

Multi-scale experimental and computational investigation of matrix cracking evolution in carbon fiber-reinforced composites in the absence and presence of voids

Mahoor Mehdikhani^{1,2*}, Nikolay Petrov¹, Larissa Gorbatikh^{1,2}, Stepan V Lomov^{1,2}

¹ KU Leuven, Department of Materials Engineering, Kasteelpark Arenberg 44, 3001, Leuven, Belgium

² SIM vzw, Technologiepark 935, B-9052 Zwijnaarde, Belgium

* mahoor.mehdikhani@kuleuven.be

Abstract. Manufacturing defects such as voids can influence matrix cracking, which is one of the main forms of damage in fiber-reinforced laminates. Characterization and investigation of matrix cracks is not straightforward, neither experimentally nor computationally. In the current study, we develop a methodology for *in-situ* analysis of matrix cracking evolution in the absence and presence of voids. The cracks are detected using digital image correlation at three different scales: macro, meso, and micro. Additionally, a combined two-scale computational approach is established for prediction of the effect of voids on matrix cracking. Local properties of the material are calculated using computational micromechanics and then serve as input for the meso-scale model based on extended finite element method. The computational approach predicts the crack density evolution in function of the applied deformation. The results of both experimental and computational studies reveal that voids initiate matrix cracks, causing earlier damage in off-axis plies. They also result in higher crack density, for a given applied strain.

1. Introduction

Voids are manufacturing defects that are inevitably formed during production of Fiber-Reinforced Composites (FRCs). Although control and reduction of voids have been widely worked on and improved, recent manufacturing techniques, such as automated tape processing and out-of-autoclave curing still face void control as one of their main challenges. Voids can affect the mechanical properties of FRCs. They primarily degrade the matrix-dominated properties such as inter-laminar shear, compressive, and transverse tensile strength. The broad research on formation, characteristics, and mechanical effects of voids in FRCs is reviewed in [1].

Voids can influence the damage development in FRCs. In particular, matrix cracking that is one of the main damage modes in off-axis plies is sensitive to the presence of voids. It has been reported [2, 3] that voids can cause initiation of the matrix cracks, but with increase of loading, fewer voids remain that have not started any cracks and cracks start forming also in regions without voids. Hence, the effect of voids on matrix cracking fades. The initiation of matrix cracks from voids is attributed to the strain concentrations caused by them detected via Digital Image Correction (DIC) at the micro-scale [4].

In order to identify the significance of the effect of voids on matrix cracking evolution, matrix cracks need to be characterized during the mechanical loading. This is not straightforward especially for carbon FRCs because of their dark color. The available approaches for characterization of matrix cracks, including (edge replication) optical microscopy, electron microscopy, X-ray radiography or computed



tomography, and ultrasonic C-scan, require stopping of the test and sometimes removing the sample before it can be characterized. Acoustic emission registration is an indirect measurement and requires interpretation of acoustic emission signals and identification of the damage type, which is challenging. Direct *in-situ* observation of the cracks has been shown feasible in transparent materials like glass FRCs. However, for carbon FRCs, this can be done, in the best case scenario, only on the specimen surfaces. It also requires manual counting of cracks that can be laborious and prone to errors, especially for unopened cracks or cracks with small opening. Therefore, a tool for detection and characterization of matrix cracks in *in-situ* captured data would be very helpful.

Due to difficulties in detection of matrix cracks as well as in controlling the parameters of voids, such as the void content, size, and distribution, a computational approach is a valuable tool for analysis of the effect of voids on matrix cracking. Though some research on prediction of the effect of voids on effective material properties of FRCs has been performed [5-7], their effect on statistically-controlled properties, such as matrix cracking has not been modeled, to the authors' knowledge. This is mainly because voids are microscopic features that interact with other heterogeneities in the material both at lower and higher scales (such microscopic fibers and cracks at the meso-scale). This issue can be resolved by a combined two-scale modeling methodology.

In the present study, we develop two methodologies to address the challenges in investigation of the effect of voids on matrix cracking in FRCs. The experimental methodology characterizes cracking in *in-situ* images captured at different scales during the test, using Digital Image Correlation (DIC). It is demonstrated on cross-ply aerospace-grade carbon/epoxy laminates and laminates with deliberately-produced voids. The computational methodology is developed in a combined two-scale framework. The degradation of local properties in the presence of voids is calculated, using computational micromechanics, and transferred to a laminate-level model that can simulate transverse cracking, using eXtended Finite Element Method (XFEM). Both methodologies allow investigation of the evolution of transverse cracking in function of applied strain, influenced by voids.

