# MODELLING CRACK NETWORK IN LAMINATED COMPOSITES USING COMPLEMENTARY OBSERVATION TECHNICS

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**Abstract**
The design of liner-less composite pressure vessels for spatial launchers requires studying the relation between damage and permeability in laminates in order to offer solutions which fulfil strength and leak rate requirements. In this paper several experimental methods based on optical microscopy and micro-tomography observations under tensile loading are proposed. These methods aim at evaluating the interactions and patterns of damage in different plies in terms of cracking threshold, crack length and relative location. Edge effects are also analysed in order to extract relevant damage density. Results obtained for several carbon epoxy laminates manufactured by Automated Fibre Placement (AFP) are presented.

## 1. Introduction

This work deals with propellant tanks (LOX, $LH_2$, $LCH_4$) and spherical pressurization vessel (He) used in launcher engines. According to the stored fluid, materials may be subjected to severe thermomechanical loadings, and fulfil leakage and chemical compatibility specifications. Those numerous requirements make light alloys very good candidates for tanks. Thanks to their high specific strength and their ability to propose made-to-measure solutions, composite materials can be interesting candidates, but they imply the insertion of a liner to address leakage specifications. The liner, made of titanium alloy or polymer, penalises the mass and the manufacturing cost of the tank. The development of composite liner-less pressure vessel is therefore a way to improve mechanical and industrial performances of space launchers.
One challenge when designing a liner-less vessel is to reach the permeability requirement with the composite wall itself. Pristine composite laminates meet the permeability requirement, but as those materials are heterogeneous, damage growth may occur in cases of low thermo-mechanical loads. Transverse cracks and micro-delamination in adjacent plies grow and may connect together (Figure 1), resulting in a leakage network through the composite wall [1,2]. Crack on-set and coalescence are driven by several design parameters such as the type of composite material (fibre and matrix), the thickness of the plies (which has an effect on cracking threshold [3,4]) and the laminate thickness and layup (choice of plies orientation). So, the design of a liner-less vessel need a predictive model able to manage all these design parameters regarding their interacting effects on damage propagation and the strength of the tank. In view of the number of design combinations, the model should be wrote at the ply scale in order to control the calculation time produced by the optimisation loop.
Many experimental or numerical studies aimed at taking into account damage mechanisms in structural analysis [5–9]. However, these studies address stiffness and strength loss due to damage at the ply scale without dealing with crack interactions and the morphology of crack network. Indeed,

crack interactions between plies have a low influence on stiffness and strength loss, while they are crucial to study the appearance of the first leak paths.

In the present work, several experimental methods have been developed to characterise the crack network according to the load level and the load direction compared to reinforcement orientation. Main results are presented with a view to build a predictive meso-model.

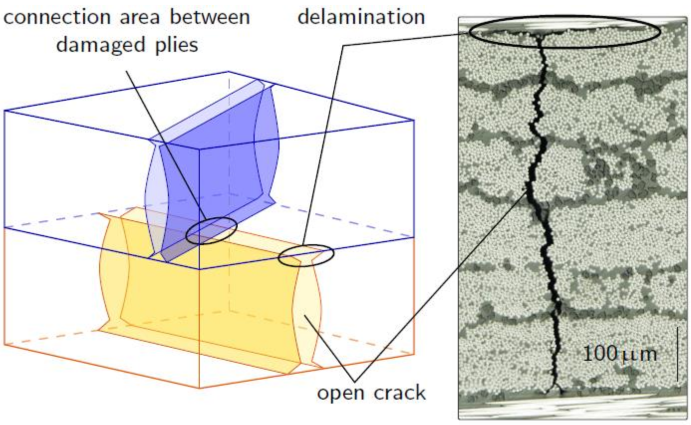


**Figure 1.** Transverse crack and delamination: crack network in two damaged plies and micrograph of one transverse crack with delamination at crack tip

## 2. Experimental methods

### 2.1. Material and manufacturing process

Plate samples were manufactured by Automated Fibre Placement (AFP). This process is increasingly used, especially in aeronautics, mainly because it makes possible to precisely steer the fibres in any position and direction on the mandrel. Thus, this process gives more freedom in terms of shapes than filament winding. For more details on this manufacturing process, the reader may refer to reference [8].

