



# X-ray computed tomography analysis of damage evolution in open hole carbon fiber-reinforced laminates subjected to in-plane shear



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## ABSTRACT

The deformation and damage mechanisms during tensile deformation of  $[\pm 45]_2$ s open hole carbon fiber composite materials were quantitatively analyzed by means of image analysis of XCT data. Damage developed progressively in each ply with deformation in two shear bands (parallel and perpendicular to the fiber direction) at  $\pm 45^\circ$  with respect to the loading direction, i.e. cracking started from these shear bands and grew following the fiber direction until encompassing the whole laminate width. Matrix cracking and fiber rotation in both types of shear bands presented different features, which were quantified by means of image processing from XCT data. A small delamination developed around the hole at the intersection from  $+45^\circ$  and  $-45^\circ$  cracks directions in adjacent plies. However, this delamination does not progress excessively with deformation until the end of the test, when fracture was triggered by the sudden propagation of two main delamination cracks tangent to opposite sides of the hole in the shear band parallel to the fiber direction.

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## 1. Introduction

Fiber-reinforced polymers present different physical failure mechanisms depending on the orientation of the stresses with respect to the fibers, leading to the final failure. Nevertheless, the failure strain is always small (below a few %) except in the case of in-plane shear deformation, where the strain-to-failure is an order of magnitude higher and the shear stress-shear strain curve is highly non-linear [1]. The shear non-linearity –which has important implications on the development of constitutive models for composites [2]– comes about as a result of the development and interaction of various deformation and failure mechanisms, which have been studied in detail, e.g. Refs. [3–7].

In plane shear deformation presents three different regimes: an initial elastic region that leads to a plateau, which is followed by a third region in which the shear stress increases linearly with the applied shear strain until fracture. X-ray tomography provided a very detailed pictured of the sequence of damage during

deformation [7]. Matrix cracking was first detected at the end of the linear region and started in the external plies. It progressed rapidly with the applied strain and reached saturation at the end of the plateau in the external plies. The minimum crack density at this stage was found in the inner plies but it increased during deformation and reached the maximum values in these plies at the end of the test. Edge delamination appeared at the end of the plateau at the outermost interfaces and undergo unstable propagation at the end of the linear hardening region, leading to the specimen failure.

Fiber rotation was independent of the ply position within the laminate and started at the beginning of the plateau region. The fiber angle between consecutive plies decreased linearly with the applied strain until the end of the test and was responsible for the linear hardening region.

The objective of this investigation was to ascertain the influence a stress concentration on the deformation and damage using the same methodology. Notches or holes induce strain gradients and it has been shown that the behavior of the laminate depends on different factors such as laminate size and thickness, notch size and geometry and ply orientation and thickness [8,9]. Lagace analyzed  $[0/90]_2$ s laminates and found that failure mechanisms changed

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from matrix-dominated to fiber-dominated as the hole diameter increased [10]. Awerbuch and Madhukar [11] summarized previous research and pointed out that notch sensitivity was highly influenced by the damage that occurred at the notch edge. Subsequent studies performed in  $[0/\pm 45/90]_{2s}$  laminates concluded that damage started in the  $90^\circ$  plies before propagating to the  $45^\circ$  plies and final fracture took place by fiber fracture in the  $0^\circ$  plies. However failure in  $[0/\pm 45]_{2s}$  laminates was caused by fiber-matrix shearing and fiber breakage [12]. More recently, Hallet et al. [13,14] analyzed size effects on the strength of notched composites by varying systematically the hole diameter, the number of plies and the total thickness of  $[45_m/90_m/-45_m/0_m]_n$  laminates. These studies demonstrated that changes in  $m$  (thickness of the block plies) or  $n$  (thickness of the sublaminate) led to dramatic changes in the failure mode ranging from brittle failure caused by fiber breakage without sub-critical damage, to failure caused by delamination or, to failure caused by pull-out of fibers with massive sub-critical damage [14].

The evolution and interaction of the different damage mechanism, which are critical to understand the macroscopic behavior, were obtained in previous studies using different techniques. For instance, Pierron et al. [8,15] used full-field optical measurements, while Hallet et al. [13,14] monitored damage in various specimens loaded up to different strain levels using radiographs in combination with a zinc-iodide dye-penetrant solution. These techniques have enough resolution but are either limited to the surface or to averaged 2D information through the thickness of the laminate, respectively. However, X-ray tomography (XCT) is able to give actual 3D information of the damage and microstructure in every ply from a number of X-ray radiographs obtained at different angles. The three dimensional and non-destructive nature of this technique allows tracking the damage evolution, thus providing very detailed information about the initiation, propagation and interaction of different damage mechanisms [7,16–19]. Moreover, new algorithms for automated tracking [7] can provide quantitative data, instead of qualitative assessments, of the progression of deformation and damage features (e.g. fiber rotation, matrix cracking). The main objective of this investigation is to use these novel quantitative XCT tools to analyze the damage evolution of open hole  $[\pm 45^\circ]_{4s}$  laminates deformed in tension. The results were compared with previous data obtained in plain coupons to ascertain the effect of the stress concentration induced by the hole on the failure processes.

