

Analysis of void morphology in composite laminates using micro-computed tomography

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Abstract. VoxTex software for quantification of micro-CT images provides useful tools for analysis of voids in fibre reinforced composites. These tools are applied in the current work to (1) carbon fibre laminate (-45/0/45/-20/0/20/90/0)so, made with automated dry fibre placement and vacuum assisted resin transfer moulding and to (2) carbon fibre laminates [0/90]ns, made by prepreg technology and cured in autoclave with regime proliferating void formation. The materials contain very different porosity levels (below 1% for (1) and few percent in (2)). The segmentation and image processing algorithms allow identification of the overall void content and quantification of the void dimensions and their orientation in the form of distribution functions. These distributions are presented and their characteristic features are discussed. A typical peculiarity of segmented micro-CT images is presence of small (few pixels in size) voids, which persist even after the spurious “void pixels” are eliminated. Whether these micro-void-features represent real microscopic voids in the material is discussed based on the comparison with SEM images.

1. Introduction

Voids are common manufacturing defects in Fiber-Reinforced Composites (FRCs), forming inevitably in all processing techniques. They can cause degradation of mechanical properties, especially those controlled by matrix properties [1]. If their effect can be precisely quantified, limits allowing certain amount of voids can be determined, depending on the loading scenario and application. Evaluation of the voids' effect asks for their accurate characterization, meaning identifying their characteristics, i.e. shape, size, orientation, and spatial distribution. Once these characteristics are known, their effect on mechanical properties can be investigated experimentally. Moreover, when the void allowance limits are determined, deciding if a composite part is accepted/rejected needs accurate characterization of the voids in that part.

Quantification of the effect of voids is also possible, and maybe even less difficult, through simulations. However, the modelling approach also needs information on voids' characteristics to produce reliable results. Therefore, characterization of voids is essential for evaluation of their effects.

There has been some research conducted on the application of micro-computed tomography (micro-CT) to characterization of voids, see more in [1]. However, works that provide a complete set of data on void characteristics in carbon/epoxy laminates are still rare. In the present study, we perform a full 3D analysis of the voids in carbon/epoxy laminates with different stacking sequences using micro-CT. Micro-CT results are processed with *VoxTex*, an in-house software, to obtain details of voids' size, morphology, orientation, and spatial distribution.



2. Materials

There are two materials studied. One of them is an aerospace-grade carbon/epoxy composite made from unidirectional prepreg tapes. The lay-up is prepared with Automated Tape Laying (ATP) and the curing is performed in an autoclave, both at *SABCA Limburg NV, Belgium*. In order to produce laminates with voids for research purpose, the manufacturer's cure cycle was not followed, instead a low-pressure-temperature cure cycle was applied, with debulking prior to cure and vacuum application during cure. Two lay-ups were produced: $[0/90]_{2s}$, and $[0/90]_{4s}$. Micro-CT samples with the size of $\sim 15 \text{ mm} \times 10 \text{ mm} \times 1.5 \text{ mm}$ for the 8-ply lay-ups and $\sim 15 \text{ mm} \times 10 \text{ mm} \times 3 \text{ mm}$ for the 16-ply lay-up were cut from the produced plates.

The other material is produced at *NLR, The Netherlands*, with Automated Dry Fibre Placement (ADFP) followed by Vacuum Assisted Resin Transfer Moulding (VARTM). The composite reinforcement is Hexcel HiTape UD126 AS7/V800E/QD2/6.35mm, which is a unidirectional dry tow of AS7 carbon fibres. The areal density of the tow is 126 g/m^2 . The tow width and thickness is 6.35 mm ($1/4 \text{ in.}$) and 0.135 mm , respectively. The dry preform is laid up as $[-45/0/45/-20/0/20/90/0]_{so}$, where the subscript "so" stands for Symmetric Odd, indicating that there is only one 0° ply in the middle of the laminate. The preform is infused with HexFlow RTM 6 resin. The nominal fibre volume fraction of the final composite is 50%. This is the same material as studied in [2].

3. Micro-CT image acquisition and processing

The ATP samples were scanned with the *HECTOR* system at *Ghent University Centre for X-ray Tomography (UGCT)*. The scan volume was $12 \times 12 \times 12 \text{ mm}^3$, and the voxel size was $6.55 \mu\text{m}$. The scan time for each specimen was about 45 minutes. The reconstruction of the projection data to tomographic slice images was performed with the *Octopus* reconstruction software.

The ADFP material was scanned with a Skyscan 1172 X-ray computed tomography system in the Department of Materials Engineering of *KU Leuven*. 2D X-ray images were acquired for 180° of tomographic rotation with a rotation step of 0.2° . The nominal spot size and the exposure time were 11.27 mm and 1767 ms . The scan volume is $6.0 \times 6.5 \times 2.0 \text{ mm}^3$, and the voxel size was $2.0 \mu\text{m}$.

