Manuscript Number: JCOMA-19-63R3

Title: Detailed characterization of voids in multidirectional carbon fiber/epoxy composite laminates using X-ray micro-computed tomography

Article Type: Research Paper

Keywords: Voids; B. Porosity; X-ray computed tomography; A. Polymer-matrix composites (PMCs)

Corresponding Author: Dr. Mahoor Mehdikhani, PhD

Corresponding Author's Institution: KU Leuven

First Author: Mahoor Mehdikhani, PhD

Order of Authors: Mahoor Mehdikhani, PhD; Ilya Straumit, PhD; Larissa Gorbatiikh, PhD; Stepan V Lomov, PhD

Abstract: We employ X-ray micro-computed tomography to generate an extensive dataset of voids' characteristics in carbon/epoxy laminates with different stacking sequences. The voids are deliberately induced by modifying the cure cycle. The three-dimensional images with voids are segmented and quantified using an in-house software package, VoxTex. The characteristics of voids, i.e. shape, size parameters, orientation, and spatial location distribution, are obtained. The majority of voids have slightly flattened cross-sections. The voids prefer to localize in the inner plies of the laminate, rather than in the outer ones. Near the laminate mid-thickness, they become more elongated. The stacking sequence affects the size and shape of voids. The planar location of voids is not random and tends to create a pattern, corresponding to the the ply orientations in the laminate. The data is reported in detail, ready to use, for example, as input for modeling studies of similar composites with voids.

Response to Reviewers: The response to the reviewer's comments is provided in a separate MS Word file, which is uploaded along with the submission.

Research Data Related to this Submission

Title: A dataset of voids' characteristics in multidirectional carbon fiber/epoxy composite laminates, obtained using X-ray micro-computed tomography

Repository: Mendeley Data

<https://data.mendeley.com/datasets/j6rkbhd64d/draft?a=3185ccec-0424-469f-9f7a-405ael44a738>

Dear reviewer,

Thanks for your effort in improving the quality of the article!

We are glad that the corrections are OK in your view. Based on your suggestion, the current resubmission includes an extensive revision of the manuscripts by a native English speaker. The corrections are highlighted in **blue** throughout the text. Regarding the **usefulness and applicability** of the results, the following part is added to the Concluding remarks (with a an extension at the end). We hope that the modifications address your suggestions.

“Whilst the voids in this study are intentionally induced and excessive, the naturally occurring voids in similar materials should have analogous shape and orientation distribution since these characteristics are controlled by the microstructure. For quantitative use of the results, one needs to account for the void content and size correction factors for their own material system. The current dataset will be in any case useful as a real-world example for virtual porosity generators.”

Kind regards,

Mahoor Mehdikhani, PhD

Composite Materials Group

Materials Engineering Department, KU Leuven

mahoor.mehdikhani@kuleuven.be

T +32 16 37 69 25

Detailed characterization of voids in multidirectional carbon fiber/epoxy composite laminates using
X-ray micro-computed tomography

Mahoor Mehdikhani ^{a,b,*}, Ilya Straumit ^a, Larissa Gorbatikh ^{a,b}, Stepan V. Lomov ^{a,b}

^a *KU Leuven, Department of Materials Engineering, Kasteelpark Arenberg 44, 3001 Leuven, Belgium*

^b *SIM M3 program, Technologiepark 935, B-9052 Zwijnaarde, Belgium*

* Corresponding author E-mail: mahoor.mehdikhani@kuleuven.be

Abstract

We employed X-ray micro-computed tomography to generate an extensive dataset of void characteristics in carbon/epoxy laminates with different stacking sequences. The voids were deliberately induced by modifying the material's cure cycle. The obtained three-dimensional images demonstrating voids were segmented and quantified using an in-house software package, *VoxTex*. The void characteristics, i.e. shape, size parameters, orientation, and spatial distribution, were obtained for analysis. It was found that voids are typically elongated features, aligned with the neighboring fibers. The majority of voids had slightly flattened cross-sections. The voids preferred to localize in the inner plies of the laminate, rather than in the outer ones. Near the laminate mid-thickness, they became more elongated. The stacking sequence was found to affect the size and shape of voids, with the void content being higher for a thicker laminate. The planar location of voids is not random and tends to create a pattern, corresponding to the ply orientations in the laminate. The data gathered during this study has been reported in detail and can be used, for example, as input for studies modeling similar composites with voids. The entire micro-CT data is presented as a *Data in Brief* article in [1].

Keywords: Voids, Porosity, X-ray computed tomography, Polymer-matrix composites (PMCs)

1. Introduction

Voids are one of the main types of manufacturing defects in Fiber-Reinforced Composites (FRCs), forming in almost every processing technique. They can cause degradation of the mechanical properties (especially those controlled by the matrix) and can influence the hygrothermal performance [2]. If their effect on mechanical properties can be precisely quantified, void allowance limits could be introduced for different applications and loading scenarios of the part in question. Defining reliable allowance limits would help to reduce the amount of manufacturing waste generated, as well as the need for certain repair, thus leading to cheaper and more sustainable FRCs production. Evaluating the effect voids have on composite performance necessitates the accurate analysis of their identifying characteristics, i.e. shape, size, orientation, and spatial distribution. Once these characteristics are known, their effect on mechanical and hygrothermal properties

can be experimentally investigated. This would enable the link between void characteristics and composite performance to be established.

Quantifying the effect voids have on mechanical performance is also possible through simulations. For an accurate prediction, this approach needs detailed information on void characteristics. Due to the lack of relevant data on voids in the literature, simplistic assumptions are often made for their characteristics, e.g. in [3-7], while some of the assumptions are not always justifiable. Therefore, characterization of voids is an essential step for understanding and forecasting their effects on mechanical and hygrothermal properties. The ability to accurately characterize voids is also of great importance to composite part quality control processes, especially when deciding whether a part should be accepted, following the establishment of void allowance limits.

Techniques used for characterization of voids have evolved over time, starting from simple methods such as density determination (Archimedes) and progressing to more advanced techniques. Details of these methods and their advantages and disadvantages can be found in [2, 8-11]. Currently, the most advanced technique is X-ray micro-Computed Tomography (micro-CT), which obtains a stack of X-ray images from different angles of an object to create a three-dimensional (3D) image of it. The accuracy of this technique depends on the image processing performed to segment and detect the voids. Micro-CT is non-destructive and provides a full 3D analysis of voids, but it is expensive. Moreover, in micro-CT systems that only rely on geometric magnification (and not exerting any optical magnification), the resolution of the results depends inversely on the sample size. This means that for high-resolution results (several micrometers/pixel), one would need very small samples (several millimeters in the maximum dimension).

Previous research has been conducted on the application of micro-CT for investigating void formation [12, 13] and analyzing the effect voids have on the thermo-mechanical behavior [14-16]. Furthermore, some studies employ micro-CT for characterization of voids. In one of the first studies, Schell et al. [17, 18] detected small spherical voids in the resin-rich regions and large voids with irregular shapes between tows in a woven glass/epoxy composite. Later, Hernández et al. [19, 20] conducted a comprehensive analysis of voids in unidirectional (UD) and quasi-isotropic carbon/epoxy laminates. The authors reported a large amount of information on the shape, size, orientation, and spatial orientation of mainly inter-laminar voids. Little et al. [8] compared different characterization techniques for voids in laminates made of UD carbon/epoxy plies. Micro-CT was found to be the most accurate technique. Using a woven carbon/epoxy composite, Nikishkov et al. [21] demonstrated the superiority of micro-CT over microscopy with regard to the section-bias error.