2. Materials

An aerospace-grade carbon/epoxy composite is studied. The unidirectional prepreps are made by *Cytec* from *Tenax® - E HTS40 F13 12K* carbon fibers and *CYCOM® 977-2* (toughened) epoxy resin. Cross-ply laminates are produced with automated tap laying and autoclave curing at *SABCA Limburg NV, Belgium*. The lay-ups are debulked at room temperature prior to cure. Two different cure cycles are defined: one high-pressure-temperature cycle to produce reference material and one low-pressure-temperature to produce imperfect material, including intra-laminar voids. $[90/0]_{2s}$ and $[90/0]_{4s}$ laminates with an average ply thickness of ~ 0.2 mm are produced. The fiber volume fraction is within 53-60% and the void content is around 1%.

3. Experimental and computational methods

3.1. Experimental methodology: *in-situ* characterization of cracking using DIC

In-situ characterization of transverse cracking is performed at three different length scales: macro, meso, and micro. The macro- and meso-scale images are acquired from $[90/0]_{2s}$ specimens under uniaxial tension. To this end, tensile specimens, from both reference and imperfect material, are prepared according to the standard ASTM D3039, one surface is paint sprayed as the required random speckle pattern for DIC, and one thickness is grinded and polished for edge crack monitoring. Macro-scale images are taken from the sprayed front surface of the specimen with a window size of ~ 50 mm \times 40 mm and meso-scale images from the polished thickness edge with a window size of ~ 5 mm \times 4 mm. Both sets of images are captured via *Limess* cameras with application of local LED lights.

The micro-scale analysis is similar to those performed in [8, 9]. The micro-scale images are acquired from a small $[90/0]_{4s}$ specimen loaded under 3-point bending inside a Scanning Electron Microscope (SEM). One of the thickness edges of the specimen is grinded and polished and the specimen is mounted in a Deben mini-tester stage, installed inside an SEM. A suitable study zone on the tensile edge of each reference and imperfect material is identified, from which SEM images are captured during loading. The micro-scale images have a window size of ~ 1500 μm \times 1300 μm .

The transverse cracks are detected via DIC in the macro- and meso-scale images since they can hardly be observed with the naked eye. The transverse cracks appear as lines with high strains in the DIC strain maps and peaks in the DIC strain profiles along a horizontal line. The detection of these peaks is carried out semi-automatically using MATLAB. The number of peaks in each image divided by the length of the profile gives a crack density at that deformation step, which can be plotted versus applied strain, resulting in crack density evolution graphs. The details of the experimental methodology are explained in [10].

3.2. Computational methodology: two-scale modeling of voids' effect on cracking

Micro-scale finite element models of a composite containing randomly distributed fibers and a void are built. Carbon fibers have transversely isotropic elastic properties without damage and the epoxy matrix is assigned with an elasto-plastic damage model, developed in [11]. A reference model is also created. Effective elastic properties through homogenization and transverse strength through simulation of transverse tension are obtained for the models. These properties are then used in the meso-level model.

The meso-level model simulates a carbon/epoxy laminate with the size of 20 mm × 20 mm × 0.6 mm. The laminate consists of unidirectional plies with homogenized properties in the stacking sequence of [0°/90°/0°]. XFEM is assigned to the transverse ply, as described in [12]. Damage in XFEM is controlled by a bilinear cohesive law, which uses the transverse strength of the material at the micro-scale as the damage initiation threshold and Mode I fracture toughness for the damage progression. To model the presence of voids, local regions, called “weak volumes”, are randomly distributed in the transverse ply. Different void contents can be considered. A reference model is also created. For the meso-models with voids, the transverse stiffness of the transverse ply is reduced to that obtained via micro-modeling, based on the void content. The transverse strength and the Mode I fracture toughness of the weak volumes is assigned to those computed via micro-modeling, based on the void size of interest. Other properties of the transverse ply and all the properties of the longitudinal plies are set to those obtained from the reference micro-model. Displacement-based uniaxial tension is applied to the meso-level model. Both micro- and meso-level simulations are performed using *Abaqus FEA*. The computational methodology is schematically shown in figure 1. The details of the computational methodology are explained in [13].

