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Short communication

In-situ tensile tests under SEM and X-ray computed micro-tomography aimed at studying a self-healing matrix composite submitted to different thermomechanical cycles

Rémi Bertrand [a,b], Olivier Caty [a,*], Vincent Mazars [a,b], Sébastien Denneulin [b], Patrick Weisbecker [a,1], Jérôme Pailhes [c], Gerald Camus [a], Francis Rebillat [a]

[a] Laboratoire des Composites Thermostructuraux, University of Bordeaux & CNRS – CEA – SAFRAN, 3 allée de la Boetie, 33600 Pessac, France
[b] SAFRAN Ceramics, rue de Touban Les 5 chemins, 33185 Le Haillan, France
[c] I2M, Institut de mécanique et d'ingénierie, University of Bordeaux, 16 Avenue Pey Berland, 33607 Pessac, France

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ABSTRACT

Ceramic matrix composite (CMC) based on SiC fibres and matrix is gradually introduced in aeronautical application, mostly in hottest parts of engines. Three dimensional (3D) structured materials are good candidates for complex parts such as turbine blades. Material is submitted to mechanical stresses at high temperatures in oxidizing and corrosive environments for long durations. During thermomechanical cycles, damage, oxidation and healing-phenomenon occur and develop in the material. X-ray computed micro-tomography (μCT) and tensile test under scanning electron microscopy (SEM) are experimental means to study these phenomenons. These techniques are implemented for the understanding of the behaviour of the oxide (solid or liquid) in the crack of the material. The influence of the oxide in the crack was analyzed during tensile test under SEM or μCT. The observation allows to determine the influence of the oxide on the reclosure of the crack during the unloading.

1. Introduction

The field of civil aeronautics seeks for replacing some of the structural components made of metallic alloys presently used in the engines [1,2]. During their use, these parts undergo high temperatures and stresses well over their elastic limit, under oxidizing and corrosive environments. SiC/SiBC composites with their resistance to high temperatures and excellent mechanical properties are candidates for such applications. However, when the applied stress is sufficiently high, a microcrack network is generated within the material. Each of these microcracks represent a path for oxygen to diffuse to the carbon interphase and the SiC fibre. Oxidation of the interphase (which is essential to avoid a brittle behaviour) and of the fibre drastically reduces the lifetime of the material starting from 400 °C [3]. If different types of matrices have been developed to limit oxidation of the interphase [4–6], a matrix that combines CVI-deposited carbides was finally developed with specific sequences of Si, C, and B generating protective oxides for a wide range of temperatures [7]. In particular, boron oxide exhibits a low melting point (almost 460 °C), and an appropriate viscosity which can fill up matrix microcracks at low temperature (almost 600 °C) [8]. This matrix can thus be used to protect the material from room temperature to high temperatures as soon as the temperature of 450 °C has reached the layers with boron oxidize creating self-healing glasses.

Previous studies have shown that this self-healing glass can modify the behaviour of the material according to the thermomechanical cycle applied to the composite [9]. Micro computed tomography (μCT) and tensile tests under SEM give the possibility to generate 3D images and to analyze in situ the material at different resolutions while loading it mechanically and thermally. Some authors have specifically analyzed the behaviour of this type of material via μCT at low temperature [10] and high temperature [11]. These techniques have thus been used to observe and analyze the behaviour and the influence of the oxide present in the microcracks on the material itself.

* Corresponding author.
E-mail addresses: remi.bertrand@lcts.u-bordeaux.fr (R. Bertrand), caty@lcts.u-bordeaux.fr (O. Caty), mazars@lcts.u-bordeaux.fr (V. Mazars), sebastien.denneulin@safrangroup.com (S. Denneulin), jerome.pailhes@ensam.eu (J. Pailhes), camus@lcts.u-bordeaux.fr (G. Camus), rebillat@lcts.u-bordeaux.fr (F. Rebillat).
[1] Deceased.

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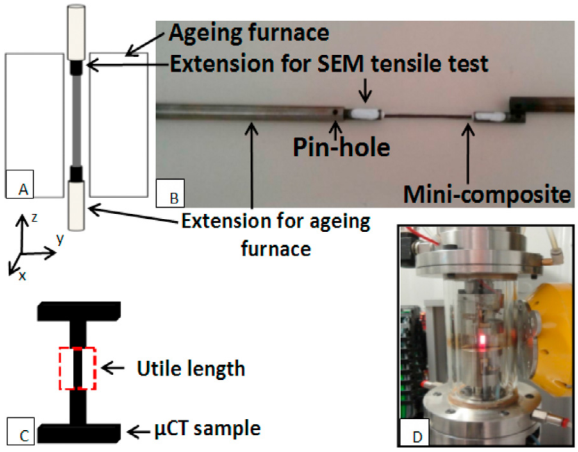

Fig. 1. (A) Schematic representation of the ageing furnace, (B) extension for the ageing furnace and the SEM tensile test, (C) μCT sample, (D) picture of the tension device in the GE/VTomx μCT with a specimen heated at 1000 °C.

