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Automatic quantification of matrix cracking and fiber rotation by X-ray computed tomography in shear-deformed carbon fiber-reinforced laminates

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ABSTRACT

The deformation and damage mechanisms of carbon fiber-reinforced epoxy laminates deformed in shear were studied by means of X-ray computed tomography. In particular, the evolution of matrix cracking, interply delamination and fiber rotation was ascertained as a function of the applied strain. In order to provide quantitative information, an algorithm was developed to automatically determine the crack density and the fiber orientation from the tomograms. The investigation provided new insights about the complex interaction between the different damage mechanisms (i.e. matrix cracking and interply delamination) as a function of the applied strain, ply thickness and ply location within the laminate as well as quantitative data about the evolution of matrix cracking and fiber rotation during deformation.

1. Introduction

The maximum load carrying capacity of multiaxial fiber-reinforced laminates (either in tension or in compression) is normally attained at low strains and is dictated by the fracture of the plies with the fibers oriented along the loading axis. This behavior often masks that off-axis plies within the laminate (deformed in shear) show a strong non-linear behavior and can withstand very large strains before fracture [1–3]. This shear nonlinearity influences the mechanical behavior of laminates [4,5] and the mechanisms of shear deformation in composites have been studied in the past.

Matrix cracking in the fiber direction is the first failure mechanism during tensile or compressive deformation of off-axis plies in a laminate, as it was documented in $[0/45/-45/90]_s$ $[0/90/-45/45]_s$ $[\pm45/90]_s$ $[90/\pm45]_s$ and $[0/\theta/0]$s carbon and glass/epoxy laminates [6–11]. Matrix cracking leads to a reduction in stiffness and the relation between both was modeled by Kashtalyan and Soutis [12] in $[0/\theta]_s$ laminates. In addition, fiber rotation also plays an important role in the non-linear behavior of $[\pm45]_s$ laminates [13] together with interply delamination, which results from extensive cracking of adjacent plies with different fiber orientation.

While these mechanisms are well documented from the qualitative viewpoint, quantitative data on the density of matrix cracks, the extent of interply delamination and the changes in fiber orientation with strain are very limited. Optical or electron microscopy of cross-sections is very time consuming while X-ray radiography only provides 2D information and it is not possible to separate the contributions from each ply [14,15]. These limitations can be overcome with the use of X-ray computed tomography (XCT), which provides actual 3D information of the damage and microstructure in every ply from a number of X-ray radiographies obtained at different angles. In addition, it is a non-destructive technique that can be used for tracking the damage evolution in different scenarios. For instance, Wright et al. [16] studied with synchrotron tomography the interaction of damage mechanisms in a centrally notched specimen made from a carbon/epoxy laminate $[90/0]_s$ and showed through-thickness fiber breakage and intralaminar cracking in close detail. In another study, the crack evolution in a double edge notched carbon/epoxy laminate $[90/0]_s$ was determined [17] and the observations were used to set the ground for enhancing the damage prediction of a three-dimensional finite element model. Sket et al. [18] has investigated the onset and evolution of the damage in three dimensions by XCT in a notched glass fiber/epoxy cross-ply laminate subjected to

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three-point bending and this information was used to determine the effective fracture resistance curve of the cross-ply laminate. Finally, Hernández et al. [19,20] have used XCT to study the effect of curing parameters on the void volume fraction, shape and spatial distribution in C/epoxy laminates manufactured by compression molding.

Within this framework, this investigation was aimed at developing automatic analysis methods capable of tracking matrix cracking and fiber rotation from tomographic data. This methodology was used to quantify the development of damage during shear deformation in each individual ply of $[\pm 45]_{2s}$ carbon fiber laminates. The evaluation is focused on the stresses necessary to nucleate cracks, the evolution of crack density and rotation of the fibers as a function of the strain.

2. Materials and mechanical characterization

The samples for the mechanical tests were obtained from panels of $300 \times 300\ mm^2$ and 1 mm in thickness. Unidirectional carbon fibers/epoxy resin T800S/M21 prepreg sheets were supplied by Hexcel®. The T800S carbon fiber (produced by Torayca®) have a nominal diameter of 5 μm and 24 K fibers per bundle. The panels were manufactured in autoclave from T800S/M21 (carbon fibers/epoxy resin) prepreg sheets with a stacking sequence $[\pm 45]_{2s}$. A standard autoclave cure cycle was applied with a maximum cure temperature of 180 °C for 120 min and a pressure of 7 bar [21]. The heating and cooling rates were set to 2 °C/min. The ply thickness was approximately 125 μm. In this paper, 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% and the resulting thickness was 1.0 mm.

