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Damage investigation and modeling of 3D woven ceramic matrix composites from X-ray tomography in-situ tensile tests

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ABSTRACT

The present paper proposes an investigation of the failure events in a melt-infiltrated SiC/SiC composite. In-situ X-ray microtomography tensile tests were performed at room temperature and at 1250 °C in air. Digital Volume Correlation has been used to identify the damage mechanisms within the material at increasing loads and to propose a damage scenario. Realistic finite element meshes have been constructed from the 3D images to numerically reproduce the experiments at the meso-scale. Elastic simulations exhibit stress concentrations in the planes containing the weft tows. The first cracks and subsequent damage localization were found to appear within these planes thanks to the analysis of the in-situ tomographic data.

1. Introduction

Ceramic matrix composites (CMC) are high performance materials displaying remarkable properties such as damage tolerance, low density and excellent thermomechanical properties at high temperatures. They appear as promising candidates for structural applications at elevated temperatures, e.g. for civil aeronautic engines [1]. CMC are complex materials because of their multi-scale structure and damage mechanisms. This complexity makes the service lifetime prediction required for such applications a real challenge. The understanding of the first failure events and of their propagation is of great interest for improving the design and the architecture of the material, and consequently the thermomechanical behavior. Damage mechanisms of CVI SiC/SiC composites have been largely studied and are well understood, at least at room temperature [2–4]. Experimental studies of damage mechanisms in melt infiltrated (MI) SiC/SiC composite have relied on electrical resistance change [5], acoustic emission and 2D microscopic observations [6,7]. These studies revealed that matrix cracks initiate in weft tows and in the matrix-rich regions. At low stress, microcracks, associated with low energy acoustic signals, develop within the weft tows. When the stress increases, these microcracks propagate until they create through-thickness cracks, associated with high intensity acoustic signals. Crack initiation and propagation within the weft tows have also been observed using Scanning Electron Microscopy (SEM) [8]. This latter study has revealed damage mechanisms such as micro-debondings around the fiber/matrix interfaces and their percolation into a larger matrix crack through the weft tow.

Coupling X-ray microtomography (μCT) and mechanical tests is a promising technique which has been increasingly used in the past decade to visualize and better understand the damage mechanisms within a material [9,10]. In-situ tests are particularly relevant for materials such as CMC to keep the cracks open during acquisition since damages initiate at very small strain levels [11]. To observe the small cracks openings, synchrotron radiation is necessary to obtain a monochromatic, parallel and high intensity beam allowing high resolution and high signal-to-noise ratio. Analysis of the resulting images provide rich 3D information of failure events and have notably been performed for polymer-matrix composites [12–15] and unidirectional ceramic matrix composites at room

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temperature [16–18] and at 1750 °C [19].

Digital Volume Correlation (DVC) can be used on reconstructed X-ray tomographic data to measure three dimensional displacement fields [20,21]. Based on the conservation of the local X-ray absorption coefficient, (i.e. the grey level of the 3D volume), DVC consists in minimizing residuals, defined as the difference between the deformed volume corrected by a kinematic field and the reference volume. In global DVC approaches, displacement fields are interpolated via finite element basis [22], which ensures the continuity of the displacement fields. Therefore, any discontinuity in the deformed image, such as a crack, could be detected through the study of the local residuals. Such analyses have already been conducted in Refs. [23–26] to study the development of cracks. Note that at very small strain level, a regularization approach can be used to improve the DVC results [27].

Three-dimensional images obtained by μCT can be used as input data for finite element modeling (FEM), allowing to account for the exact geometry of the sample. In the case of metal foams, calculated strain and stress fields localizations have been successfully compared to the observed deformation mechanisms under loading at the cell scale [28,29] and the strut scale [30]. FE models based on 3D images are of great interest to provide a physical explanation to the observed damage events and to develop damage laws consistent with experiments. In this study, simulations at the mesoscopic scale have been performed where the yarns are modeled as homogeneous orthotropic materials. This scale seems relevant for image-based modeling from tomographic data since it enables taking into account the reinforcement architecture, yet not being too heavy for FE calculations, as compared to micro-scale simulations. Many authors are currently developing FE models at the meso-scale for composites either by creating a virtual material [31–33] or directly using 3D images [34,35].

The purpose of this paper is to propose a damage scenario of a SiC/SiC at both room temperature and 1250 °C under tensile loading using X-ray μCT. Image processing and DVC residuals are used to identify the damage mechanisms at increasing loads. Elastic FE calculations directly based on the 3D images are also presented to validate the locations of the first cracks initiations within the material.