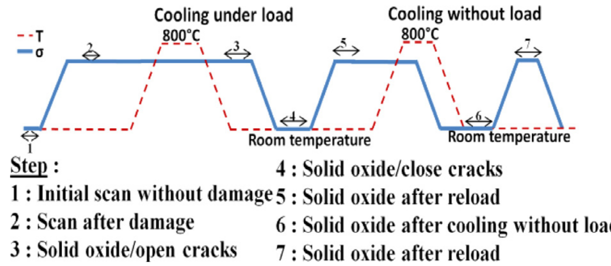

Fig. 2. Schematic description of in situ tension tests performed at high temperature in μCT.

2. Experimental

2.1. Material and devices

The SEM tensile tests were performed using a microtest tensile device (Deben Microtest) equipped with a 1 kN load cell. Tensile macroscopic strain rate are in the range of 0.002–0.04 min^{-1}. The SEM tested material is a so-called mini-composite composed of about 500 unidirectional SiC fibres (Hi-Nicalon fibres) coated with a carbon interphase and 50 μm of [Si–B–C] matrix both processed by chemical vapour deposition. Six samples have been tested.

A special tension device (Fig. 1) has been developed for in situ tension tests in μCT scanners. This device is adapted for small μCT scanners using a hydraulic actuator and a joule effect heater. The tests are performed in a GE-VTomx μCT. The working atmosphere is ambient air and the temperature was measured by a bichromatic pyrometer. This temperature appeared to be stable ($\pm$1%) in the utile length of the sample. This device is designed for small specimen with a section up to 5 mm $\times$ 5 mm and a maximum utile length of 30 mm. It is a 3D structural materials representative of the material used for aeronautical parts. In this paper the utile zone of the sample was 2.5 mm $\times$ 2.5 mm $\times$ 10 mm to fully follow this zone at a resolution of 7 μm. One sample has been tested by in situ μCT.

2.2. SEM and μCT tensile tests

Both tests were designed to analyze healing of the microcracking by a liquid oxide and in more particularly the variation of crack opening due to the quantity of solid oxide present in the crack (depending if the cooling occurs after or before the sample was unloaded).

Prior to being tested under tension in a SEM, each sample has been damaged and oxidized in an ageing furnace (Fig. 2). Samples are loaded at 150 MPa to open cracks. Then heating is applied at 800 °C under air during 6 h. Samples 1, 2 and 3 were cooled down to room temperature with 150 MPa load. For samples 4, 5 and 6, the load is removed before loading. All the samples were then mounted on the tensile stage in the SEM and reloaded in situ at 150 MPa. This reloading was performed at room temperature and environmental SEM atmosphere. During the test, the SEM observations were focused on one of the crack with the aim of following its opening/closing behaviour. Specific extensions were designed to be plugged in the ageing furnace and to hold SEM extensions for tensile tests (Fig. 1). These extensions were unmounted by removing the pins.

The sequences describing the test performed under the μCT device are shown in Fig. 2. A stress of 150 MPa has been imposed in order to damage the material without global failure. The temperature was then raised to 800 °C to liquefy the oxide without reaching the creep temperature of the SiC phases. The sample was initially scanned without load at room temperature (scan 1). Then the load was applied to create microcracks in the material (scan 2) and while the load was maintained, the temperature was increased to oxidize and heal the material (800 °C). After cooling down the sample while the max-load was maintained an image was taken while maintaining the load (scan 3). The load was then removed (scan 4) to analyze possible crack closure impediment. The sample was then reloaded (scan 5) and the temperature was increased to 800 °C to liquefy again the oxide. For scan 6, the load was removed before the temperature in order to analyze microcracks closure when the oxide is in a liquid state. Finally, scan 7 was performed with load being applied again in order to observe the effect of the previous overflow of the liquid oxide out of the crack obtained during step 6.

3. Results

3.1. SEM tensile tests

For SEM samples 1, 2 and 3, after the sample has been thermally treated, the temperature was then decreased down to room temperature, before the load was decreased down to zero. In this case, the solid oxide present in the microcrack impedes its closure. When the load was again applied under the SEM while the sample was at room temperature, a failure in black appeared along the interface between the matrix crack surface in grey and the solid healing oxide in white (highlighted by arrows in Fig. 3A).

Conversely, for SEM samples 4, 5 and 6 when cooling was carried out after having unloaded the sample, the liquid healing oxide was ejected from the microcrack (see the arrow in Fig. 3B). The liquid oxide at 800 °C wets well enough the SiC matrix and can thus coat the composite as soon as it overflows out of the closing crack. When the load was again applied at room temperature, some microcracks appear in the solid oxide layer present inside the crack (see arrows in Fig. 3C). It has also been possible to quantify the crack opening during the tensile under SEM ($\approx$1.8 $\pm$ 0.1 μm, for the crack magnified in Fig. 3B).

3.2. μCT tensile test

Fig. 4A exhibits the evolution of a crack in the middle of the utile section of the sample for the seven loading steps previously described. If, initially, no crack is present in the material (step 1, 0N), scan 2 reveals the crack, generated by loading at room temperature and has evolved during the ageing in air (step 3). After the first thermomechanical cycle (i.e. cooling while the max-load was maintained), the crack was still opened whereas no load was imposed during the scan (step 4), which illustrates the impediment of crack