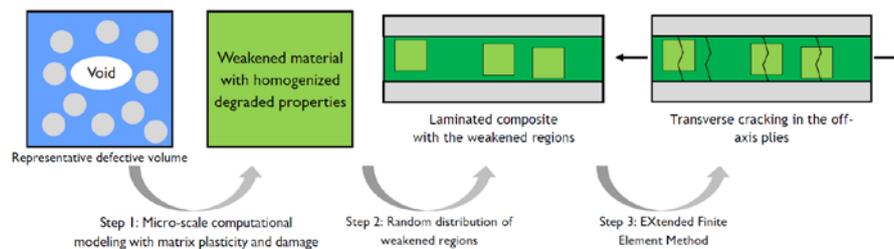


Figure 1. Schematics of the computational methodology for simulation of transverse cracking in the presence of voids using computational micromechanics and XFEM.

4. Results and discussions

4.1. Experimental results

4.1.1. Macro- and meso-scale analysis. Macro-scale horizontal strain (E_{xx}) maps are obtained via DIC on the images of the front surface. As can be observed in figure 2, the transverse cracks in the reference specimen tend to start from the edge and span, sometimes gradually, the width of the specimen. In the imperfect material, cracks initiate in early stages of loading at the specimen's edge, as visible in figure 3, but they slowly/rarely propagate toward the other edge. This may be because initiation of cracks is more favorable than their propagation due to the presence of the voids, as strain concentrators. The crack density along the mid-width line of the reference specimen is obtained via detection of peaks in the corresponding strain profiles (figure 3b) and plotted versus the applied strain in figure 3c.

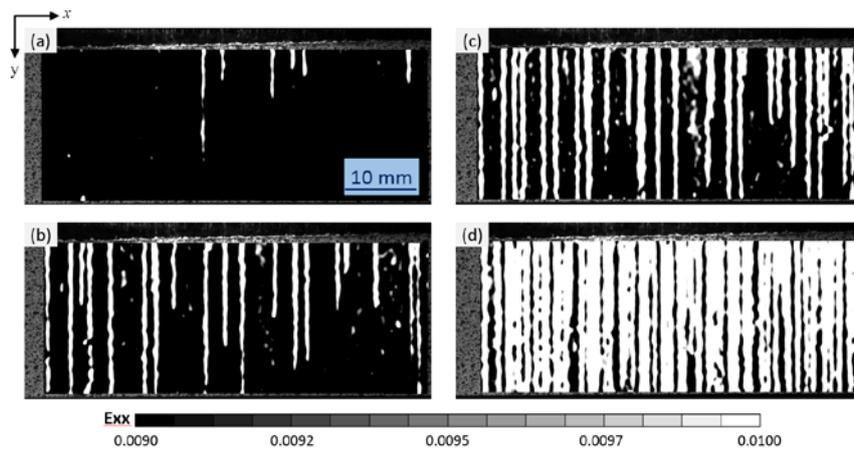


Figure 2. Cracking evolution in the outer ply of the $[90,0]_{2s}$ reference material under tension (applied in the horizontal direction), detected in the DIC horizontal strain maps at the applied strain of (a) 0.63%, (b) 0.77%, (c) 0.87%, and (d) 1.12%.

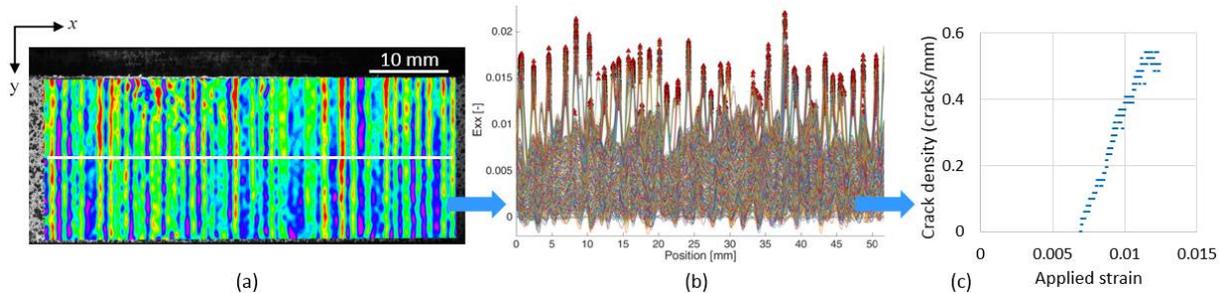


Figure 3. Macro-scale strain map at the last deformation step on the front surface of the $[90,0]_{2s}$ in the reference specimen; DIC strain profiles of all deformation steps along the mid-width line of the reference specimen; the processed crack density in function of the applied strain for the reference.

