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In situ X-ray microtomography characterization of damage in SiC_f/SiC minicomposites

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ABSTRACT

The purpose of the present study is to characterize matrix crack propagation and fiber breaking occurrences within SiC/SiC minicomposite in order to validate later on a multiscale damage model at the local scale. An in situ X-ray microtomography tensile test was performed at the European Synchrotron Radiation Facility (ESRF, ID19 beamline) in order to obtain 3-dimensional (3D) images at six successive loading levels. Results reveal a slow and discontinuous propagation of matrix cracks, even after the occurrence of matrix crack saturation. A few fiber failures were also observed. However, radiographs of the whole length (14 mm) of the minicomposites under a load and after the failure were more appropriate to get statistical data about fiber breaking. Thus, observations before the ultimate failure revealed only a few fibers breaking homogeneously along the minicomposite. In addition, an increase in fiber breaking density in the vicinity of the fatal matrix crack was observed after failure. These experimental results are discussed in regards to assumptions used in usual 1-dimensional (1D) models for minicomposites.

1. Introduction

SiCf/SiC composites are prospective candidates for functional uses in future nuclear reactors – such as gas cooled fast reactors (GFR) – because of their favorable mechanical properties at high temperatures and after irradiation. The composites under investigation are made from a 2D fibrous preform composed of the new near-stoechiometric SiC fibers (Hi-Nicalon type S or Tyranno SA3 fibers), using the Chemical Vapor Infiltration (CVI) process. The material exhibits a nonlinear behavior due to the accumulated damage occurring between and inside the woven tows, such as through matrix cracking, fiber/matrix debonding as well as fiber breaking. Thus, a characterization of damage mechanisms within the tow is required to build and validate at local scale a multiscale predictive model. Due to their simple geometry, minicomposites (unidirectional composites containing a single bundle of fibers) are well suited to study these mechanisms. They are also frequently used to optimize the fiber/matrix interphase which dictates the matrix crack deflection along the fibers and consequently the nonlinear behavior of the composite [1–3].

Several 1D statistical models of the evolutional damage have previously been reported [4–11]. They are based on matrix and fiber failure probability laws (such as the Weibull law) and are complemented by a stress redistribution assumption in the vicinity of matrix cracks. These models may lead to satisfactory predictions of the macroscopic response. However, microscopic phenomena cannot be fully validated because of the lack of experimental damage characterization. In fact, if the qualitative damage evolution is accepted, then observations reported in other literature were limited to the sample surface and were mostly collected after the ultimate failure [1,6,12,13].

The purpose of this article is to present an experimental characterization of damage in SiCf/SiC minicomposites under a tensile load using X-ray microtomography. As reviewed by Stock [14], microtomography has been successfully used in material research. In particular, it has been used to study damage or fatigue cracks [15] in several materials such as fiber reinforced metal [16–18] or polymer [19–21] matrix composites, aluminum alloys [22,23] or polymers (PMMA) [24]. However, tomography applied to SiCf/SiC composites has been limited to porosity observations [25–27], crack observations requiring a very high resolution because openings are smaller than 1 μm. In order to investigate matrix crack propagation through SiCf/SiC minicomposite sections, 3D images of a minicomposite under several tensile loading levels

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were acquired using the X-ray synchrotron source provided by the European Synchrotron Radiation Facility (ESRF). These images, reconstructed from a large number of radiographs, have been analyzed to detect matrix cracks within a small volume. Therefore, the morphology and kinetics of crack propagation through the minicomposite section can be described. In order to get statistical data on the fiber failure locations, fiber breaking has also been directly observed using a single radiograph of the entire sample. This was done for a single tensile loading level (about 80% of the stress to failure) and after failure.

2. Material and methods

2.1. Minicomposites

The studied minicomposites were made [3] from a fibrous yarn constructed from 500 Hi-Nicalon type S fibers, with an average diameter of 13 μm. The 100 nm interphase (pyrocarbon) and the SiC matrix were deposited on the fibers using the CVI process. The residual porosity due to the CVI process, and fiber fractions were estimated at 0.12 (±0.04) and 0.58 (±0.09) from polished cross-section pictures (Fig. 1a).

Like the woven composite, minicomposites have an elastic, damageable behavior. Their macroscopic behavior (Fig. 2) follows typical successive steps in accordance with damage evolution [3,12]. The first one is an elastic domain of the minicomposite behavior: no cracking occurs. A second nonlinear step is associated with the matrix cracking (Fig. 1b) until saturation of crack number density (reached for a total strain of about 0.3%). A second linear domain associated with the additional elastic deformation of fibers is then observed after matrix crack saturation. The final step is characterized by a slight non-linearity associated with fiber breaking just before the ultimate failure (close to ≈0.7%).

