Non-oxide ceramic matrix composites such as $SiC_{(f)}/PyC_{(i)}/SiC_{(m)}$ consist of SiC matrix reinforced with SiC fibers and pyrocarbon (PyC) interfacial coating. These composites exhibit a low density associated with high thermomechanical properties and are potential candidates to replace the current nickel-based alloys for a variety of long-term applications in the aerospace field. In these applications, $SiC_{(f)}/PyC_{(i)}/SiC_{(m)}$ components can be subjected to service conditions that include mechanical loading under intermediate to high temperatures and high pressure complex environment containing oxygen and steam. The oxidation of the PyC weak interphase can occur under dry air at a temperature lower than 500 °C and leads to interfacial degradations of $SiC_{(f)}/PyC_{(i)}/SiC_{(m)}$. $SiC_{(f)}/PyC_{(i)}/[Si, C, B]_{(m)}$ composites with a sequenced self-sealing matrix have been developed[1,2] and investigated[3–11] to protect the PyC interphase against oxidation effects up to 1400 °C. The principles of the self-sealing approach are to consume part of the incoming oxygen and limit access of residual oxygen to the PyC interphase by sealing the matrix microcracks with a SiO_2–B_2O_3 oxide phase. However, previous studies showed that B_2O_3[4–6,8,12,13] and SiO_2[15,17] can volatilize, respectively, at 600 and 1100 °C under water vapor-containing environments. This phenomenon can cause the self-sealing capability to degrade, thus reducing the lifetime of the $SiC_{(f)}/PyC_{(i)}/[Si, C, B]_{(m)}$.

The matrix layers are SiC, B_4C and a phase noted Si–B–C. The efficiency of the self-sealing process under environments containing both oxygen and water vapor, results from the competition between the oxidation of the matrix layers and the volatilization of the generated oxide phase.

Under dry air, B_4C undergoes oxidation and volatilization reactions below 600 and 900 °C, respectively, as shown below[4–6,8,12,13]:

$$B_4C_{(s)} + 4O_{2(g)} = 2B_2O_{3(l)} + CO_{2(g)} \quad (1)$$

$$B_2O_{3(l)} = B_2O_{3(g)} \quad (2)$$

Under water vapor-containing environments, $B_2O_{3(l)}$ may react significantly at 600 °C to form hydroxydes by the following

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reactions[4–6,8,12,13]:

$$\frac{3}{2}B_2O_{3(l)} + \frac{3}{2}H_2O_{(g)} = H_3B_3O_{6(g)} \quad (3)$$

$$\frac{3}{2}B_2O_{3(l)} + \frac{1}{2}H_2O_{(g)} = H_3BO_{3(g)} \quad (4)$$

$$\frac{1}{2}B_2O_{3(l)} + \frac{1}{2}H_2O_{(g)} = HBO_{2(g)} \quad (5)$$

The competition between the oxidation (1) and volatilization reactions (2)–(5) can lead to the recession of B_4C.

In high temperature environments and under dry air, the active oxidation of SiC occurs above 1000 °C and below $P_{O_2} = 100$ Pa according to (6). At a higher P_{O_2}, a paralinear oxidation of SiC[14] occurs to form a protective SiO_2 scale according to (7). In environments containing O_2 and H_2O, the formation of SiO_2 is dramatically enhanced by reaction (8)[14–16]:

$$SiC_{(s)} + O_{2(g)} = SiO_{(g)} + CO_{(g)} \quad (6)$$

$$2SiC_{(s)} + 3O_{2(g)} = 2SiO_{2(s)} + 2CO_{(g)} \quad (7)$$

$$SiC_{(s)} + 3H_2O_{(g)} = SiO_{2(s)} + CO_{(g)} + 3H_{2(g)} \quad (8)$$

Under steam-containing environments, the SiO_2 scale may volatilize, by the main following reaction[17]:

$$SiO_{2(s)} + 2H_2O_{(g)} = Si(OH)_{4(g)} \quad (9)$$

The volatilization rate of SiO_2[15,17] is much lower than for B_2O_3.[4–6,8,12,13] However, as for boron matrix layers, the competition between the oxide formation (7) and (8) and the oxide phase volatilization (9) reactions can lead to the recession of SiC.[18–20]

The Si–B–C matrix layer can be described as a mixture of SiC nanocrystals in an amorphous B_4C phase.[7] A previous study investigated the oxidation of Si–B–C coatings under oxygen and steam-containing environments by thermogravimetric analysis.[4,5] It has been shown that SiC nanocrystals can oxidize significantly at 600 °C and at atmospheric pressure, simultaneously to the B_2O_3 formation, to form silica according to reaction (7).