The meso-scale horizontal strain (E_{xx}) maps are obtained via DIC on the images of the thickness edge. These maps are used to extract the crack density at the edge of the specimen. This is performed for an outer ply of both materials and presented in figure 4. The figure shows that voids can cause earlier start of cracking and increase the cracking rate and saturation level in the outer ply.

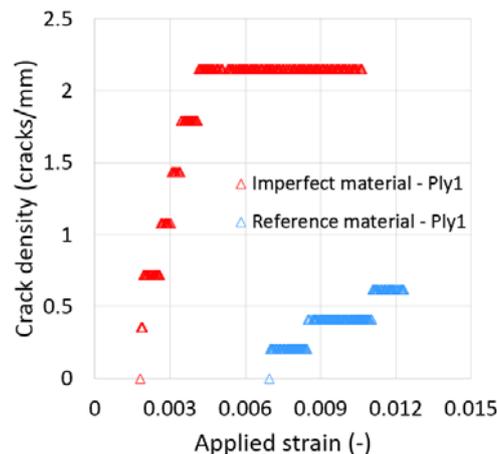


Figure 4. Crack density evolution in an outer ply (Ply1) of the reference and imperfect materials obtained via meso-scale DIC analysis.

4.1.2. Micro-scale analysis. Analysis at this scale is similar to that at the meso-scale, but allowing to see the fibers, voids, and cracks together. Therefore, a correlation between voids and crack formation can be made. For the reference specimen, no cracks are detected in the study zone on the second transverse ply from the bottom. In contrast, cracks do occur in the imperfect material. The first two cracks form from the two voids in the study zone and the second two cracks form in regions without voids. The cracks detected via DIC with their order of formation are shown in figure 5.

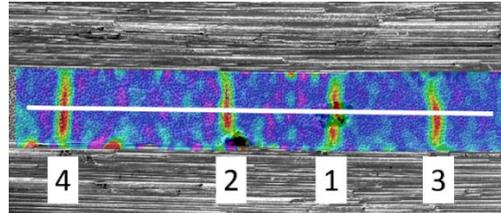


Figure 5. Transverse cracks detected via DIC at the micro-scale – the numbers show the sequence of crack formation, showing the first cracks initiating from voids.

4.2. Computational results

Micro-scale modeling reveals a reduction in transverse stiffness, transverse strength, and fracture toughness of the unidirectional ply. It also shows that the crack initiates from the void, which is due to the strain concentration caused by the void.

The total surface area of all cracks divided by the volume of the ply gives crack density for a given increment of the applied strain. This is calculated for all the meso-models and plotted versus the applied strain in figure 6. This figure shows that the initiation of cracking can be influenced by voids. This is particularly more significant for the model with the highest number of weak volumes, causing a high level of agglomeration. The predicted crack density close to the saturation level can be affected by the modeling parameters, e.g. the number of enriched regions, where cracks can occur. This region is shaded in figure 6. The detailed analysis of models with different void contents and sizes are presented in [13].

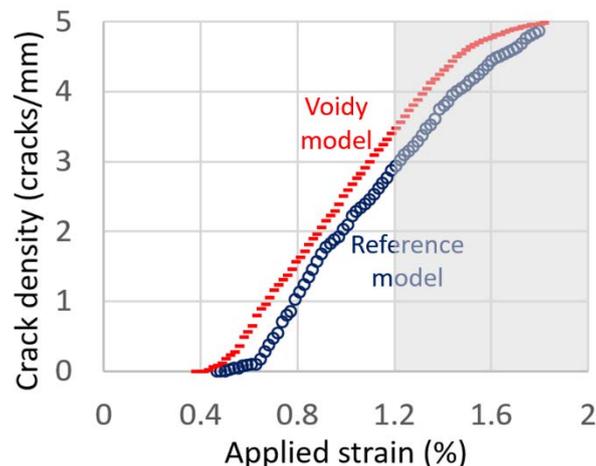


Figure 6. Predicted crack density evolution influenced by voids.