## 2. Experimental

### 2.1. Material

The samples for the mechanical tests were obtained from panels of  $300 \times 300 \text{ mm}^2$  and 1 mm in thickness manufactured in autoclave from T800S/M21 (carbon fibers/epoxy resin) prepreg sheets with a stacking sequence of  $[\pm 45]_{2s}$ . A standard autoclave cure cycle was applied with a maximum cure temperature of  $180^\circ \text{C}$  for 120 min and a pressure of 7 bar [20]. The heating and cooling rates were set to  $2^\circ \text{C}/\text{min}$ . The ply thickness was approximately  $125 \mu\text{m}$ . The different plies will be named ply 1 to ply 7, being ply 1 and 7 the outermost plies and ply 4 the central one with twice the thickness. The nominal fiber volume fraction after consolidation was 57%.

Five specimens of  $200 \times 20 \times 1.0 \text{ mm}^3$  (length  $\times$  depth  $\times$  thickness) were machined from the laminates with the external plies at  $+45^\circ$ . The specimens were very carefully cut by milling using a hard metal tool with drill end and cooling liquid. A circular hole of 4 mm in diameter was carefully machined in the middle of every sample using a standard drill. Fiber glass composite tabs were fixed to the specimen's borders to avoid

damaging the samples with the jaws of the mechanical testing machine. The distance between tabs was 100 mm.

### 2.2. Mechanical characterization

The mechanical tests (tensile and cyclic loading tests) were carried out in an electromechanical universal testing machine (Instron 3384) at a constant cross-head speed of 0.6 mm/min. The load was monitored with a 10 kN load cell, while strains in the tensile and cyclic loading tests were measured with an extensometer with 50 mm gage length placed in the center of the samples. Additionally, a commercial digital image correlation (DIC) system in combination with an artificial speckle pattern was used for strain monitoring. Both, extensometer and digital image correlation systems, provided similar results over the whole sample for the far field strains but the DIC also provided the local strain maps in the outer surface of the specimens. The shear stress and shear strain were computed following the ASTM D3518/D3518M-94 standard [21].

Three specimens were monotonically loaded until failure and one was periodically unloaded and reloaded at different strains to determine the evolution of the elastic modulus with deformation. Finally, another specimen was loaded up to selected strain states. The test was stopped at each strain state, and the specimen was immersed in a dye penetrant liquid during 30 min while holding the displacement constant. There is some unavoidable stress relaxation in this procedure, which was not checked. However, even if there is some relaxation, the cracks remain open and the dye penetrant can infiltrate into them. This procedure enhances the contrast between the cracks and the composite material. The liquid was composed of 60 g of ZnI in 10 ml of water, 10 ml of ethanol and 10 ml of Kodak Photo-Flo 200. After each strain step, the specimen was removed from the tensile machine and inspected by XCT as detailed below. Afterwards, the sample was reloaded in the tensile machine up to the next step, and the procedure was repeated again for each strain state. The liquid was dried by evaporation before XCT inspection. While some of the liquid might have squeezed out from the cracks once the load was released, some of the ZnI particles remained attached to the crack surface providing the necessary contrast for tomography. In addition, an inspection in one sample with undried liquid (just after the load was released) and with dried liquid (after several days) was performed to check the possibility of contrast fading due to this effect. The results of the reconstructed volumes were the same. It should be noticed that the shear stress-strain curves from different specimens were practically superposed, indicating that neither periodic unloading nor liquid immersion modified the laminate properties.

### 2.3. X-ray computed tomography

The spatial distribution of the deformation and failure mechanisms was studied by XCT using a Nanotom 160NF (General Electric-Phoenix). The tomograms were collected at 90 kV and 100  $\mu\text{A}$  using a tungsten target. For each tomogram, 2000 radiographs were acquired with an exposure time of 750 ms. Tomogram voxel size was set approximately to  $10 \mu\text{m}/\text{voxel}$ . The tomograms were then reconstructed using an algorithm based on the filtered back-projection procedure for Feldkamp cone beam geometry. The damage in the reconstructed volumes was qualitatively and quantitatively analyzed using the freeware ImageJ software, subroutines written in Matlab and VGStudio Max 2.2 commercial software. Accurate quantification of crack number, density and degree of rotation was possible because of the use of a dye penetrant liquid containing ZnI. The dye caused the cracks to appear brighter in the tomograms due to the higher X-ray absorption coefficient of ZnI as

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