The segmentation and data processing is carried out using *VoxTex* software [3] developed at *KU Leuven*. After segmentation, voids are fitted, in *VoxTex*, to equivalent ellipsoids. For each ellipsoid, the minor, medium, and major axes as well as the location of its centroid and orientation are calculated. The orientation of the voids is defined as the angle between their major axis and the scan direction.

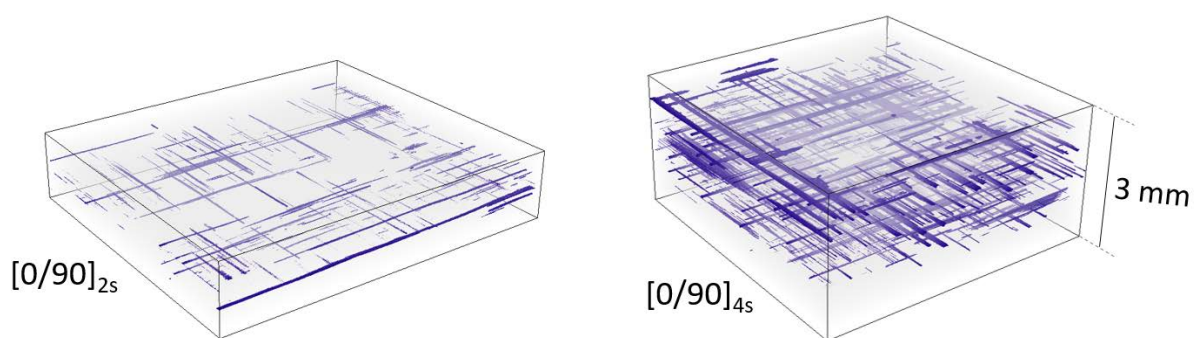


Figure 1. 3D representations of voids (in purple) in the two ATP laminates detected with micro-CT.

4. Voids characterization in ATP materials

The 3D representation of the segmented voids in the scanned laminates is visualized in figure 1. Voids, in purple, have elongated features orientated in the fiber directions in each ply of the laminates. There is a higher level of voidage in the $[0/90]_{4s}$ laminate (16 plies) than in the $[0/90]_{2s}$ laminate (8 plies).

For each void, fitted to an ellipsoid after segmentation, three size parameters are defined: semi-minor, semi-medium, and semi-major axis of the ellipsoid. The frequency distribution of these parameters is shown in figure 2.

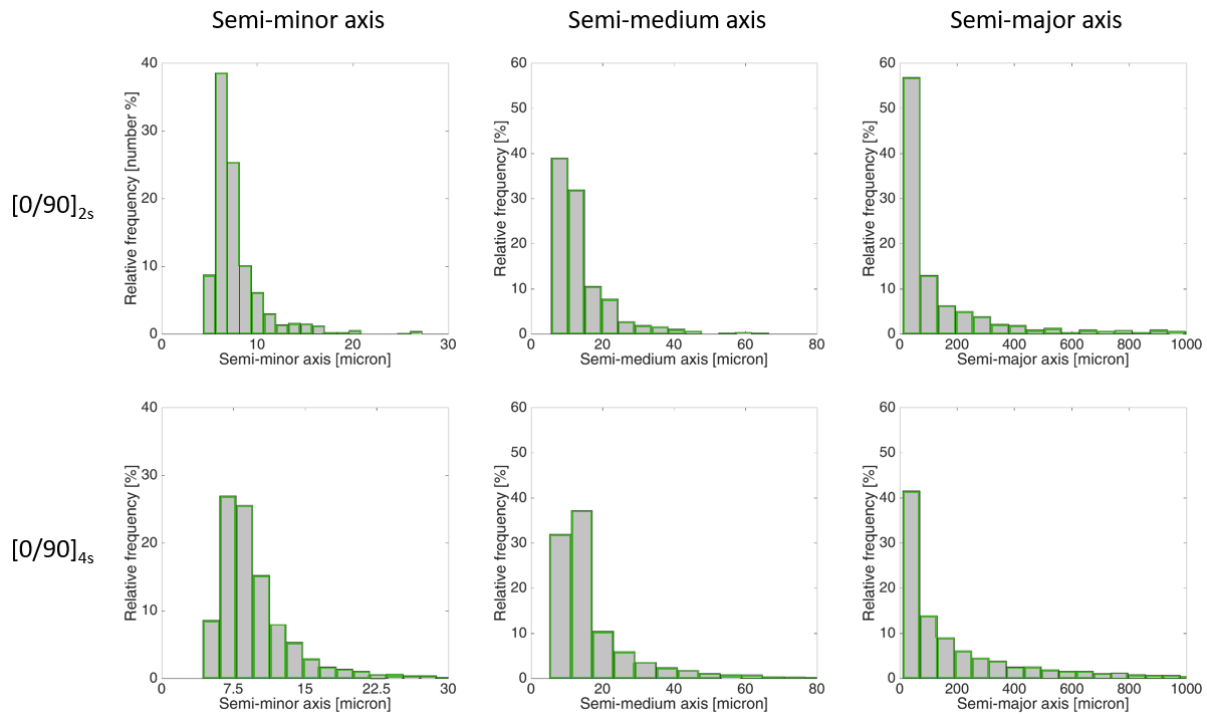


Figure 2. Frequency distribution of the size parameters of the detected voids.