The structure is similar to that of unidirectionnal composite laminates. Damage mechanisms, and consequently the majority of damage models developed for composite laminate as well as characterisation methods are appropriate to describe structures manufactured by automated fibre placement. The material is carbon fibre T700 and thermoset matrix M21. The thickness of the elementary layer is 0.26 mm. Several layers of the same orientation can be stacked together in order to obtain a thicker ply, e.g. in a $[0_2/90_3/0_2]$ laminate the thickness of the 90° ply is 0.78 mm.

### 2.2. Optical microscopy under tensile loading test

Transverse cracks are generated by transverse stress (mode I) and shear stress (mode II). This requires testing different laminates in order to characterise damage growth in the ply in both modes or in mixed mode I + II. In this study, three different laminates were tested: $[0_2/90_n/0_2]$, $[+45/-45]_{2s}$ and $[0/+67.5/-67.5]_s$. The effect of ply thickness is also studied by making the number $n$ vary from 1 to 3.

In the present work, the assessment of the damage densities was carried out by optical microscopy for several loading levels. Tensile test were performed on specimens polished on one edge. Transverse crack and delamination at crack tips were identified with a travelling optical microscope (Figure 2(a) and Figure 2(b)). The observation area was chosen quite large, i.e. about 100 mm, in order to evaluate statistical effects.

### 2.3. Cross section examinations by optical microscopy

A specific protocol was applied in order to bypass the issue of free edge effects. Observation of the surface under loading was combined to cross-section examination : polishing of the edge was performed after loading in order to remove from 15µm to 2mm of the edge surface and observe the damage state in bulk. After polishing, the sample was loaded to a lower level so that damage did not propagate but cracks opened and became more easy to distinguish. Those steps were repeated for increasing maximal loading to obtain the evolution laws of damage in bulk.

Beside crack growth characterisation, performing several cross-section examinations through the width of a specimen also allows to study the arrangement of cracks in three dimensions.

### 2.4. X-ray microtomography under tensile loading test.

X-ray tomography provides non-destructive access to the internal structure of the material. It is particularly applicable to crack detection due to the density difference between the material itself and the air contained in the cracks. However, its implementation is not trivial: cracks are not easily visible when they are closed, and increasing the resolution of images involves reducing the size of the area observed. To maintain an observed area of reasonable size with respect to the crack network, for example an observable length of 10mm, the size of the voxel cannot be less than 6µm in accordance with the resolution of the detector used. In the case of a single ply, which is the most critical from this point of view, the cracks are open from 0.1 to 2µm in a stress free specimen, and from 2 to 10µm when the specimen is loaded at a deformation of 0.75%.
To keep the cracks open during observation, an integrated tensile device was developed for the Phoenix V/TOME/SX, GE microtomograph. This device is presented in the tomograph in Figure 2 (c). It consists of two jaws placed in aluminium alloy tubes. The upper jaw can be moved vertically, its position in the tube is controlled by the clamping nut, while the lower jaw is fixed vertically but free to rotate. An X-ray transparent PMMA tube is positioned between the two aluminium alloy bases. The upward movement of the movable jaw generates a tensile force in the specimen and a compression force in the PMMA tube. The loading progressiveness is ensured by a series of spring washers. To avoid torsion in the specimen, the nut is tightened by holding the assembly only by its upper part, the lower parts being free to turn. The specimen is equipped with a gauge, and a self-contained housing allows the deformation to be measured throughout the tensile setting as well as during observation.
The device allows to carry out observations under loading with a voxel size going down to 5µm and by applying up to 12kN to a specimen of length between 200 and 250mm and a maximum width of 25mm. For the laminates studied with two 0° plies and specimen widths greater than 10mm, the maximum deformation that can be applied is about 0.9%.