Their work revealed dependency of the results on the scan resolution and on the material-void gray value threshold. Using sub-pixel contouring for the average gray values of the air and material, the authors developed a high-accuracy void detection methodology.

Madra et al. [22] performed a micro-CT analysis of voids in a woven glass/thermoplastic composite at three scales: the composite laminate, the textile layer, and the single yarn. To this end, the authors developed a set of image processing algorithms, including a learning algorithm for segmentation of each image into matrix, fiber, and void as well as a filtering process to remove the noise arising from thresholding. The void content measured in the laminate analysis was higher than that which resulted from using the density determination (Archimedes) technique. This may be because the latter excludes the voids connected to the specimen's outer surface. The difference between void content measured via micro-CT and that measured via other techniques was also reported in [23-26].

Using synchrotron radiation, Sisodia et al. [27] conducted a multi-scale analysis of voids within an epoxy matrix composite manufactured from quasi-UD non-crimp fabric, consisting of both carbon and glass fibers. Straumit et al. [28] used the k -means clustering algorithm to segment the voxel model of a 3D woven carbon/epoxy composite into yarns, matrix, and voids in the *VoxTex* software, based on both gray value density and image anisotropy. A detailed analysis of void content, size distribution, and location in tubular braided Kevlar/epoxy composites was conducted by Melenka et al. [29].

In addition to enhancement of the micro-CT technique and the relevant image processing methods, a few studies have reported detailed quantitative information about voids' characteristics, obtained with micro-CT. Probably the most detailed data was provided by Hernández et al. [19, 20]. They noticed that in their material most of the voids were inter-laminar with a rod-like shape, resulting from air entrapment between plies. Voids were found to be elongated predominantly in the fiber direction of their (adjacent) ply, with void length increasing with size. This was also confirmed by Tserpes et al. [30]. Quantitative results are also reported in other studies including [27, 29, 31], which are summarized in Section 4.1. According to Hernández et al. [19, 20], along the laminate's width, voids were almost periodically distributed, which was attributed to the cell structure created by fiber-rich zones. Through the thickness, their distribution was irregular in the quasi-isotropic laminates, while in the UD laminates, there were more voids in the inner plies.

Despite the existence of several research studies, focusing on different aspects of using micro-CT to analyze voids in composites, a complete dataset of void characteristics in carbon/epoxy laminates is still lacking. The creation of such a dataset is of great importance, especially for modeling studies investigating the

effect voids have on the mechanical behavior of carbon FRCs . In fact, the idea of the current study was born when the authors were conducting a simulation study on the effect of voids on ply cracking evolution [32]. In order to build realistic models, the authors required a dataset containing the statistical distribution of void characteristics. Due to the lack of such data in the open literature, they had to experimentally obtain the statistics through micro-CT investigation.

In the present study, we perform a full 3D analysis of voids in carbon/epoxy laminates with different stacking sequences using micro-CT. The voids are intentionally induced in the laminates during production by changing the processing parameters. Micro-CT results are processed with *VoxTex*, an in-house software package, used to obtain details of void shape, size, orientation, and spatial distribution. Although the voids have been intentionally induced, naturally occurring voids are expected to have similar characteristics, at least qualitatively. This is because the shape and orientation of voids, which for this type of material are typically the unimpregnated volumes between the fibers, are controlled by the microstructure of the composite. Carbon/epoxy is selected as a study material since it is widely used in industries where voids are traditionally unwelcome such as aeronautics. The results however are qualitatively generic for UD-ply laminates made of other constituents (e.g. glass fibers and epoxy).

This paper describes and analyzes the data trends and presents the data in graphical form. The full dataset and the original image stacks are presented in a *Data in Brief* article [1] and available in the *Mendeley Data* repository.

2. Composite laminates

The material studied is an aerospace-grade carbon/epoxy composite made from UD prepreg tapes, but with intentionally induced voids. The prepreg is a product of *Cytec* and includes high-strength standard-modulus carbon fibers, *Tenax® - E HTS40 F13 12K*, impregnated with a toughened epoxy resin, *CYCOM® 977-2*. The lay-up was prepared with automated tape laying and cured in an autoclave, both at *SABCA Limburg NV, Belgium*. Prior to cure, debulking was applied to the lay-ups once or several times, depending on the laminate's thickness. More specifically, a release film was placed over the laminate and a vacuum film was applied on top. The laminate system underwent a vacuum level of 0.5 to 0.7 bar for a minimum of 10 minutes at room temperature. Afterwards, the vacuum and release films were detached, and the final bagging started. In order to produce laminates with voids, the manufacturer's cure cycle (i.e. curing at 180 °C and 6 bar for 3 hours, followed by post-curing) was not used. Instead, a low-pressure-temperature cure cycle, similar to [33],

was applied. This cycle included curing at 150 °C and 0.5 bar for 3 hours, before post-curing. The vacuum was applied during the cure.

Five lay-ups were produced: $[\pm 45]_{2s}$, $[\pm 67.5]_{2s}$, $[67.5/22.5]_{2s}$, $[0/90]_{2s}$, and $[0/90]_{4s}$. These lay-ups were chosen based on the material description of a ply degradation meso-model, described in [34], to investigate the effect of defects on the model damage parameters in a separate study. The atypical lay-ups of some of these laminates make them interesting particularly for exploring the effect of stacking sequences on void characteristics. The laminates were produced in 200 mm $\times$ 200 mm dimensions, with the cured ply thickness being ~ 0.17 mm. One micro-CT specimen was cut from the inner area of each produced plate (not from the edges). The nominal specimen dimensions were ~ 15 mm $\times$ 10 mm $\times$ 1.4 mm for the 8-ply lay-ups and ~ 15 mm $\times$ 10 mm $\times$ 2.8 mm for the 16-ply lay-up.

Damage development with regard to the defects in this material was studied in [32, 35], where the effect of voids on matrix cracking evolution in cross-ply laminates was experimentally and numerically investigated. It was shown that the presence of voids leads to the early initiation of matrix cracking in the transverse plies.

3. Micro-CT image acquisition and processing

The micro-CT was performed using the HECTOR system [36] from Ghent University Centre for X-ray Tomography (UGCT). The system uses an XWT 240-SE micro-focus source from X-RAY WorX and a 40 $\times$ 40 cm² PerkinElmer 1620 CN3 CS flat-panel detector. The specimens were mounted and centered on the scanner rotation stage. The scanning volume was 12 $\times$ 12 $\times$ 12 mm³, and the scan resolution was 6.56 μ m/pixel. The scan time for each specimen was about 45 minutes. The rotation step was 0.15° with 1 s exposure time for each projection. The scan voltage and power were 80 kV and 10 W, respectively. The reconstruction of the projection data to tomographic slice images was performed with the Octopus reconstruction software [37].