5. Conclusions

Two approaches, experimental and computational, for investigation of the effect of voids on matrix cracking are developed and examined. The experimental approach, using DIC, characterizes the cracks in the *in-situ* images acquired during mechanical tests at three scales: macro, meso, and micro. The detection and counting of cracks is performed semi-automatically using *MATLAB*. The computational approach is performed by linking micro- and meso-level finite element models. The degradation of the

properties in the presence of voids is calculated via computational micromechanics. The meso-scale model takes the predictions of the micro-level model as input and simulates crack initiation and development using XFEM. Both approaches show influence of voids on transverse cracking, especially on its initiation.

Acknowledgments

The authors gratefully acknowledge *SIM* (Strategic Initiative Materials in Flanders) and *VLAIO* (Flemish government agency for Innovation and Entrepreneurship) for their support of the *IBO project M3Strength*, which is part of the research program *MacroModelMat (M3)*, coordinated by Siemens (*Siemens PLM Software*, Belgium). The authors highly appreciate the collaboration of *SABCA Limburg* (K. Verjans and S. Wathiong) in production of the carbon fiber laminates. Stepan V. Lomov is a holder of *Toray Chair for Composite Materials* at KU Leuven, which provided partial funding of the work reported here.

References

- [1] Mehdikhani M, Gorbatikh L, Verpoest I and Lomov SV 2018 *Voids in fiber-reinforced polymer composites: a review on their formation, characteristics, and effects on mechanical performance*. *J. Compos. Mater.* In print
- [2] Huang Y, Varna J and Talreja R 2014 *Statistical methodology for assessing manufacturing quality related to transverse cracking in cross ply laminates*. *Compos. Sci. Technol.* **95** 100
- [3] Sisodia S, Gamstedt EK, Edgren F and Varna J 2014 *Effects of voids on quasi-static and tension fatigue behaviour of carbon-fibre composite laminates*. *J. Compos. Mater.* **49** 2137
- [4] Aratama S, Hashizume R, Takenaka K, Koga K, Tsumura Y, Miyake T, Nishikawa M and Hojo M 2016 *Microscopic observation of voids and transverse crack initiation in CFRP laminates*. *Adv. Compos. Mater.* **25** 115
- [5] Ashouri Vajari D, González C, Llorca J and Legartha BN 2014 *A numerical study of the influence of microvoids in the transverse mechanical response of unidirectional composites*. *Compos. Sci. Technol.* **97** 46
- [6] Carraro PA, Maragoni L and Quaresimin 2015 *M Influence of manufacturing induced defects on damage initiation and propagation in carbon/epoxy NCF laminates*. *Adv. Manuf. Polym. Compos. Sci.* **1** 44
- [7] Liebig WV, Viets C, Schulte K and Fiedler B 2015 *Influence of voids on the compressive failure behaviour of fibre-reinforced composites*. *Compos. Sci. Technol.* **117** 225
- [8] Mehdikhani M, Aravand M, Sabuncuoglu B, Callens MG, Lomov SV and Gorbatikh L 2016 *Full-field strain measurements at the micro-scale in fiber-reinforced composites using digital image correlation*. *Compos. Struct.* **140** 192
- [9] Mehdikhani M, Matveeva A, Aravand MA, Wardle BL, Lomov SV and Gorbatikh L 2016 *Strain mapping at the micro-scale in hierarchical polymer composites with aligned carbon nanotube grafted fibers*. *Compos. Sci. Technol.* **137** 24
- [10] Mehdikhani M, Steensels E, Standaert A, Vallons K, Gorbatikh L and Lomov SV 2018 *Multi-scale digital image correlation for detection and quantification of matrix cracks in carbon fiber composite laminates in the absence and presence of voids controlled by the cure cycle*. *Compos. Part B-Eng.* under review
- [11] Melro AR, Camanho PP, Andrade Pires FM and Pinho ST 2013 *Micromechanical analysis of polymer composites reinforced by unidirectional fibres: Part I – Constitutive modelling*. *Int. J. Solids. Struct.* **50** 1897
- [12] Petrov NA, Gorbatikh L and Lomov SV 2018 *A parametric study assessing performance of eXtended Finite Element Method in application to the cracking process in cross-ply composite laminates*. *Compos. Struct.* **187** 489
- [13] Mehdikhani M, Petrov N, Melro AR, Lomov SV and Gorbatikh L 2018 *The effect of voids on matrix cracking in composite laminates revealed through combined modeling at the micro and meso-scales*. In preparation.