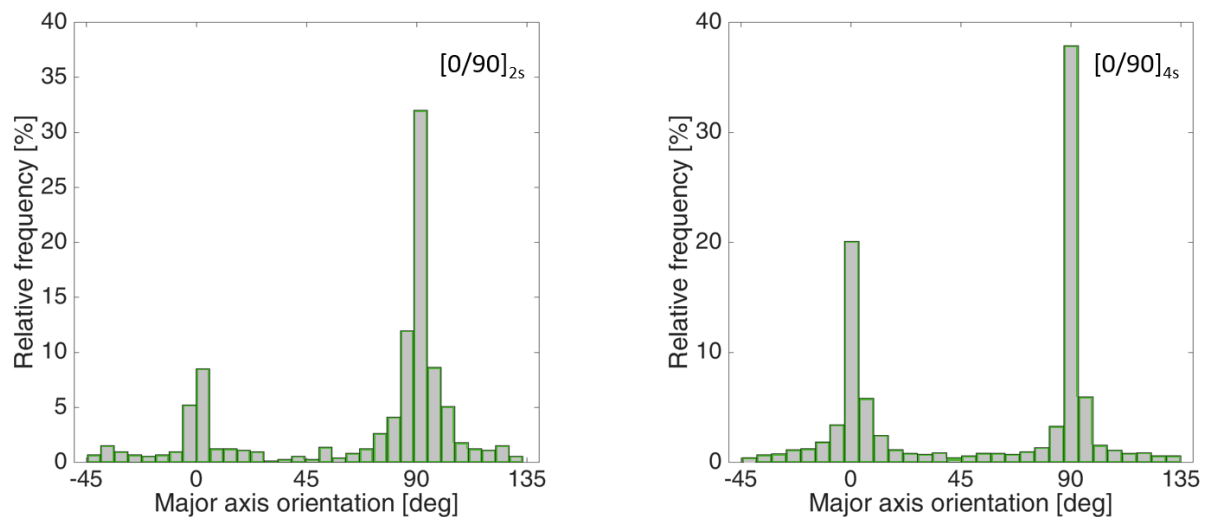


Figure 3. Frequency distribution of the orientation of the detected voids.

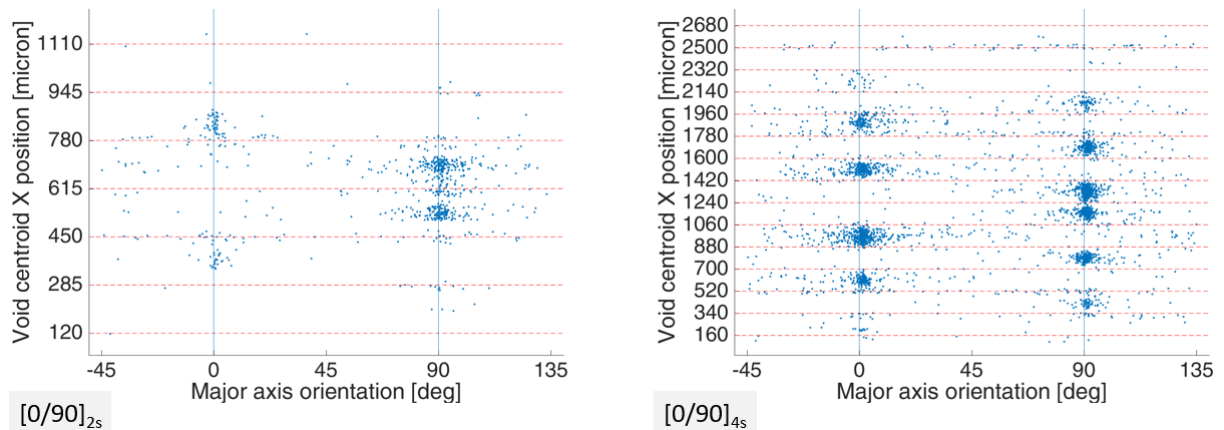


Figure 4. Voids' in-plane orientation, defined as the angle between the major axis and the scan orientation, through the thickness of different laminates.

The frequency distribution of voids' orientation is shown in figure. As can be noted, the majority of voids are oriented in the nominal fiber direction in the plies of each laminate.