The segmentation and data processing were carried out using the VoxTex software [28], developed for post-processing of micro-CT data in the Department of Materials Engineering (MTM), KU Leuven. The segmentation of each image into domains of “void” and “material” is performed by classifying each pixel of the image into two sets: void or non-void. In VoxTex, this classification is based on gray value and can be performed either manually with a predefined gray value threshold or automatically with definition of a “minimum void classification confidence”. The automatic classification is carried out in two steps. First, a statistical model is constructed, describing the distribution of gray value in the image. Then, using the statistical model, the probability of each pixel of the image belonging to the void domain is calculated. The

statistical model of the gray value distribution, $P(g)$, is described using the two-component Gaussian mixture model:

$$P(g) = \alpha_v N(g|\mu_v, \sigma_v) + \alpha_m N(g|\mu_m, \sigma_m),$$

where α is the component weight parameter, μ and σ are estimates of the mean and standard deviation respectively, subscripts v and m denote “void” and “material” respectively, and $N(x|\mu, \sigma)$ is the Gaussian probability density function:

$$N(x|\mu, \sigma) = \frac{1}{\sqrt{2\pi}\sigma} e^{-\frac{(x-\mu)^2}{2\sigma^2}}.$$

This model is valid for such an analysis because the noise in a micro-CT image is distributed normally, although in some cases the actual gray value histogram may deviate from the two-component Gaussian shape, for example, **when** in the presence of severe uncorrected artefacts, or when a histogram is **truncated** such that **parts** of the gray value range is lost.

Fitting of the model to the histogram is performed using the Expectation-Maximization (EM) algorithm. In order to reduce the computational costs to a manageable level, the algorithm is run on a random sample of 5×10^4 gray values that correspond to 5×10^4 randomly selected pixels in the image. The EM algorithm is an iterative algorithm with a fast convergence. To ensure accuracy of resulting values for the model parameters, the number of iterations is set to 100. The output of the algorithm is the optimal set of the model parameters, i.e. $\{\alpha_v, \alpha_m, \mu_v, \mu_m, \sigma_v, \sigma_m\}$.

Classification of a pixel into the two classes (step 2) is performed by calculating the probability for the pixel to belong to a void domain, i.e.

$$P_{void}(g) = \frac{\alpha_v N(g|\mu_v, \sigma_v)}{P(g)}.$$

If the probability P_{void} is high enough ($P_{void} >$ the predefined minimum void classification confidence), then the pixel is classified as a void.

For the current study, automatic segmentation with a minimum confidence of 95% **was** carried out. For each image stack corresponding to each specimen, a region of interest **was** selected such that it **covered** the whole processable volume of the stack. This **caused** small boundary regions to be excluded from the analysis and led to slightly different sizes for the regions of interest in different specimens. A threshold of two cubic voxels for the minimum void volume **was** defined, meaning that only voids equal to or larger than two cubic voxels, i.e. $2 \times 6.55^3 = 562 \mu\text{m}^3$, **were considered**. The two-voxel threshold is the lowest allowed value in the

software. With this threshold, micro-voids comparable in size with carbon fibers and smaller, will not be included in the analysis. Such voids were rarely observed in microscopy analysis of the material.

After segmentation, voids can be fitted in *VoxTex* to equivalent ellipsoids, based on finding an ellipsoid with the same inertia tensor¹ as that of the void. For each ellipsoid, the minor, medium, and major axes were calculated, along with the location of its centroid and orientation. The orientation of the voids is defined as the angle between their major axis and the scan orientation. The frequency distribution of each of these parameters can be obtained, providing detailed statistics of voids' characteristics. The statistical analysis was performed using *MATLAB*. For obtaining 3D representations of the segmented voids, *ParaView* software [38] was used.

4. Results and discussion

The measured void content in $[\pm 45]_{2s}$, $[\pm 67.5]_{2s}$, $[67.5/22.5]_{2s}$, $[0/90]_{2s}$, and $[0/90]_{4s}$ specimens is 0.22%, 0.40%, 0.78%, 0.26%, and 1.24% respectively. Although the void content values are quite small, the number of voids in each specimen is large enough to create reliable statistics. The 3D visualization of the segmented voids in the scanned specimens is shown in Fig. 1. Qualitatively, voids (in blue) appear mainly as elongated features, oriented in the ply directions of each laminate. This is because for this type of material, voids are usually unimpregnated volumes between fibers. The voidage in the $[0/90]_{4s}$ specimen (containing 16 plies) seems to be higher than that in the other specimens (with 8 plies). It is worth noting that micro-CT analysis of a similar material, but produced with the manufacturer's recommended cure cycle, does not show any voids, as investigated in [39].

Tomographic slices, showing the cross-section of the voids, can be seen in Fig. 2, with zoomed-in views on the right side. Voids appear as dark features with irregular cross-sections. The distribution of voids through the thickness was found to be non-uniform, with more voids existing in the inner plies than in the outer ones. Since the voids are elongated features, the size of their cross-section in the 2D images depends on the section orientation. Mainly due to the difference in orientation, voids appear with different cross-sections in different plies, as shown in Fig. 2. Generally, the cross-sectional slices do not reveal much information about void morphology and size, at least without correcting for the ply orientations. That is why single-view microscopy is not a good approach for void characterization. A detailed 3D analysis of void shape and

¹ A tensor consisting of nine components I_{ij} , where on the main diagonal $i = j$, and elsewhere $i \neq j$ ($i, j: x, y, z$). I_{ii} is the moment of inertia about the i -axis, and I_{ij} is the ij product of inertia.

distribution is provided in the following sections. The full dataset as well as the original image stacks is uploaded in [1].

4.1. Void size and shape analysis

4.1.1. Size analysis

Each void is represented as an ellipsoid, for which three size parameters are defined: semi-major (a), semi-medium (b), and semi-minor (c) axis. These parameters are fitted based on the inertia tensor as explained above, and they are schematically shown in Fig. 1 (bottom right). As a rough estimate for the goodness of fit, the ratio of the fitting ellipsoid's volume and the actual void's volume is calculated for each void. The average of this ratio in different specimens is within 1.4 – 1.6 and the corresponding standard deviation is within 0.5 – 2.0. For the majority of voids (more than 97%), the fitting ellipsoid's volume is larger than the actual void's volume. Note that the void contents, mentioned previously, were calculated based on the image segmentation prior to ellipsoid fitting. They still include a discrete segmentation error, which is presumed to be negligible.

“Voids” detected with a semi-minor axis below 4.15 μm (minor axis of 8.3 μm) were deemed to be false. This is concluded because they created non-physical clusters in the relative frequency distribution of semi-minor axis and orientation of voids, as shown in Fig. 3 for the $[\pm 45]_{2s}$ specimen, which is also representative for the other specimens. Another reason to support that these features were falsely detected is that their calculated orientations are exact integers, such as 0 or 90, which is not the case for the real voids. Thus, these features are considered to be noise created during the micro-CT acquisition, reconstruction, or processing and have therefore been filtered out prior to conducting further data analysis. After filtering the false features, the relative frequency distribution of the new data was recalculated and plotted (e.g. shown in the right-hand graphs in Fig. 3 for the $[\pm 45]_{2s}$ specimen). The asymmetry seen in the filtered data of the major axis orientation (Fig. 3) is physical and explained in Section 4.2. Detected features with semi-medium axis above 900 μm have also been filtered out since they are too large to be voids. Most likely they are delaminations or flat cracks, introduced during the preparation of small specimens for micro-CT. Such large cracks were not observed on the cross-sections of larger specimens under optical microscopy.

The values of size parameters for the voids in all the studied specimens, i.e. $[\pm 45]_{2s}$, $[\pm 67.5]_{2s}$, $[67.5/22.5]_{2s}$, $[0/90]_{2s}$, and $[0/90]_{4s}$, are represented in Fig. 4. Several of the larger values have been excluded to enhance the visualization of the majority of the data, although they were not omitted from the conducted further analysis.