2.2. Experimental procedure

The in situ microtomography tensile test was carried out on the ID19 beamline at the ESRF, in Grenoble, France. A specific in situ tensile testing machine dedicated to ID19 was used to manually load the specimen (called specimen #1). The minicomposite was glued onto aluminum tabs and had a gauge length of 14 mm. Only the load was monitored using a 500 N load cell. The test was interrupted at six successive loading levels (50, 68, 74, 86, and 92 N) to record the tomography images. The corresponding load levels are reported on the typical load–strain curve presented in Fig. 2, and were obtained from another sample of the same batch with a classical macroscopic device.

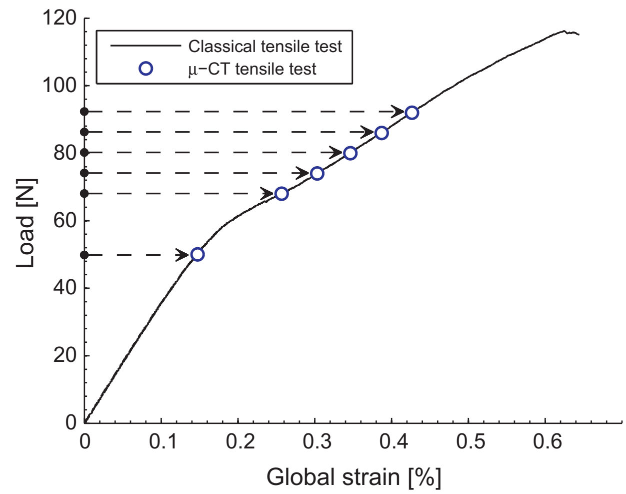

Fig. 2. Macroscopic tensile behavior obtained with a macroscopic device. The loading levels at which the tensile test was interrupted for the μ-CT observations are also reported.

Microtomography consists in recording a set of radiographs acquired at high resolution and at various angular positions of the sample with respect to the X-ray beam [28]. Appropriate algorithms are then used to reconstruct the 3D image from this set of radiographs. A high resolution was necessary to observe both cracks and the microstructure of the tow. Such a resolution with moderate acquisition times could only be reached through a synchrotron radiation, which gives a monochromatic, parallel and high intensity beam. The highest resolution provided on the ID19 beamline at the ESRF is a voxel (volumetric pixel) size of 0.28 μm, identical in all three directions. To observe a 1.65 mm total length, three acquisitions at three successive axial positions were required. In addition, these acquisitions, or scans, were performed at four distinct distances (8, 14, 26 and 36 mm) from the sample to the camera (a Fast Readout Low Noise – FreLoN – 14 bit CCD camera with a resolution of 2048 × 2048 pixels [29]), using a 20.5 keV energy beam. From these four scans, two 3D images were reconstructed from the radiographs recorded at the two shorter distances, using the standard absorption mode (filtered back projection reconstruction) and a third one using the holotomographic mode (based on phase contrast). 3D holotomographic images were reconstructed by combining the four scans [30], using the specific algorithm proposed by Langer [31,32]. Finally, these scans were performed for each loading level (50, 68, 74, 80, 86 and 92 N). In total, the entire experiment required 72 scans (3 fields of view × 4

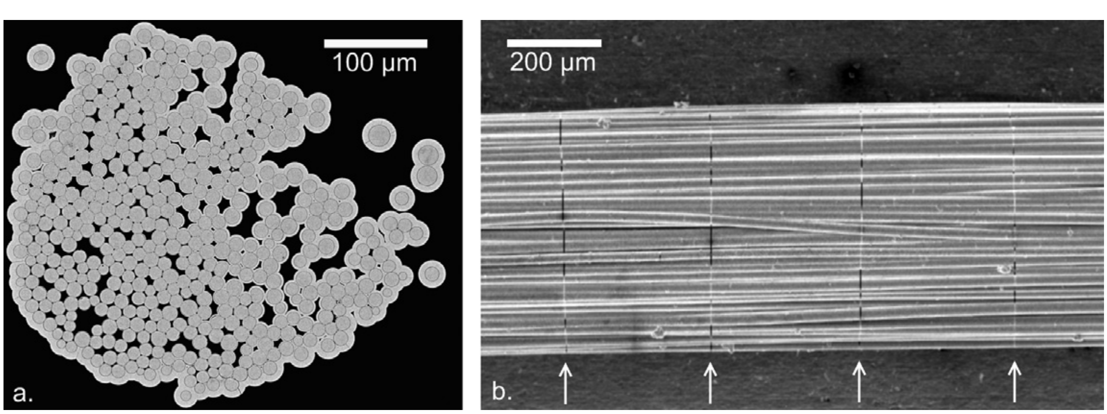

Fig. 1. SEM micrographs of (a) minicomposite polished cross section; (b) cracked minicomposite under tension (75 N), arrows point out matrix cracks.