# APPLICATION OF X-RAY COMPUTED MICRO-TOMOGRAPHY TO THE STUDY OF DAMAGE, SELF HEALING AND OXIDATION OF THERMOSTRUCTURAL COMPOSITES

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**Abstract**
Thermostructural composites are three dimensional (3D) structured materials. Weakening phenomena (mechanical and chemical) are 3D and take place inside the material. X-Ray Computed Micro-Tomography (µCT) is then a recent solution which allows experimental studies of these phenomenons. The technique is applied to the study of failure for mechanical loading and to the oxidation of self healing phases. Crack network was analysed during tension at room temperature and at high temperature (800°C to 1250°C). Crack shape were also studied to determine oxidation and healing processes. Finite element models based on the actual material were built to simulate the thermo-mechanical behaviour. The model geometry is based on µCT images using tetrahedral meshes. The comparison between damage kinetics measured on experiments and stress field calculated is then possible and helps to better understand the relation between material's structure and its behaviour.

## 1. Introduction

Thermostructural composites are generally composed of a woven ceramic or carbon fibre reinforcement and a ceramic and/or carbon matrix. This class of material exhibits interesting thermo-mechanical properties, especially at very high temperatures. Indeed, the intrinsic brittleness of the components is counteracted by crack deviation mechanisms, leading to a material fragilizeation and a controlled progressive damage; the excellent stiffness of the refractory constituents is thus fully utilized [1]. This material is therefore used in strategic and high-tech domains where their thermo-mechanical behaviour must be perfectly controlled.
The development of complex weavings and multilayer matrix have made the material more efficient. In the same time the study and the understanding of its behaviour needs a three-dimensional vision. Micro computed tomography (µCT) gives the possibility of in-situ 3D image and analyse the material at different resolutions while mechanically and thermally loading it, giving access to a very precise insight of damage ([2], [3]). This technique is used here for the in-situ study of damage at different stress states and at different temperatures up to 1200 °C on small samples.
µCT are useful data to better understand the effect of the structure and of the different type of matrix on damage kinetics. To visualise oxidation and analyse the effect of temperature on crack propagation these analyses must be done at high temperature.
To better understand tests, finite element models (FE) based on the actual material architecture were developed. Two modelling strategies are used, either through a virtual reconstruction of the weaving then of the matrix infiltration or by using directly a non structured mesh of images. These models, their development, their validation by comparing with tests will be presented.

## 2. In-situ tension tests at high temperature

### 2.1. Tension and heating device

A special tension (figure 1) device was developed for in-situ tension tests in µCT scanners. This device is adapted for the GE-VTomx µCT and most of the synchrotron beamlines. The design was inspired from the one developed at INSA de Lyon [4] adapted for small µCT scanners using an hydraulic actuator and a joule effect heater. The device is designed for small specimen with a section

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up to 5x5 $mm^2$ and an utile length of 30 mm max. In this paper the utile length of the sample is 2.5x2.5x10 $mm^3$ to fully image this section at a resolution of 7 µm. The maximum load is 5000 N and the maximum temperature available with this specimen and the power supply used (16V/16A) is 1250°C. The device is cooled by air, the temperature is measured by a pyrometer and the temperature is stable (+/- 1%) in the utile length of the sample.

**Figure 1.** picture of the tension device in the GE/VTomx µCT with a specimen heated at 1000°C.

**2.2 Tests**

Two type of tests were carried on the material. First tension tests (0 MPa to failure) at constant temperature (20°C to 1250 °C) were done. As described on figure 2.a, five µCT scans on a GE-VtomX device at a resolution of 7 µm were acquired to study the failure kinetics inside the sample while loading it. The analyse at high temperature allow to compare the behaviour under or without oxidation processes but the comparison is not obvious and the healing phases at this resolution are not always visible. Thus a second type of test where developed to analyse healing and in particular crack blockages due to the healing phases and healing phases expulsions. This test is described on figure 2.b. First the utile length of the sample is scanned without load at ambient temperature (scan 1). Then a load is applied to open few failures in the material (scan 2). Remaining load, the temperature is raised to oxidise the material and liquefy healing phases (scan 3). Temperature is back down to room temperature (RT) while maintaining load to observe oxidation consumptions (scan 4). Then the load is removed to analyse crack blockages (scan 5). Crack are reopen (scan 6) and the temperature is raised to 900°C to liquefy healing phases. Then load is removed before temperature to analyse healing phases expulsions (scan 7). Load is again applied to observe effect of this expulsion on failure morphology (scan 8).