Although the size parameters fall in a wide range, narrower ranges can be defined to present the size parameters of the majority of voids. The average 50th percentile of the semi-minor, semi-medium, and semi-major axis among different specimens is 15.4, 46.1, and 929 μm , respectively. Therefore, half of the voids have their corresponding size parameters below these values. The average semi-minor, semi-medium, and semi-major axis among different specimens is 8.8, 20.3, and 221.6 μm , respectively. This shows that voids are elongated features. Moreover, they tend to have elliptic, rather than circular, cross-sections. These findings agree with those reported in [4, 11, 49, 51, 81, 95, 97, 139]. The coefficient of variation (defined as mean divided by standard deviation) was used to measure the scatter in the data and was found to be rather large for the size parameters.

The average void volume among different specimens is $3.8 \times 10^5 \mu\text{m}^3$. The average 50th and 95th percentiles are 0.3×10^5 and $15.1 \times 10^5 \mu\text{m}^3$, respectively. The detailed statistics of the size parameters and volumes for the voids in each specimen are reported in Table 1. The grand average among the different specimens was calculated including all voids from all the specimens. The averages of minimum, maximum, and percentiles were calculated by averaging their values over all the specimens.

In order to check whether the size parameters are interdependent, the correlation coefficient² between pairs of them was calculated and presented in Table 2. A coefficient of ± 1 means perfect correlation and a coefficient of 0 means no linear relationships. The resulting values do not differ significantly for different specimens. The highest correlation, with an average coefficient of 0.75, was found between the semi-major and semi-minor axis, meaning that thicker voids are usually longer. It is noted that the semi-medium axis has a moderate linear relationship with the other two semi-axes (average correlation coefficient = 0.5).

For both size parameters and volume in the $[\pm 67.5]_{2s}$ and $[0/90]_{4s}$ specimens, the mean value and the 50th and 95th percentiles were larger than those found for the other specimens. For the $[\pm 67.5]_{2s}$ specimen, the reason may lie in the smaller difference between the nominal ply orientations, which is equal to 45°, compared to that in the other specimens, where it equals to 90°. The smaller the difference between the nominal ply orientations and 0°, the less significance ply interfaces will have on restricting void growth, enabling the voids to become larger. Considering the through-thickness analysis of void location (Section 4.3), it seems that the interface between two differently orientated plies will cause void growth to be suppressed close to their interface. As shown in Section 4.3, the voids are larger and more frequent when

² The Pearson correlation coefficient, a measure of the linear dependence between two random variables, A and B , is defined as $\rho(A, B) = \frac{1}{N-1} \sum_{i=1}^N \left(\frac{A_i - \mu_A}{\sigma_A} \right) \left(\frac{B_i - \mu_B}{\sigma_B} \right)$, where μ and σ are the mean and standard deviation, respectively, and denoted the number of scalar observations (MATLAB R2018b Documentation).

there are two adjacent plies with the same nominal orientation. It is also noted that the concentration of voids inside the plies occurs less in the $[\pm 67.5]_{2s}$ specimen compared to the other specimens. For the $[0/90]_{4s}$ specimen, the larger voids can be attributed to the increased thickness of the laminate, leading to a higher number of inner plies and thus to more voids (discussed further in Section 4.3). Therefore, the stacking sequence, i.e. the number and orientation of plies, seems to influence the size parameters of voids.

The size of the void cross-sections was quantified with the geometric mean of their transversal semi-axes. Geometric mean is chosen (over arithmetic mean) because a circle with a radius equal to the geometric mean of the semi-axes of an ellipse has the same area of that ellipse. Therefore, the geometric mean of the transversal semi-axes represents the ellipsoid's cross-section area. The former is calculated for each void and plotted in Fig. 5 for all the studied specimens (several of the larger values have been excluded to enhance the visualization of the majority of the data). Its average among different specimens is 12.5 μm . The average 50th and 95th percentiles are 9.7 and 25.7 μm , respectively. The statistical data corresponding to the geometric mean in all the specimens is reported in Table 1. Similarly to the size parameters and volume, the mean and the 50th and 95th percentiles of the transversal semi-axes geometric mean in the $[\pm 67.5]_{2s}$ and $[0/90]_{4s}$ specimens were found to be larger than those in the other specimens. This can be explained by the same reasoning given previously.

4.1.2. Shape analysis

In order to investigate the shape of voids, two shape parameters are defined: “cross-section roundness” and “elongation” factors. The former is the ratio of the minor and medium axes and the latter is the ratio of the major axis and the geometric mean of the transversal axes. These two parameters are calculated for each void and shown in Fig. 5 for all the specimens. Several of the larger values have been excluded to enhance the visualization of the majority of the data.

The average elongation factor among all the specimens is 13.5. This means that voids are on average 13.5 times larger in their major direction than in their radial direction, confirming that they are elongated. The average 50th and 95th percentiles of the elongation factor are 8.2 and 45.2, respectively.

The average cross-section roundness factor among all the specimens is 0.61. A roundness factor of 0 means a totally flat cross-section, whilst 1 is equal to a fully circular cross-section. There is not much difference between the mean roundness factors of voids in different specimens, all being around 0.6. For visualization, an elliptic cross-section with a roundness factor of 0.6 is illustrated in Fig. 1. The average 50th and 95th percentiles of the cross-section roundness factor are 0.64 and 0.89, respectively. This means that half

of the voids have a roundness factor above 0.64. The elliptic (flattened) cross-section of voids agrees with the results in [4, 11, 49, 51, 81, 95, 97, 139]. The statistical data relating to the two shape parameters for all the specimens is presented in Table 1. The skewness results in this table show that the parameters of size and shape as well as volume are highly skewed (positively), except for the cross-section roundness factor, which is moderately (negatively) skewed.

It can be noted, in the 3D view of voids (Fig. 1), that larger voids are normally more elongated, which is also reflected in a high correlation between the minor and major axes noted above. This can be investigated by plotting the elongation factor of the fitting ellipsoids versus the actual volume of the corresponding voids (illustrated in Fig. 6). Despite the scatter in the graph, a moderate positive correlation between the volume and elongation of voids seems to exist. The correlation coefficient is used as a rough measure to quantify the linear correlation between these two parameters. Its value in different specimens is presented in Table 2. The average value of 0.40 shows a moderate positive linear correlation. However, no correlation could be found between the cross-section roundness factor of voids and their volume.

4.1.3. Comparison with the literature

There has been some micro-CT quantification of voids, with less statistical analysis, reported in the literature. The quantitative results are summarized below, with a comparison to the results of the current study, shown in parenthesis immediately after each reported result from the literature. Hernández et al. [19, 20] fitted each void in UD and quasi-isotropic carbon/epoxy laminates into an elliptic cylinder with an equivalent volume, centroid, and moments of inertia. Semi-minor and semi-major axes of the cylinders were plotted against their volume. The graphs show that the semi-major axis was below 800 μm (6000 μm), and the semi-minor axis was mainly between 8 and 30 μm (4 and 60 μm). The “elongation factor” was within 3 – 40 (1.2 – 150), and the “flatness ratio”, which is the ratio between the medium and minor axes, was within 1 – 4 (1 – 50). One of the reasons for the large difference in the results is that the values corresponding to [19, 20] are obtained from graphs that might have truncated ranges. In general, comparing only the ranges does not provide valuable information. However, it is the only possible way here since more statistical data from [19, 20] is not available.