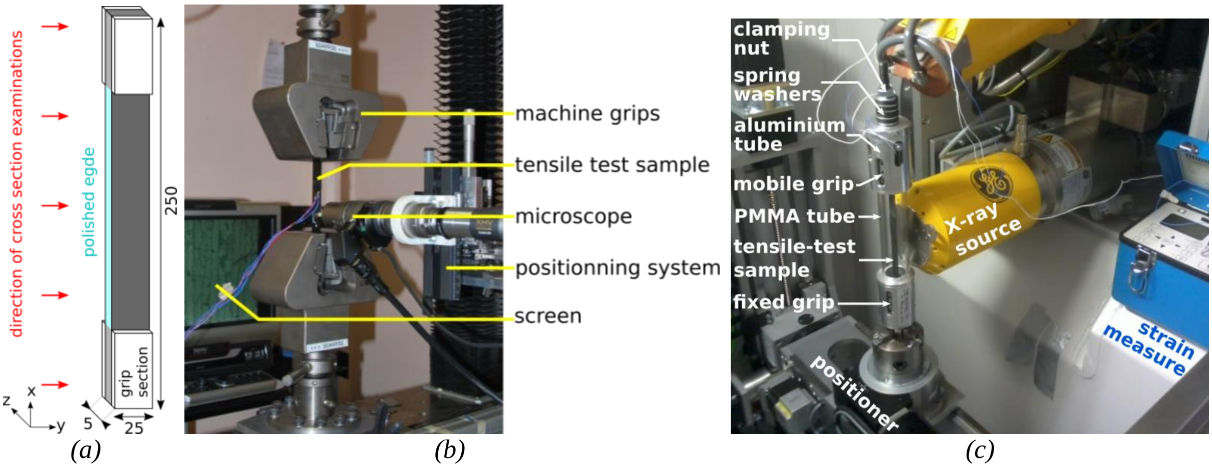


**Figure 2.** (a) Dimensions of specimens, (b) Damage observation under tensile loading test, (c) Observation device under traction loading placed in tomograph

## 3. Results

### 3.1. Edge effects and crack densities

Optical microscopy under tensile loading test and cross section examinations were carried out on specimens of $[0_2/90_n/0_2]$ and $[+45/-45]_{2S}$ layups in order to evaluate damage densities. All the results can be found in [10], only the main conclusions are presented in this section.

Cross section examinations on cross-ply laminates $[0_2/90_n/0_2]$ show that, independently of the ply thickness, the micro-delamination rate tends quickly to zero when we explore the depth of the material. Micro-delamination generally observed at crack tips of transverse crack seems to be mainly due to edge effect at least for the studied material. Cross section examinations were continued to verify the length of transverse cracks through the width of the specimen. Cracks were shown to be continuous for double and triple 90 plies, while a few cracks disappeared in the $[0_2/90_1/0_2]$ lay-up. This variation of crack rate for the simple ply is about 15% of the crack rate at the surface.

The $[+45/-45]_{2s}$ layup was loaded in order to introduce a shear strains up to 4%, polished and then observed. For the highest strain and after removing 1.5mm, only a few transverse cracks remained in the central (and double) ply and in external plies. Deeper, after removing 3mm of the surface, no crack remained at all. To exclude the eventuality of cracks being too closed to be observable, the sample was observed under loading. The results demonstrates that no transverse cracks nor micro-delamination occur in the bulk under shear load.

These results can be due to the use of a toughened matrix (M21) with thermoplastic nodules. For another material, e.g. with a more fragile matrix, delamination would likely be lower in bulk than at the surface but may persist in bulk. Although no meso-damage is observable in $[+45/-45]_{2s}$ laminates, this layup nevertheless undergoes irreversible strains and stiffness loss due to diffuse damage at the micro (fibre) scale. However, if shear loading alone does not lead to transverse cracking in this material, the shear component associated to a transverse loading will likely contribute to crack growth.

Concerning the evolution of transverse crack densities, optical microscopy under tensile loading test on cross-ply laminates makes it possible to highlight the effect of ply thickness on cracking threshold and rate widely described in the literature [3,4]. But the goal of these tests was to take advantage of observing a large enough and hence representative area to study the effect of the variability of the material properties. Depending on the piece of sample one chose to focus on, initiation and slope of crack growth may be very different, at least concerning the beginning of crack growth. It is likely due to weak areas (defect locations) that drive the position of the first cracks. The effect of defects on the damage threshold vanishes when damage increases: for higher strains, the distance between consecutive cracks becomes more homogeneous, and no major difference can be observed between different areas of the specimen. The average crack rate computed on the whole length reveals a preliminary step with a progressive beginning of cracking. It is worth noting that the modelling of the preliminary step is fundamental for the prediction of first leak paths.

### 3.2. Morphology of the crack network

To study the shape of a damage network, damage observation under loading and cross-section examination were applied to $[0/+67.5/-67.5]_s$ specimens. The interest of this lay-up is that transverse cracking can occur in three different and consecutive plies, leading to the creation of a network. For this we have relied on two complementary experimental techniques: micrographic observation after successive abrasions and X-ray microtomography under tensile loading detailed respectively in Sections 2.3 and 2.4

The centre ply, -67.5°, is also a double ply, and thus has a lower cracking threshold. In this ply, cracks occurred first and were continuous through the width.