Previous works[9] focused on the corrosion behavior of the $SiC_{(f)}/PyC_{(i)}/[Si, C, B]_{(m)}$ at high pressure and at 600 °C. The aim of this study is to evaluate, at a higher temperature, the effects of both oxygen and water vapor on the self-sealing process of $SiC_{(f)}/PyC_{(i)}/[Si, C, B]_{(m)}$ composites subjected to high-pressure environments. Corrosion tests are conducted for long periods of time on $SiC_{(f)}/PyC_{(i)}/[Si, C, B]_{(m)}$ composites at 1200 °C in oxygen and steam-containing environments at atmospheric pressure and high pressure. Post-exposure mechanical tests are performed at room temperature (RT) to investigate the effects of corrosion phenomena on the retained mechanical properties.

2. Materials and test specimens

The material investigated is the CERASEP® A410[10,11] (C410) manufactured by Snecma Propulsion Solide (France) via chemical vapor infiltration (CVI). It is a woven-SiC-fiber reinforced [Si, C, B] sequenced matrix composite (Fig. 1). The

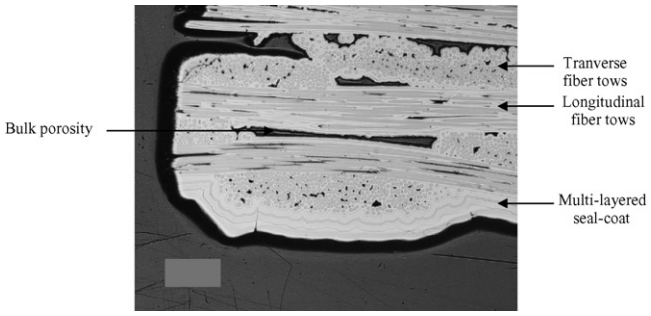

Fig. 1. Polished cross-section of the as-received C410 material.

different matrix layers are crystallised SiC, amorphous B_4C and a SiC–B_4C phase named Si–B–C which can be described as a mixture of SiC nanocrystals in a B_4C amorphous phase.[7] The sequenced matrix is reinforced by Hi-Nicalon® SiC fibers and plane multi-layer reinforcement is used to eliminate delamination sensitivity that is common for 2D ceramic matrix composites. Fiber volume fraction, material density and mainly closed bulk porosity, as reported by the composite manufacturer, are, respectively, 34%, 2.25 ± 0.05 and 13 ± 1%. The interphase is pyrocarbon.

The test specimen geometry used in this study has a reduced gauge section (Fig. 2). It is 200 mm long, with a grip section width of 24 mm, a reduced gauge section width of 16 mm and a thickness of 4.4 mm. Two different material batches, manufactured in similar conditions, are used for these works. Test samples are machined from composite plates using diamond grinding and then are seal-coated with CVI layers of SiC, B_4C and Si–B–C. The sequenced seal-coat thickness, with a SiC final layer, is 120 μm on the composite surface and about 40 μm on the machined edges. Corrosion tests are also performed on Si–B–C coating and SiC/SiC coupons for comparison purposes. The Si–B–C coating, with a thickness of 30 ± 3 μm, is deposited on SiC chips (diameter of 8 mm and thickness of 2 mm) via chemical vapor deposition (CVD).

3. Test procedures

3.1. Pre-damaging

The dog-bone specimens are loaded in tension monotonically at room temperature to a tensile stress of 150 MPa (the stress cor-

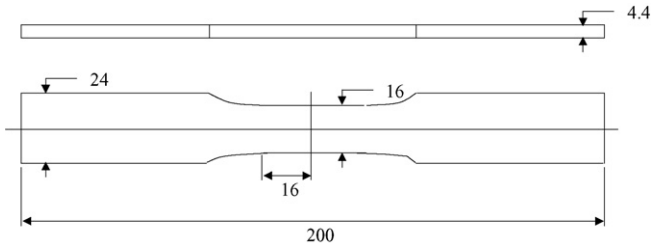

Fig. 2. C410 specimen geometry used in this study. Dimensions are in millimeters.

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