Using synchrotron micro-CT, Scott et al. [31] measured two average diameters of 12.3 and 9.9 μm (20.3 and 8.8 μm) in the two transverse directions and an average length of 84.7 μm (221.6 μm) in the fiber direction for intra-laminar voids in a filament wound carbon/epoxy composite. Similarly using synchrotron micro-CT, Sisodia et al. [27] found that in a quasi-UD non-crimp fabric composite of carbon and glass fibers

with epoxy matrix, the volume of most of the voids is within $10 - 1000 \mu\text{m}^3$, while the full range is around $0.2 - 3 \times 10^4 \mu\text{m}^3$ ($1124 - 5642 \times 10^4 \mu\text{m}^3$). Detection of very small voids is due to the high resolution of the synchrotron scans, i.e. $0.65 \mu\text{m}/\text{pixel}$, in the referenced study. They suggested a bell-shape frequency distribution for the volume. The difference in the results is attributed to variations in material systems studied. Additionally, the large difference when compared to the results of the current study is most likely due to the fact that the modification of the processing parameters (and hence the voidage) in the current study is more excessive compared to the other studies.

4.2. Void orientation analysis

The out-of-plane orientation of voids (which is not the interest of this study) is close to zero, as can be noted in the tomographic slices of the cross-ply specimens in Fig. 2. The in-plane orientation of each void is defined as the angle between its major axis and the scan orientation. The latter is close (within few degrees) to the 0° direction of the laminate. The orientation is calculated in *Voxtex* and transformed to fall in the range $[-90^\circ, 90^\circ]$ for the $[\pm 45]_{2s}$ and $[\pm 67.5]_{2s}$ specimens and in $[-45^\circ, 135^\circ]$ for the $[67.5/22.5]_{2s}$, $[0/90]_{2s}$, and $[0/90]_{2s}$ specimens. Frequency distribution graphs of the orientation are plotted in Fig. 7 for the voids in each specimen. As can be noticed, the majority of voids are aligned in the nominal fiber directions existing in each laminate. For example, in the $[\pm 45]_{2s}$ specimen, most of the voids have 45° and -45° angles. It can be interpreted that voids are most likely aligned in the nominal fiber direction in each ply. Furthermore, in total, there are more voids oriented along the fibers in the middle plies than those aligned in the orientation of the outer plies.

The orientation of voids through the laminate thickness is analyzed and the results presented in Fig. 8. It shows the location of the centroids for the voids (in the thickness direction), projected on a line, versus their orientation. The clusters in this figure indicate that the majority of voids in each ply have the same orientation, which is close to the ply orientation. This figure also reveals that there exist more voids in the inner plies of the laminates than in the outer plies (explained in Section 4.3). This is the reason why in the orientation distribution graphs of all the specimens (Fig. 7), the number of voids with the orientation of the outer plies is smaller than that corresponding to the middle plies. This confirms the visual observation in tomographic slices (Fig. 2) that there are less voids in the outer plies. In the tomographic slices of the specimens, the cross-section of voids in plies with higher off-axis angles appears smaller, since voids are elongated features. As the two extreme cases, in the 0° plies, they appear as very long features, while in the 90° , they have small cross-sections.

The mean orientation of the voids that are (almost) aligned in one of the two ply directions in the laminate (ϕ_1, ϕ_2), the corresponding standard deviation (σ_1, σ_2), and the difference with the nominal ply orientations are reported in Table 3. The standard deviation around the mean orientations is within $10^\circ - 18^\circ$. The difference of the calculated mean with the nominal ply orientations is below 4° . This value may not be exactly equal to the actual misalignment of voids with the corresponding ply orientation since the plies themselves can be aligned slightly off from the nominal orientation. The highest total misalignment with the nominal orientations is found in the $[67.5/22.5]_{2s}$ specimen. The smallest corresponds to the $[\pm 67.5]_{2s}$ and $[0/90]_{4s}$ specimens, which, as explained in Section 4.1, have larger voids. It can be concluded that larger voids have better alignment with the fibers. For the $[\pm 67.5]_{2s}$, this can be also attributed to the smaller difference in the nominal orientations of plies, as explained in Section 4.1.1.

4.3. Void spatial location analysis

Location of voids is determined by the location of the centroid of their fitting ellipsoids. In order to obtain the cross-sectional (thickness-view) location of the voids in each specimen, the z coordinate of their centroids is plotted in Fig. 9 versus their centroids' x coordinate. This creates a projection of all void locations into an xz plane, where z and x represents the laminate thickness and length direction, respectively. This figure shows some concentrations of voids through the length.

Through the thickness, voids tend to exist more in the inner plies of the laminate than in the outer plies. This is because air evacuation is more difficult in the plies positioned further away from the laminate's surface. A probable explanation is that the temperature gradient generated through the laminate during the heat-up phase results in the inner plies curing after the outer plies, preventing further air evacuation. As the middle plies cure last, they therefore have the highest number of residual voids. Moreover, since the middle plies of each laminate have the same nominal orientation, they form a thicker ply, which is more likely to have voids. Fig. 9 shows that there are less intra-laminar voids close to the interfaces of plies with different nominal orientations. Instead, voids tend to concentrate more inside those plies. For example, the concentration of voids inside the plies is less significant in the $[\pm 67.5]_{2s}$ specimen, most likely due to the smaller difference between its plies' orientation, being 45° , compared to the other specimens, in which the difference is 90° .

Based on the higher number of voids in the inner plies, it can be hypothesized that the main source of voidage in the present study is entrapped air, both inside and in between the plies. Therefore, it is expected that voids in the inner plies are larger, and consequently longer. This is investigated by plotting their

elongation factor through the thickness (Fig. 10). It can be observed that the longest voids in the inner plies have a higher elongation factor than that of the longest voids in the outer plies.

The planar location of the voids in each specimen is obtained via plotting the x coordinate of their centroids versus their centroids' y coordinate, where y is the laminates' width direction. The graphs in Fig. 11, especially those corresponding to the $[\pm 45]_{2s}$ and $[\pm 67.5]_{2s}$ specimens, show some patterns, in which the ply orientations can be noticed. These graphs indicate that the location of voids in the planar view is not completely random and somehow follows the ply orientation, especially the orientation of the middle plies which host most of the voids. This suggests that air entrapment in each ply is not spatially uniform, but more significant in certain regions, which form concentration bands aligned in the ply orientation. A cause for the concentration can be the gaps known to exist between the tapes within each ply. They are a result of the laying process and, according to the manufacturer, are on average 1 mm in size. Plies with the same orientation, are laid with an offset of 20 mm, meaning that concentration bands of 1 mm with a spacing of 20 mm are expected to form.

5. Concluding remarks

A comprehensive analysis of voids' characteristics in carbon/epoxy composite laminates is performed. X-ray computed tomography is used to acquire 3D images of specimens with intentionally produced voids. Voids are segmented with gray value thresholding and fitted into equivalent ellipsoids. Five different lay-ups are analyzed with respect to voids' shape, size, orientation, and spatial distribution. Statistical results, including means, standard deviations, percentiles, and ranges of the semi-minor, semi-medium, and semi-major axes are reported. Smaller ranges can be defined to present the size parameters of the majority of voids.

A fairly strong linear relationship is found between the semi-minor and semi-major axes of voids. Their volume and elongation factor have a moderate linear correlation. Voids are found to be elongated. They are on average 13.5 times larger in their axial direction than in their radial direction. Voids have an irregular cross-section, which can be simplified to an ellipse for shape quantification. It is found that half of the voids have a cross-section roundness factor above 0.64, where a roundness factor of 0 means a totally flat cross-section and 1 equates to a fully circular cross-section. The majority of voids are aligned in the nominal fiber directions existing in each laminate, meaning that they are oriented in the fiber direction in each ply, with an average misalignment below 4° .