2. Materials and methods

2.1. Materials

The investigated material is a SiC/SiC composite developed by Safran Group. Fibrous yarns are made of 500 Hi-Nicalon S fibers with an average diameter of 12 μm and woven in a 3D architecture. Fibers are coated with a thin interphase, followed by MI route.

2.2. Experimental procedure

Two in-situ tests have been carried out on the ID19 beamline at the European Synchrotron Radiation Facility (ESRF) in Grenoble at room temperature and 1250 °C using a special tensile device presented on Fig. 1 (a). The specimens have a reduced gauge length of 10 mm, a thickness of 3.2 mm and a width of 2 mm. A hydraulic pump is used to apply the load. Load and displacements are monitored through a load cell and a displacement sensor located respectively on the lower and the upper jaw. Two glass tubes are used to bear the compression load balancing the sample traction. Tests can be performed at high temperatures by heating the samples using Joule effect (Fig. 1 (b)). The sample temperature was measured with a bichromatic pyrometer. Tests were paused during approximately 20 min to carry out the X-ray acquisitions at 0.5 σ_R, 0.7 σ_R and 0.85 σ_R where σ_R is the failure stress of the tested samples at room temperature and at 1250 °C. The tensile strengths of the observed specimens were very close at both considered temperatures but were approximatively only half of the corresponding values for larger macroscopic samples of a similar material [36]. Due to the small size of the specimens, the measured values of strengths should be considered with caution: this will be discussed further later in Section 4.3. The specimen was placed at 190 mm from the CDD camera and a 35 keV beam energy was used. To observe the entire length of the specimen, several acquisitions were performed at a resolution of 0.9 μm for each loading level, each scan containing 2160 projections, in standard absorption mode.

2.3. Image processing

2.3.1. Crack segmentation based on images differences

A coarse crack segmentation is first proposed. Images are stitched together and downscaled by a factor of 2. For each loading step, the rigid body motion between the reference image $f(x)$ and the deformed one $g(x)$ is partially corrected using the normalized mutual information metric [37]. The difference between the reference $f(x)$ and the registered deformed volume $g'(x)$ is computed, $d = f - g'$. As the cracks are mainly located in yz planes where $\vec{x}$ is the loading axis, they could be extracted by performing morphological operations with a horizontal structuring element on each xy slice of $d(x)$. This is followed by a morphological closing with a circular structuring element to retain only the most significant cracks. A manual control of the user is required to isolate the cracks. This coarse segmentation procedure allows to quickly visualize the network of main cracks during loading and to locate the main areas of interest.

2.3.2. Crack segmentation based on DVC residuals

A finer segmentation procedure based on DVC residuals is proposed hereafter. The Correli-C8R code developed by LMT Cachan has been used [25,26], based on a weak formulation with C8 finite elements and a regularized approach [27]. Damage analysis is carried out on small Regions Of Interest (ROIs) of $300 \times 300 \times 300$ voxels extracted in the regions where cracks were observed with the previous coarse segmentation procedure. No rescaling is used here. The size of the elements for the calculation is 20 voxels and the length of regularization is 100 voxels. The residuals $\Phi(x)$ are obtained in each voxel as the difference between the reference image $f(x)$ and the deformed image corrected by the DVC displacement field $g(x + u(x))$. If the displacement field were a single translation, then the residuals Φ would be comparable to the previous difference $d(x)$. Residuals are filtered using a 3D non-local mean algorithm to remove noise.

2.3.3. Yarns segmentation

To separate the weft and warp yarns, image gradients ∇f are first computed using the spatial convolution mask defined in Ref. [38]. Then, we define the structure tensor $T(f)$ of the image in each voxel as the product $\nabla f \otimes \nabla f$. The structure tensor is a 3×3 matrix which can be diagonalized: the eigenvector $\vec{v_1}$ corresponding to the smallest eigenvalue λ_1 indicates the direction of smallest grey level variation which is expected to be aligned with the fibers direction [34,39]. The x and y components of $\vec{v_1}$ are thresholded in order to get masks of the warp and the weft yarns. Morphological operations similar to those used in Ref. [35] are applied to improve the quality of the masks.

2.4. Image-based FEM

2.4.1. FE model construction

A FE model is constructed from the tomographic data to recreate numerically the experimental tests. Only the test carried out at