**Figure 2.** Shématic description of in-situ tension tests at high temperature carried out to study the damage and healing of the material

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### 2.3. Image treatments

3D images obtained must be treated to analyse and identify new failures or crack propagations. The goal of the treatment is to reveal the structure and to compare images of the same sample at different temperature and/or loading rates. The initial image (Figure 3.a) is first approximately manually realigned then a automatic registration is applied using FEI/avizo [5] software to ensure all images match together (figure 3.b). Then a non local mean filter is applied to improve the contrast of the texture of the image and in particular to reveal cracks (figure 3.c). The difference between images (figure 3.d) at different states displays only new events like new cracks, crack propagation, oxidation processes or healings.

**Figure 3.** Image treatment steps to improve images acquired on the lab. µCT. (a) initial image loaded at 190 MPa, (b) after manual realignment and automatic registration, (c) after application of a non local mean filter, (d) difference between the initial state at 0MPa and the state at 190 MPa.

## 3. Results

### 3.1. Crack network

New failures are isolated from the initial texture (often pre-existing failures) making the difference between images at different loading states (see figure 4) or temperature states. It is then possible to visualise cracks in 3D inside the composite and study their evolution vs. temperature and load. In this case failure are mostly perpendicular to the loading and begin on a side of the sample. At high temperatures failures are less straight as it will be presented during the session.

**Figure 4 :** (a) 3D rendering of a part of a SiC/SiC sample loaded at 190 MPa. (b) 3D visualisation of the cracks and of their development in the sample volume.

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### 3.2. Oxidation and healing

Comparing two states acquired using the second procedure described in figure 1.b, the effect of the healing is more obvious. Figure 5 illustrates the evolution of a crack observed on scan 2 (150 Mpa, RT, no oxidation) and scan 4 (150 MPa, RT, 2h of oxidation at 800°C under air). The composite sample is in grey, crack in yellow and the healing oxide created between the two states is in white. The failure is now almost filed by the healing oxide and the composite is protected by this phase.

**Figure 5 :** µCT 3D rendering (a) of a crack (yellow) inside the sample (grey) (b) of the crack (yellow) and the healing oxide (white) created à 800°C under air with 150 MPa of loading.

### 3.3. Models

To better understand crack initiation and propagation, FE models were developed using µCT 3D images to build the mesh of the sample. Images are thirst threshold then a marching cubes algorithm is applied to mesh the surface of the material. The very thin mesh obtained is decimated and with an advancing front algorithm the structure is meshed in 3D with tetrahedrons. The software used were Avizo Fire [5]. This mesh is imported in Abaqus FE software to apply the tensile test boundary conditions, material properties and run the computation. In figure 6.b and c, material properties are isotropic, but as describe in the next section new models are under development to use orthotropic material properties with directions directly measured on images. Despite this simplification, stress concentrations are determined (figure 6.c) and some defects are detected on the border of the sample (figure 6.d) near the final break (see figure 6.a).

**Figure 6 :** (a) µCT 3D rendering of the sample after failure. (b) FE model base on µCT images loading in tension, and (c) a zoom on the failed zone, colormap of S11. (d) a µCT slice showing the defects that create the stress concentration.

As described in figure 7, new models will be presented during the ECCM17 session. One based on images with an advanced threshold of the image to isolate warp/weft yarns individually. This complex segmentation is then meshed and orientations are directly determined on images using the structure tensor of the image (figures 7.c and d). The second strategy is based on a virtual reconstruction of the composite using 3D µCT images to determine the weaving of a representative volume.

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**Figure 7 :** Illustration of the two strategies developed to model the material. (a) Original µCT used for modelling, (b) treatments done on the µCT image to reveal the structure. Image based strategy using an (c) & (d) automatic 3D detection of the yarn directions, visualisation of the results respectively on the warp and weft patterns. Strategy based on a virtual material and comparison of (e) the uCT and (d) the virtual mesh.

## 4. Conclusion

3D µCT images are used to analyse crack initiation and propagation in self healing 3D CMC. These analyses were made in-situ using a high temperature tension device specially developed for this material and for µCT. Two type of tests were carried out. One to analyse crack propagation at ambient and high temperature and one to observe the behaviour of healing phases submitted to load and temperature. Images presented were treated to better analyse cracks and phases. Cracks are rather straight and perpendicular of the load at ambient temperature but located on a side of the sample. Most of the time they don't propagates through the whole section. At high temperature the crack is more winding. Healing oxide were also observed and can block the failure or be ejected depending on its state while removing load. To understand all these observations, models based on the actual structure of the material were developed. Analyses were done on a simple model but more accurate solutions were developed. One is an image based model with complex segmentation and actual measurements of orientations and one is a virtual material model that uses the weaving identified on images.

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