Moving through the thickness from the surface to the middle of the laminate, the population of voids increases, and they become more elongated. This can most likely be attributed to the difficulty with air

evacuation from the inner plies, during the cure cycle. There are more voids in the thicker laminate. The laminate's stacking sequence influences the size and shape of voids. The in-plane location of voids is not completely random and tends to be associated with the fiber direction.

This study demonstrates successful application of micro-CT to the characterization of voids. It emphasizes the importance of using 3D analysis for investigating void characteristics, particularly their shape and size. It provides a broad dataset on void characteristics, which can be useful in various studies, including in modeling works that simulate the effect of voids on mechanical and hygrothermal properties of similar composite systems. The full dataset and the original image stacks are presented as a *Data in Brief* article in [1]. Whilst the voids in this study are intentionally induced and excessive, naturally occurring voids in similar materials should have analogous shape and orientation distribution since these characteristics are controlled by the microstructure. For quantitative use of the results, one needs to account for the void content and size correction factors for their own material system. The current dataset will be in any case useful as a real-world example for virtual porosity generators.

Acknowledgements

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* (VLAIO-project number 140147/140158), which is part of the research program *MacroModelMat (M3)*, coordinated by *Siemens (Siemens PLM Software, Belgium)*. They also acknowledge the cooperation of K. Verjans and S. Wathiong in production of the composite laminates at *SABCA Limburg NV*, Belgium. The study is also supported by the KU Leuven Research Council, project C24/17/052. Revision of the manuscript by Paul Woody is highly appreciated. S.V. Lomov holds *Toray Chair* for Composite Materials at KU Leuven, support of which is gratefully acknowledged.

References

1. Mehdikhani, M., I. Straumit, L. Gorbatikh, and S.V. Lomov, *A dataset of voids' characteristics in multidirectional carbon fiber/epoxy composite laminates, obtained using X-ray micro-computed tomography*. Data in Brief. **In press**.
2. Mehdikhani, M., L. Gorbatikh, I. Verpoest, and S.V. Lomov, *Voids in fiber-reinforced polymer composites: a review on their formation, characteristics, and effects on mechanical performance*. Journal of Composite Materials, 2018: p. In print.
3. Huang, H. and R. Talreja, *Effects of void geometry on elastic properties of unidirectional fiber reinforced composites*. Composites Science and Technology, 2005. **65**(13): p. 1964-1981.

- 1 4. Chowdhury, K., R. Talreja, and A.A. Benzerga, *Effects of manufacturing-induced voids on local failure in polymer-based*
2 *composites*. Journal of Engineering Materials and Technology, 2008. **130**(2): p. 021010.
- 3
- 4 5. Ashouri Vajari, D., C. González, J. Llorca, and B.N. Legarth, *A numerical study of the influence of microvoids in the*
5 *transverse mechanical response of unidirectional composites*. Composites Science and Technology, 2014. **97**: p. 46-54.
- 6
- 7 6. Maragoni, L., P.A. Carraro, and M. Quaresimin, *Effect of voids on the crack formation in a [45/−45/0]_s laminate*
8 *under cyclic axial tension*. Composites Part A: Applied Science and Manufacturing, 2016. **91**, Part 2(493-500).
- 9
- 10 7. Dong, J. and N. Huo, *A two-scale method for predicting the mechanical properties of 3D braided composites with internal*
11 *defects*. Composite Structures, 2016. **152**: p. 1-10.
- 12
- 13 8. Little, J.E., X. Yuan, and M.I. Jones, *Characterisation of voids in fibre reinforced composite materials*. NDT & E
14 *International*, 2012. **46**: p. 122-127.
- 15
- 16 9. Birt, E. and R. Smith, *A review of NDE methods for porosity measurement in fibre-reinforced polymer composites*. Insight-
17 *Non-Destructive Testing and Condition Monitoring*, 2004. **46**(11): p. 681-686.
- 18
- 19 10. Adams, R.D. and P. Cawley, *A review of defect types and nondestructive testing techniques for composites and bonded joints*.
20 *NDT International*, 1988. **21**(4): p. 208-222.
- 21
- 22 11. Judd, N.C.W. and W.W. Wright, *Voids and Their Effects on the Mechanical Properties of Composites - An Appraisal*.
23 *Sampe Journal*, 1978. **14**(1): p. 10-14.
- 24
- 25 12. Centea, T. and P. Hubert, *Measuring the impregnation of an out-of-autoclave prepreg by micro-CT*. Composites Science
26 *and Technology*, 2011. **71**(5): p. 593-599.
- 27
- 28 13. Vila, J., F. Sket, F. Wilde, G. Requena, C. Gonzalez, and J. Llorca, *An in situ investigation of microscopic infusion*
29 *and void transport during vacuum-assisted infiltration by means of X-ray computed tomography*. Composites Science and
30 *Technology*, 2015. **119**: p. 12-19.
- 31
- 32 14. Nikishkov, Y., G. Seon, and A. Makeev, *Structural analysis of composites with porosity defects based on X-ray computed*
33 *tomography*. Journal of Composite Materials, 2013.
- 34
- 35 15. Centea, T., L.K. Grunenfelder, and S.R. Nutt, *A review of out-of-autoclave preregs – Material properties, process*
36 *phenomena, and manufacturing considerations*. Composites Part A: Applied Science and Manufacturing, 2015. **70**: p.
37 132-154.
- 38
- 39 16. Gigliotti, M., Y. Pannier, R.A. Gonzalez, M.C. Lafarie-Frenot, and S.V. Lomov, *X-ray micro-computed-tomography*
40 *characterization of cracks induced by thermal cycling in non-crimp 3D orthogonal woven composite materials with porosity*.
41 *Composites Part A: Applied Science and Manufacturing*, 2018. **112**: p. 100-110.
- 42
- 43
- 44
- 45
- 46
- 47
- 48
- 49
- 50
- 51
- 52
- 53
- 54
- 55
- 56
- 57
- 58
- 59
- 60
- 61
- 62
- 63
- 64
- 65

17. Schell, J.S.U., M. Renggli, G.H. van Lenthe, R. Müller, and P. Ermanni, *Micro-computed tomography determination of glass fibre reinforced polymer meso-structure*. Composites Science and Technology, 2006. **66**(13): p. 2016-2022.
18. Schell, J.S.U., M. Deleglise, C. Binetruy, P. Krawczak, and P. Ermanni, *Numerical prediction and experimental characterisation of meso-scale-voids in liquid composite moulding*. Composites Part a-Applied Science and Manufacturing, 2007. **38**(12): p. 2460-2470.
19. Hernández, S., F. Sket, J.M. Molina-Aldareguía, C. González, and J. Llorca, *Effect of curing cycle on void distribution and interlaminar shear strength in polymer-matrix composites*. Composites Science and Technology, 2011. **71**(10): p. 1331-1341.
20. Hernández, S., F. Sket, C. González, and J. Llorca, *Optimization of curing cycle in carbon fiber-reinforced laminates: Void distribution and mechanical properties*. Composites Science and Technology, 2013. **85**: p. 73-82.
21. Nikishkov, Y., L. Airoidi, and A. Makeev, *Measurement of voids in composites by X-ray Computed Tomography*. Composites Science and Technology, 2013. **89**: p. 89-97.
22. Madra, A., N.E. Hajj, and M. Benzeggagh, *X-ray microtomography applications for quantitative and qualitative analysis of porosity in woven glass fiber reinforced thermoplastic*. Composites Science and Technology, 2014. **95**: p. 50-58.
23. Yang, P. and R. Elhajjar, *Porosity Content Evaluation in Carbon-Fiber/Epoxy Composites Using X-ray Computed Tomography*. Polymer-Plastics Technology and Engineering, 2014. **53**(3): p. 217-222.
24. Zhang, D., D. Heider, S.G. Advani, and J.W. Gillespie. *Out of autoclave consolidation of voids in continuous fiber reinforced thermoplastic composites*. in *Proceedings of the SAMPE conference, Long Beach, CA, USA*. 2013.
25. Abdelal, N. and S.L. Donaldson, *Comparison of methods for the characterization of voids in glass fiber composites*. Journal of Composite Materials, 2017. **0**(0): p. 0021998317710083.
26. Di Landro, L., A. Montalto, P. Bettini, S. Guerra, F. Montagnoli, and M. Rigamonti, *Detection of Voids in Carbon/Epoxy Laminates and Their Influence on Mechanical Properties*. Polymers & Polymer Composites, 2017. **25**(5): p. 371.
27. Sisodia, S.M., S.C. Garcea, A.R. George, D.T. Fullwood, S.M. Spearing, and E.K. Gamstedt, *High-resolution computed tomography in resin infused woven carbon fibre composites with voids*. Composites Science and Technology, 2016. **131**: p. 12-21.
28. Straumit, I., S.V. Lomov, and M. Wevers, *Quantification of the internal structure and automatic generation of voxel models of textile composites from X-ray computed tomography data*. Composites Part A: Applied Science and Manufacturing, 2015. **69**: p. 150-158.