Conversely, cracks in the simple +67.5° plies were short and located around the cracks of the double ply. Hence, existence of cracks in an adjacent ply drives the position and the length of the new cracks. Moreover, the cracking threshold of the simple plies is also modified: cracks occur almost simultaneously in the three plies despite their different thicknesses.

Tests on $[0/+67.5/0/67.5]_s$ specimens in which cracked plies are isolated are scheduled. Comparing the results obtained with isolated and not-isolated plies will make it possible to quantify the effect of the interaction between cracks in adjacent plies.

Micrographic observations after successive abrasions are made using a mark on the edge of the specimen as well as the patterns of the areas rich in matrix and thermoplastic nodules to position the images of the different sections relative to each other and thus access a discrete but three-dimensional description of the network. However, many iterations are required to describe the network accurately, and the execution of the protocol and the exploitation of the results are tedious. Moreover, because of the destructive nature of this method, it does not allow studying the evolution of the same network of cracks for different load levels. Nevertheless, it is a precise and reliable tool that makes it possible to elaborate and validate X-ray observations, but also to obtain fine information that is not easily accessible by X-rays, such as the shape of the cracks, their opening and the morphology of the connections (see Section 3.4).

Micrographs show that transverse cracks appear simultaneously in all three plies. On the surface or through the micrographic sections, we observe that each crack in the double ply corresponds to one or more cracks in each of the single plies, which can be aligned or offset. The simultaneous and grouped occurrence of cracks in plies of different thicknesses means that the position and threshold of crack occurrence in single plies are modified and controlled by the occurrence of cracks in the adjacent double ply. There is therefore an interaction between the damage kinetics of a ply and the damage state of adjacent plies. Three-dimensional reconstructions of crack networks obtained by microtomography, such one example is shown in Figure 3, show that cracks in the central and double plies at -67.5° pass through the entire specimen, while in single plies at +67.5°, cracks are short - i.e., one length in the plane of layering in the order of the ply thickness, and connected to cracks in the central ply. These observations confirm and refine the observations made thanks to micrographic sections.

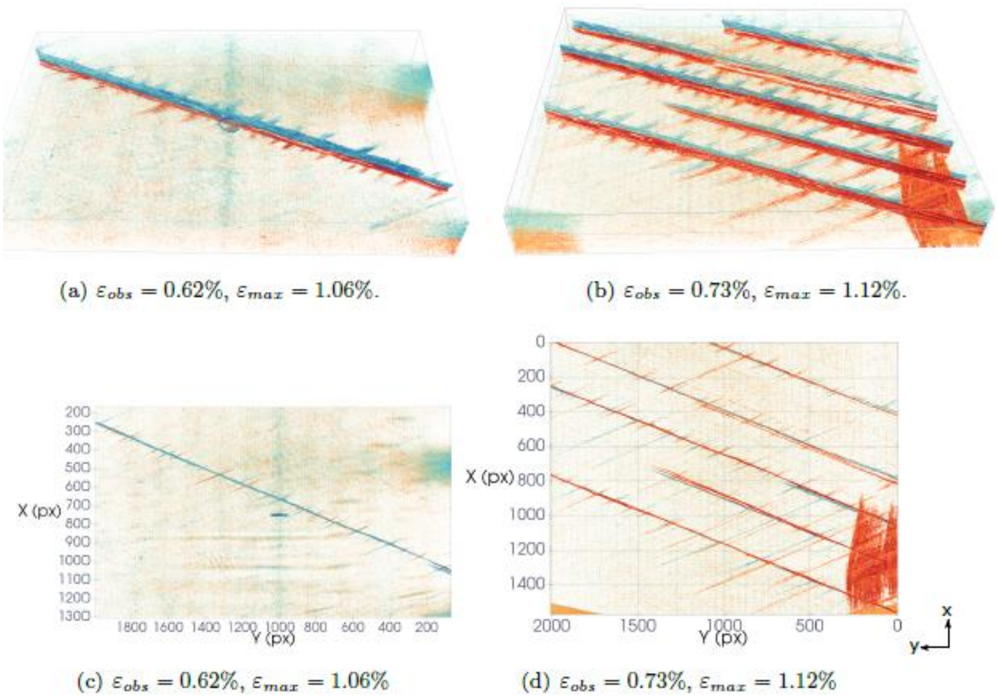


**Figure 3.** 3D reconstruction by thresholding a network of cracks for two maximum strains, two colors are assigned to the cracks to differentiate the two interfaces

