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Complete recovery of high temperature oxidation resistance in carbon fiber reinforced SiC composites by a recoating repair methodology

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ABSTRACT

The current work reports on the oxidation behavior and residual flexural strength of carbon fiber–reinforced silicon carbide composites (C/SiC) after induction of thermal crack damage by heat treatment (HT) at 1900 °C and the effect, therein, of a repair process involving recoating by SiC. As-prepared, heat-treated and heat-treated/recoated specimens, were subjected to static oxidation tests in air at a temperature range of 500–1500 °C for 10 h and then tested in three-point bending. It was found that composite weight of heat-treated samples decreased dramatically with increasing oxidation temperature with weight loss values of ~30% being systematically observed for oxidation temperatures above 800 °C. On the other hand, as-prepared and heat-treated/SiC-recoated specimens reached almost their original weight after oxidation. The residual flexural strength of C/SiC composites with thermally-induced crack damage decreased significantly compared to as-prepared specimens, while SiC recoating was found to efficiently enable strength enhancement. Microstructural analysis showed that HT was associated with increased population and dimensions of micro-cracks on the C/SiC surface while SiC recoating enabled repair of HT-induced thermal crack damage hence leading to oxidation resistance recovery of the material.

1. Introduction

Carbon fiber reinforced silicon carbide matrix composites (C/SiC) not only combine the advantages of metal, ceramic and carbon materials in terms of thermo-structural and functional performance, but also overcome some of their shortcomings. Due to their remarkable mechanical properties and high temperature stability, C/SiC composites are considered an increasingly important candidate for aeronautical and astronautical applications [1,2]. A considerable amount of research has focused on the investigation of the oxidation behavior of C/SiC composites in different environments [3–7]. Halbig et al. studied the oxidation kinetic regimes for carbon fibers in a cracked silicon carbide matrix under both stressed and stress-free conditions [8]. It was found that micro-cracks play a key role in the oxidation process in both cases. Heat treatment (HT) has been reported as an important method to modify the thermal, mechanical, and functional properties of the fibers and their reinforced composites. Mei et al. confirmed that HT is associated with increases in the strength and toughness of C/SiC with a thin PyC interphase [9]. According to [10], HT can also act as a high temperature test for C/SiC, wherein the population and dimensions of matrix micro-cracks increase sharply hence causing significant changes in oxidative behavior.

The aim of this study is two-fold: (i) to investigate the effect of thermal crack damage on the oxidation resistance of C/SiC composites and (ii) to explore the effectiveness of recoating of the material by SiC as a damage repair method. Thermal crack damage was introduced by exposing C/SiC composites to a simulated high temperature environment of 1900 °C in argon atmosphere for 2 h. After this HT, part of the specimen batch was recoated with SiC via a chemical vapor deposition (CVD) methodology. As-prepared, heat-treated and heat-treated/SiC-recoated specimens were subjected to static oxidation tests in air and three-point bending tests. Infrared thermal wave imaging and scanning electron microscopy (SEM) were used for analyzing the thermally-induced damage and observing the microstructures of C/SiC, respectively.

2. Experimental

2.1. Materials

A two-dimensional (2D) preform was fabricated by laminating T-300™ carbon fiber (Toray Inc., Japan) cloths to a fiber volume fraction of ca. 40%. Pyrolytic carbon (PyC) layer was first deposited on the