29. Melenka, G.W., E. Lepp, B.K.O. Cheung, and J.P. Carey, *Micro-computed tomography analysis of tubular braided composites*. Composite Structures, 2015. **131**: p. 384-396.
30. Tserpes, K.I., A.G. Stamopoulos, and S.G. Pantelakis, *A numerical methodology for simulating the mechanical behavior of CFRP laminates containing pores using X-ray computed tomography data*. Composites Part B: Engineering, 2016. **102**: p. 122-133.
31. Scott, A.E., I. Sinclair, S.M. Spearing, M.N. Mavrogordato, and W. Hepples, *Influence of voids on damage mechanisms in carbon/epoxy composites determined via high resolution computed tomography*. Composites Science and Technology, 2014. **90**: p. 147-153.
32. Mehdikhani, M., N.A. Petrov, I. Straumit, A.R. Melro, S.V. Lomov, and L. Gorbatikh, *The effect of voids on matrix cracking in composite laminates as revealed by combined computations at the micro- and meso-scales*. Composites Part A: Applied Science and Manufacturing, 2019. **117**: p. 180-192.
33. Kakasery, J., V. Arumugam, K. Abdul Rauf, D. Bull, A.R. Chambers, C. Scarponi, and C. Santulli, *Cure cycle effect on impact resistance under elevated temperatures in carbon prepreg laminates investigated using acoustic emission*. Composites Part B: Engineering, 2015. **75**: p. 298-306.
34. Ladeveze, P. and E. LeDantec, *Damage modelling of the elementary ply for laminated composites*. Composites Science and Technology, 1992. **43**(3): p. 257-267.
35. Mehdikhani, M., E. Steensels, A. Standaert, K.A.M. Vallons, L. Gorbatikh, and S.V. Lomov, *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*. Composites Part B: Engineering, 2018. **154**: p. 138-147.
36. Bert, M., D. Manuel, L. Denis Van, N.B. Matthieu, B. Loes, P. Elin, C. Veerle, and H. Luc Van, *HECTOR: A 240kV micro-CT setup optimized for research*. Journal of Physics: Conference Series, 2013. **463**(1): p. 012012.
37. Vlassenbroeck, J., B. Masschaele, V. Cnudde, M. Dierick, K. Pieters, L. Van Hoorebeke, and P. Jacobs, *Octopus 8: A High Performance Tomographic Reconstruction Package for X-ray Tube and Synchrotron micro-CT*. Advances in X-ray Tomography for Geomaterials, 2010: p. 167-173.
38. Ahrens, J., B. Geveci, and C. Law, *Paraview: An end-user tool for large data visualization*. The visualization handbook, 2005. **717**.
39. Nguyen, N.Q., M. Mehdikhani, I. Straumit, L. Gorbatikh, L. Lessard, and S.V. Lomov, *Micro-CT measurement of fibre misalignment: Application to carbon/epoxy laminates manufactured in autoclave and by vacuum assisted resin transfer moulding*. Composites Part A: Applied Science and Manufacturing, 2018. **104**: p. 14-23.

Figure captions

Figure 1. 3D visualization of voids (in blue) in different specimens detected via micro-CT and the schematics of an ellipsoid used for fitting of voids and of an elliptic cross-section with a roundness factor of 0.6 – gray represents the volume of investigation and V_v denotes the void content.

Figure 2. Example tomographic slices of different specimens, showing cross-sections of voids – on the right side, the corresponding blow-ups of the indicated windows are shown.

Figure 3. Frequency distributions of the semi-minor axis and the orientation of voids in the $[\pm 45]_{2s}$ specimen (left) before and (right) after filtering.

Figure 4. Size parameters, i.e. semi-minor, semi-medium, and semi-major axes, of the ellipsoids fitted to the detected voids in different specimens – several of the larger values have been excluded to enhance the visualization of the majority of the data.

Figure 5. Geometric mean of the transversal semi-axes and shape parameters (elongation and cross-section roundness factors) of the ellipsoids fitted to the detected voids in different specimens, where a , b , and c are respectively the semi-major, semi-medium, and semi-minor axis of the ellipsoids – several of the larger values have been excluded to enhance the visualization of the majority of the data.

Figure 6. Elongation factor of the voids' fitting ellipsoids versus their actual volume in different specimens, showing that larger voids are typically longer – several of the larger values have been excluded to enhance the visualization of the majority of the data. The corresponding correlation coefficients are presented in Table 2.

Figure 7. Frequency distribution of voids' in-plane orientation, defined as the angle between the major axis and the scan orientation, in different specimens.

Figure 8. Voids' in-plane orientation, defined as the angle between the major axis and the scan orientation, through the thickness (projected on a line) for different specimens – some boundary regions from top and bottom are excluded because the corresponding raw data was not processable – the location of the ply interface lines is approximate.

Figure 9. Cross-sectional (thickness-view) location of the voids' fitting ellipsoids, projected into xz one plane, in different specimens – some boundary regions are excluded because the corresponding raw data was not processable – the location of the ply interface lines is approximate.

Figure 10. Elongation factor of the voids' fitting ellipsoids through the thickness (projected on a line) for different specimens – some boundary regions from top and bottom are excluded because the corresponding raw data was not processable – the location of the ply interface lines is approximate.

Figure 11. Planar location of the voids' fitting ellipsoids in different specimens – the gray regions were not analyzed because the corresponding raw data was not processable.

Tables

Table 1. Statistics of the size and shape parameters of the voids detected in different specimens.