To characterize the evolution of a crack network as a function of loading level, the same areas of a specimen are observed for several loading levels between which the specimen is placed in the tomographic observation device under loading. This implies being able to find the area observed using reference points which can be traces of glue, glued pencil leads, or even a gauge (the quality of the image will be lower because of the high absorption of X-rays by the metallic elements of the gauge, but observation remains possible). Figure 3 shows the networks obtained for two loads. From the rebuilt volumes, we can observe the specimen in the xy plane to count the connections by separating the interfaces between the plies. For the first loading, the cracks in the single plies are short, but for the second loading, we notice that these cracks propagate over several ply thicknesses and that they can intersect several cracks in the central ply at -67.5°. The density contrast between the cracks of the single plies and the composite is lower than for the other cracks: their opening is at the limit of visibility for this resolution.

### 3.3. Crack crossings

The cracks in two adjacent plies intersect at the interface between the plies. In order to quantify those crack crossings, tomographs of the laminate are divided into two parts. Each part contains one interface with crack crossings. In Figure 3, the interface +67.5/-67.5 is drawn in blue, and the interface -67.5/+67.5 in orange. Then, crossings in each part can be separately assessed using Figure 4. It is also possible to measure the crack length in each ply, and compute a crack density.

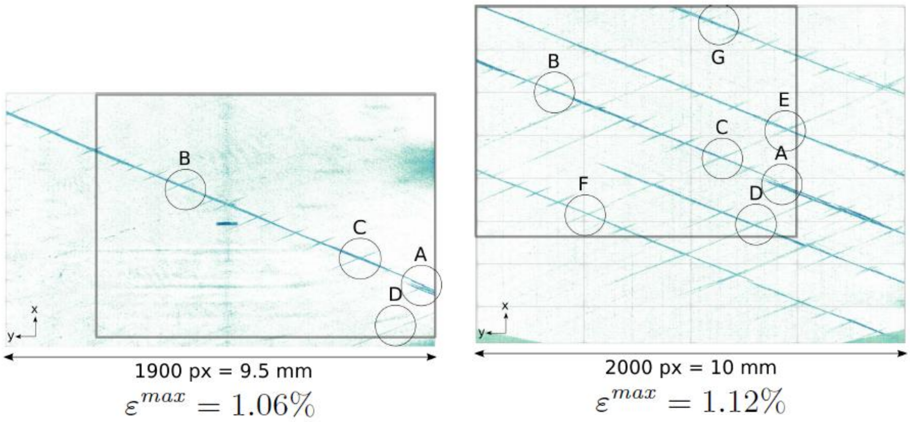


**Figure 4.** Illsutration of the evaluation of the number of crack crossings beween two plies

The evolution of the crack network has been observed for four maximal strains, in the area delimited by the gray rectangle in Figure 4. The damage scenario is drawn in Figure 5. First, a long crack appear in ply 2. Ply 2 is thicker than ply 1. Thus, in the case the plies do no interact with another (e.g. if they are isolated by 0° plies), ply 2 would have a lower cracking threshold than ply 1. However, here, at the same time ply 2 reaches its cracking threshold, cracks appear also in ply 1. Those cracks are short and start from the crack in ply 2. This shows an interaction with the damage in the adjacent plies. This interaction leads to a configuration that maximizes the number of crack crossings for a given crack density in ply 1. Damage kinetics of the two plies are also impacted by this interaction. It is worth to note that this interaction is usually not modeled for the sake of simplicity, and because short cracks have a negligible effect on the elastic behaviour. To address the issue of leak path in laminates, it will be essential to take this interaction into account.

After the first step in the damage scenario, new cracks appear in ply 2, coupled with short cracks in ply 1, while some cracks in ply 1 propagate and become longer and longer. The increase in crack crossing with respect to the crack density slows down.