The orientation of voids through the thickness of the laminate is shown in figure 4. The clusters in this figure indicate that the majority of voids in each ply have the same orientation, and it 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.

5. Voids characterization in ADFP material

The ADFP material has a very low void content, below 0.2%. Segmentation of the image for these small voids presents a challenge, as the size of some voids is on the level of the image noise. The image segmentation was enhanced (the image noise reduced) by applying Gaussian filter to the micro-CT image and by using a reduced greyscale dynamic range on original data set. The processed and thresholded images show agreement between the size and shape of clearly visible voids on the Micro-CT cross-section images and the segmented images (Figure 5).

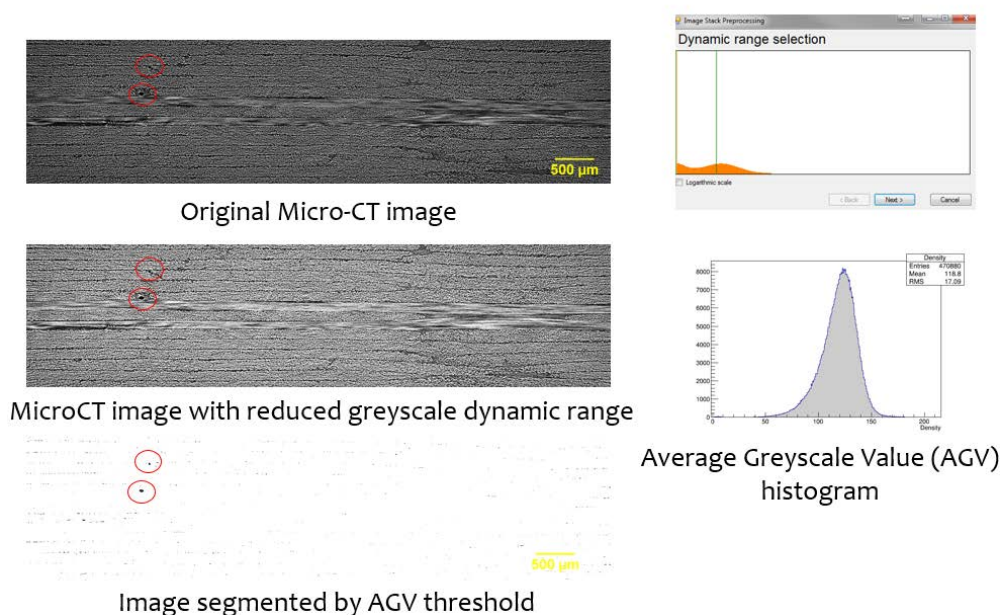


Figure 5. Image processing and thresholding for the ADFP material.

However, a large number of small voids that are seen in the segmented images and not clearly recognisable in the section are suspicious. The question is whether they are really present in the material, or simply a result of rudiments of the image noise? The former (they are real) seems to be supported by localization of these features: they clearly follow the tape layers boundaries. To verify or disprove their presence in the material, scanning electron microscopy (SEM) was performed (Figure 6). This additional characterization has confirmed that these micro-voids are indeed present in the laminate, and their dimensions are comparable with the dimensions seen in the segmented micro-CT images.

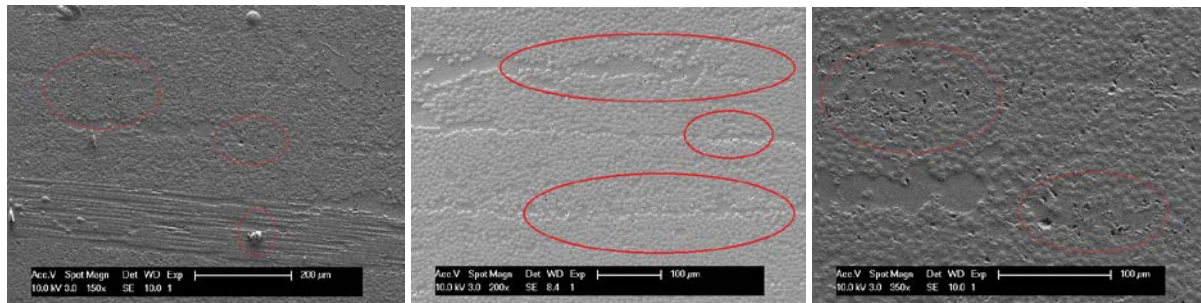


Figure 6. Micro-voids seen with SEM, ADFP material.

6. Conclusions

Micro-CT, and specifically VoxTex software, can be used not only to determine the void content in carbon fibre laminates composite but also other important information such as voids' spatial distribution and their size and shape. With this technique even very small voids (few pixels in size) in the composite with the overall void content below 0.2% could be captured.

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