Parameter	Laminate	Mean	Standard deviation	Coefficient of variation (%)	Skewness (no unit)	Minimum	Maximum	50 th percentile	95 th percentile
Semi-minor axis (μm)	$[\pm 45]_{2s}$	7.8	3.4	43.9	6.0	4.2	52.6	7.2	11.7
	$[\pm 67.5]_{2s}$	10.0	4.6	45.8	3.9	4.7	60.3	8.8	18.1
	$[67.5/22.5]_{2s}$	7.7	3.3	43.6	3.5	4.2	36.4	6.7	13.5
	$[0/90]_{2s}$	7.9	3.3	41.6	3.5	4.3	32.3	7	14.4
	$[0/90]_{4s}$	10.1	4.7	47.4	2.8	4.3	48.0	8.6	19
	Average	8.8	-	-	-	4.3	45.9	7.7	15.4
Semi-medium axis (μm)	$[\pm 45]_{2s}$	16.2	34.0	209.7	12.4	6.4	651.8	10.4	37.8
	$[\pm 67.5]_{2s}$	25.7	58.8	228.5	8.2	6.2	760.9	13.5	61.8
	$[67.5/22.5]_{2s}$	17.2	30.3	176.2	16.5	6.1	770.9	12.2	37.8
	$[0/90]_{2s}$	18.2	41.7	228.8	11.2	5.6	614.1	11.4	35.6
	$[0/90]_{4s}$	23.3	52.0	223.1	10.5	5.2	900.0	13	55.2
	Average	20.3	-	-	-	5.9	739.5	12.2	46.1
Semi-major axis (μm)	$[\pm 45]_{2s}$	175.1	351.9	200.9	6.8	10.1	4781.4	78.9	685.6
	$[\pm 67.5]_{2s}$	328.3	483.5	147.3	3.0	10.1	4341.2	147.9	1297.6
	$[67.5/22.5]_{2s}$	161.3	328.2	203.5	4.9	9.4	4084.9	38.7	744.5
	$[0/90]_{2s}$	190.8	418.1	219.1	6.8	9.4	5251.3	51.5	874.6
	$[0/90]_{4s}$	265.1	457.3	172.5	5.1	9.4	5760.4	100.3	1040.3
	Average	221.6	-	-	-	9.7	4843.9	84.4	929
Volume (μm^3)	$[\pm 45]_{2s}$	2.0×10^5	12.2×10^5	593.9	13.4	1124	261.5×10^5	0.2×10^5	5.3×10^5
	$[\pm 67.5]_{2s}$	5.6×10^5	18.6×10^5	334.4	9.8	1124	334.4×10^5	0.7×10^5	29.4×10^5
	$[67.5/22.5]_{2s}$	2.5×10^5	12.6×10^5	512.1	11.0	1124	263.1×10^5	0.1×10^5	9.0×10^5
	$[0/90]_{2s}$	2.1×10^5	10.6×10^5	499.1	12.9	1124	176.1×10^5	0.1×10^5	10.4×10^5
	$[0/90]_{4s}$	5.4×10^5	24.5×10^5	458.6	11.5	1124	564.2×10^5	0.4×10^5	21.5×10^5
	Average	3.8×10^5	-	-	-	1124	319.9×10^5	0.3×10^5	15.1×10^5
Geometric mean of transversal semi-axes (μm)	$[\pm 45]_{2s}$	10.6	9.0	85.0	8.6	5.8	156.7	8.6	19.1
	$[\pm 67.5]_{2s}$	14.6	14.4	98.3	7.3	6.0	214.3	11.2	32.8
	$[67.5/22.5]_{2s}$	11.0	7.9	71.6	7.2	5.9	159.3	9	22.2
	$[0/90]_{2s}$	11.2	9.0	80.4	7.1	5.5	125.1	8.9	22.4
	$[0/90]_{4s}$	14.1	12.2	86.4	5.9	5.1	164.6	10.6	31.3
	Average	12.5	-	-	-	5.7	164.0	9.7	25.7
Elongation factor	$[\pm 45]_{2s}$	13.2	13.8	104.6	3.0	1.4	122.2	8.9	39.5
	$[\pm 67.5]_{2s}$	18.0	17.4	96.5	1.4	1.3	105.1	12.3	54.0
	$[67.5/22.5]_{2s}$	10.3	13.1	127.5	2.4	1.2	98.5	4.1	37.7
	$[0/90]_{2s}$	12.8	16.7	130.5	2.9	1.3	150.7	5.8	49.7
	$[0/90]_{4s}$	15.2	15.8	104.1	2.0	1.2	129.2	9.1	45.1
	Average	13.5	-	-	-	1.3	121.2	8.2	45.2
Cross-section roundness factor	$[\pm 45]_{2s}$	0.66	0.18	27.89	-0.95	0.03	0.98	0.68	0.90
	$[\pm 67.5]_{2s}$	0.63	0.21	32.73	-0.71	0.03	0.99	0.67	0.90
	$[67.5/22.5]_{2s}$	0.58	0.19	32.21	-0.12	0.03	1.00	0.58	0.86
	$[0/90]_{2s}$	0.61	0.18	29.45	-0.46	0.03	0.97	0.62	0.87
	$[0/90]_{4s}$	0.63	0.19	30.32	-0.60	0.02	1.00	0.65	0.90
	Average	0.61	-	-	-	0.03	0.99	0.64	0.89

Table 2. Results of linear relationship analysis between some of the void characteristics in different specimens.

Laminate	Correlation coefficient between			
	semi-major axis & semi-minor axis	semi-major axis & semi-medium axis	semi-medium axis & semi-minor axis	elongation factor & volume
$[\pm 45]_{2s}$	0.77	0.47	0.57	0.37
$[\pm 67.5]_{2s}$	0.69	0.54	0.61	0.36
$[67.5/22.5]_{2s}$	0.79	0.48	0.51	0.44
$[0/90]_{2s}$	0.75	0.44	0.45	0.49
$[0/90]_{4s}$	0.73	0.41	0.47	0.36
Average	0.75	0.47	0.52	0.40

Table 3. Orientation statistics of the voids detected in different specimens.

Laminate	Calculated mean orient. ϕ_1 (°)	Standard deviation σ_1 (°)	Difference of ϕ_1 with the nominal ply orient. 1 (°)	Calculated mean orient. ϕ_2 (°)	Standard deviation σ_2 (°)	Difference of ϕ_2 with the nominal ply orient. 2 (°)
$[\pm 45]_{2s}$	48.0	17.5	3.0	-43.8	11.5	1.2
$[\pm 67.5]_{2s}$	66.4	16.9	1.1	-66.5	10.2	1.0
$[67.5/22.5]_{2s}$	69.8	15.7	2.3	18.6	13.6	3.9
$[0/90]_{2s}$	-1.9	17.5	1.9	91.3	12.2	1.3
$[0/90]_{4s}$	0.9	13.6	0.9	90.8	11.5	0.8

Data in Brief

[Click here to download Data in Brief: Datainbrief_voids microCT dataset_revised.docx](#)

CONFLICT OF INTEREST DECLARATION

We wish to confirm that there are no known conflicts of interest associated with this publication and there has been no significant financial support for this work that could have influenced its outcome.

We confirm that the manuscript has been read and approved by all named authors and that there are no other persons who satisfied the criteria for authorship but are not listed. We further confirm that the order of authors listed in the manuscript has been approved by all of us.

We confirm that we have given due consideration to the protection of intellectual property associated with this work and that there are no impediments to publication, including the timing of publication, with respect to intellectual property. In so doing, we confirm that we have followed the regulations of our institutions concerning intellectual property.

We understand that the Corresponding Author is the sole contact for the Editorial process (including Editorial Manager and direct communications with the office). He is responsible for communicating with the other authors about progress, submissions of revisions and final approval of proofs. We confirm that we have provided a current, correct email address which is accessible by the Corresponding Author and which has been configured to accept email from (mahoor.mehdikhani@kuleuven.be).

Signed by Mahoor Mehdikhani, the corresponding author, on behalf of all the authors, i.e. Mahoor Mehdikhani, Ilya Straumit, Larissa Gorbatiikh, Stepan V. Lomov

Mahoor Mehdikhani

04 Jun 2019