Five specimens of $200 \times 20 \times 1.0\ mm^3$ (length × depth × thickness) were machined from the laminates with the external plies at +45°. The specimens were very carefully cut by milling using a hard metal tool with drill end and cooling liquid. Very little damage was introduced during machining. Fiber glass composite tabs were fixed to the specimens borders to avoid damaging the samples with the jaws. The distance between tabs was 100 mm. Tensile tests to determine the shear response were carried out in an electromechanical universal testing machine (Instron 3384) at constant cross-head speed of 0.6 mm/min. Load was monitored with a 10 kN load cell. The strain was measured with an extensometer with 50 mm gage length. In addition, strains were monitored by digital image correlation (DIC). An artificial speckle pattern was created with black and white paints in order to monitor de displacements on the specimen's surface. A commercial DIC system from Correlated Solutions, model VIC-2D 2009 was used for this purpose. Images with a 2452×2056 pixels definition were stored with an 8-bit format and the processing system allowed to measure surface displacements with a spatial resolution of ≈80 μm/pixel through a CCD camera (stingray F-504 from Allies vision technologies). Both techniques provided equivalent results. The shear stress and shear strain were obtained according to the ASTM D3518/D3518M-94(2001) Standard Test Method for "In-plane shear response of polymer matrix composite materials by tensile test of a ±45° laminate".

Two specimens were loaded monotonically until failure and one was periodically unloaded and reloaded at different strains to determine the evolution of the elastic modulus with deformation. Finally, another two specimens, denominated S1 and S2, were loaded up to a given strain. The tests were stopped and the specimens were immersed in a dye penetrant liquid during 30 min while holding the displacement constant to enhance 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. The specimens were removed from the machine and inspected by XCT as detailed below. Afterwards, the samples were taken back to the testing machine and reloaded to the next step. The whole procedure was repeated several times in both specimens. It should be noticed that the shear stress–strain curve of all the specimens were practically superposed, indicating that neither periodic unloading nor liquid immersion modified the laminate properties.

3. X-ray computed tomography

The spatial distribution of the deformation and failure mechanisms was studied by XCT using a Nanotom 160NF (Phoenix). The tomograms were collected at 90 kV and 100 μA 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 μm/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 and subroutines written in Matlab. Accurate quantification of crack number, density and degree of rotation was possible because of the use of a dye penetrant liquid containing ZnI which caused the cracks to appear brighter in the tomograms due to the higher X-ray absorption coefficient of ZnI as compared with the carbon fibers or the polymeric matrix.

3.1. Automatic evaluation of fiber rotation

An automatic procedure was developed to analyze damage development (fiber rotation and matrix cracking) once the pre-processing of one sample set was complete, including the tomographic volumes obtained after deforming the specimen at different strain levels. The procedure was performed in two steps.

The evaluation of fiber angles in non-cracked specimens was determined through the evaluation of fiber bundles which were visible when cracks were not present. The identification of single fibers was not possible since the resolution used for these relatively large specimens was not high enough. Rotation angle evaluation in cracked specimens was based on the rotation of intralaminar matrix cracks, which are inherently linked to the rotation of fibers. In the present case, the specimens were scanned after unloading and therefore the fiber rotation corresponds to the contribution of the inelastic strain (a combination of damage and plastic strain). However, the elastic contribution to fiber rotation at a certain point of the stress–strain curve can be calculated considering the actual elastic properties which can be obtained from periodic unloading tests.

Fiber bundles (or cracks) were segmented and fitted in 2D with ellipses of equal area and moment of inertia. Using this approximation, the semi-axes of the fiber bundles can be determined from the eigenvalues of the moment of inertia tensor (principal moments of inertia). The orientation of the each fiber bundle (or crack) was characterized by the angle between the principal axes of the equivalent ellipse (corresponding to the minimum principal moment of inertia) and the loading direction (reference axis). The average of the orientation angle of all the fiber bundles (or cracks) was used as the absolute fiber orientation of the ply. In order to avoid errors arising from the small misalignments during sample rotation to the reference axis, the difference of the average orientation angles between two consecutive plies was computed and followed through the different loading stages. In this way it was possible to automatically evaluate the changes in orientation in fiber bundles and