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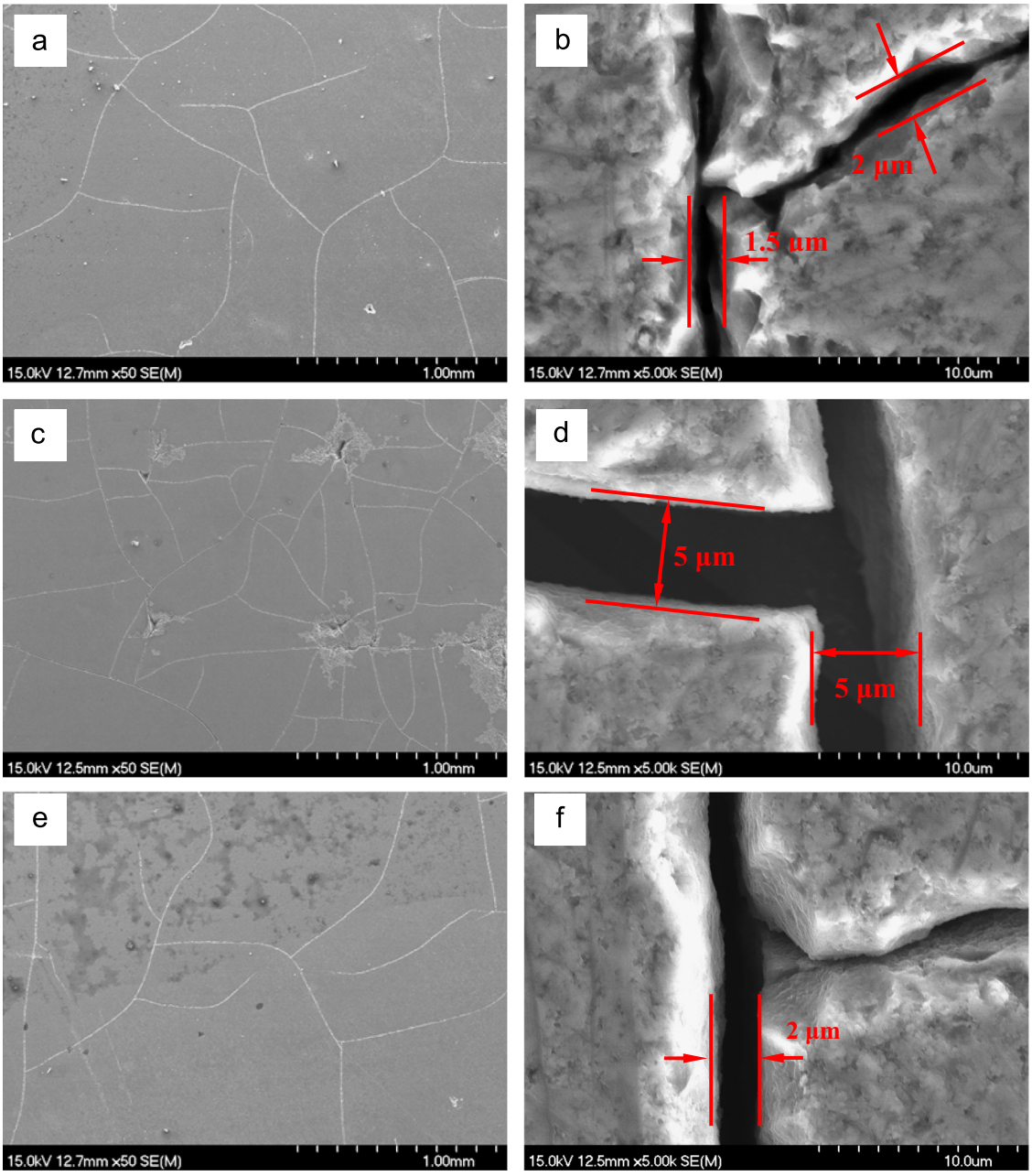

Fig. 1. SEM micrographs for specimen surfaces before oxidation: (a) and (b) S1; (c) and (d) S2; (e) and (f) S3.

surface of fibers by CVD at 900 °C. Subsequently, SiC matrix was infiltrated on the PyC interphase inside the 2D preform by chemical vapor infiltration (CVI). Methyltrichlorosilane (MTS, CH_3SiCl_3) was used in combination with hydrogen as carrier gas. The infiltration was performed at a temperature of 1000 °C, with a H_2: MTS ratio of $\alpha = 10$ and a pressure of 4 kPa. Three-point bending prisms with dimensions of $40 \times 5 \times 3.5$ mm^3 (length × width × thickness) were cut from the as-fabricated composite plates and further coated with SiC by the same CVI technique until the coating thickness reached a value of approximately 50 μm.

As-prepared C/SiC specimens were heat-treated at 1900 °C in argon for 2 h to inflict thermal crack damage into the composites. A part of the heat-treated specimen batch was further processed to remove the damaged SiC coating and samples were recoated by SiC through the same CVI methodology. As-prepared, heat-treated and heat-treated/SiC-recoated specimens were denoted as S1, S2 and S3, respectively.

Table 1
Statistical crack linear density, crack width and thermal exposure area for as-prepared (S1), heat-treated (S2) and SiC-recoated heat-treated (S3) composites.

Specimen type	Crack linear density (mm^{-1})	Crack width (μm)	Thermal exposure area (mm^2)
S1	1.80 ± 0.69	1.28 ± 0.36	1.57 ± 0.54
S2	5.30 ± 0.85	4.30 ± 0.67	15.5 ± 0.96
S3	2.00 ± 0.80	1.50 ± 0.42	2.04 ± 0.34

2.2. Measurements and observations

A horizontal oxidation furnace (Model LTF 12/50/300-3, Lenton Corp., UK) was used for oxidation tests. The experiments were performed in air atmosphere for 10 h at 11 different temperatures, ranging from 500 to 1500 °C with a step of 100 °C. Five specimens were tested for each oxidation temperature to allow for a