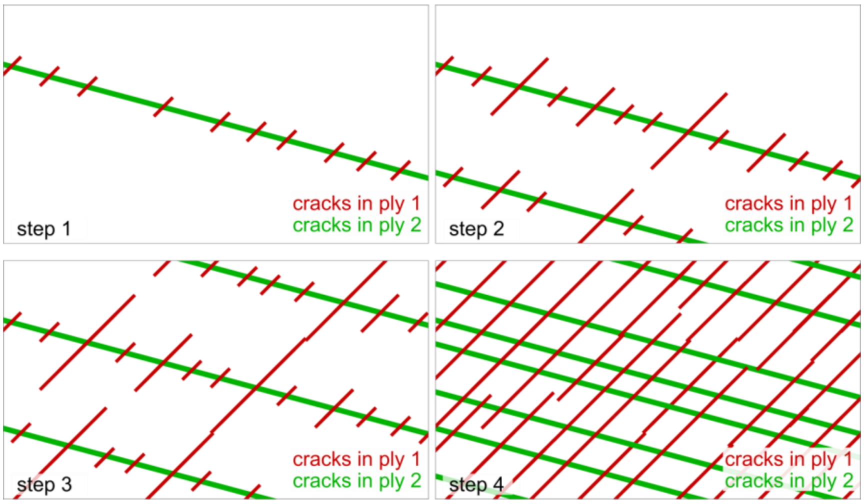


**Figure 5.** Illustration of damage scenario

### 3.4. Morphology of connection at crack crossings

Cross section examinations on the same laminates validate the previons observations, and furthermore give interesting details of the crack crossings. Figure 6 shows the intersection of the same three cracks in a [0/+67.5/-67.5$_2$/+67.5/0] laminate, for five different cross sections. The interface between +67.5° and -67.5° plies is highlighted by two yellow boxes. At the interface on the left, delamination connects the cracks. This makes the connection area larger than the Crack Opening Displacement (COD) at crack tip and may increase the leakage rate induced by a leak path. However, the morphology of the connection may be more complex and tortuous, as observed at the interface on the right side of Figure 6.

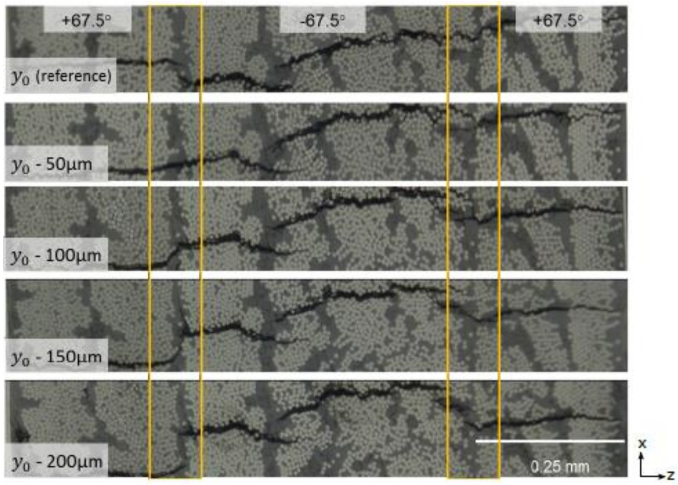


**Figure 6 :** Intersection between the cracks of three plies in a [0/+67.5/-67.5]s for five polishing depth, the yellow boxes hightlight the two intefaces

## 5. Conclusion

Transverse crack and delaminations at crack tips were identified with an optical microscope during tensile loading. A length of 100mm was observed for several loading levels. This allowed to highlight a preliminary step in the damage scenario with small crack densities and progressive growth before a second step with a steeper growth. In bulk, cross-section examinations showed that no delamination occurred at crack tip in the material of the study. Cross-section examinations were also performed for the observation of [+45/-45]$_{2s}$ and [0/+67.5/-67.5]$_s$ layups in order to bypass the issue of free edge effects. Damage state in those layups was shown to be significantly different through the width of the specimens than at the surface of the edges. Particularly, there is no transverse crack in $[+45/-45]_{2s}$ specimens subjected to shear strains up to 4%. It was also observed that crack growth and crack length are modified by the damage state of adjacent plies. These elements are fundamental for the assessment of permeability performance, and thus will be introduced in the predictive model.

Predicting the percolation of the network also requires to describe the network in terms of number of connections between two adjacent plies. Cross-section examinations give an insight into the network pattern, nevertheless this method is restrictive because it is destructive, not very accurate and time-consuming. Experiments involving X-ray tomography were therefore performed to accurately characterise the crack network pattern. This kind of experiment remains a challenge because of the mismatch between the size of the crack pattern (3mm in the case of the $[0/+67.5/-67.5]_s$ specimen) and the size of its elements (COD ~ 2µm when the specimen is unloaded). To keep the cracks open during observation, a dedicated tensile device was developed in order to perform microtomography on specimen subjected to a tensile loading. Those experiments allows to assess the effect of the interactions between damage of consecutive plies and quantify the number of crack crossings.

These complementary experimental methods supply relevant data to build a predictive meso-model. Nevertheless, the amount of information required to entirely identify a predictive model and the arduousness of experimental procedures push into completing the physical tests with numerical virtual testing.

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