3D Characterisation of Void Distribution in Resin Film Infused Composites

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Abstract
This paper presents an investigation of voids in carbon fibre reinforced epoxy composites manufactured by resin film infusion using X-ray computed tomography (XCT). Two panels were investigated, one formed with a high viscosity resin, the other with a lower viscosity resin. The study focusses on the characterisation of the 3D distribution of voids in the panels. A new approach to the measurement of defect distribution demonstrated that in both panels, the voids were located close to the binder yarn. When the low viscosity resin was employed, the void distribution was more uniform throughout the panel thickness whereas for the high viscosity resin, the voids were mainly localised in the central part of the panel. Both qualitative and quantitative data were obtained giving extensive, three dimensional information which aids a better understanding of the manufacturing process.

Keywords: X-ray computed tomography, CT, carbon fibre reinforced composite, toughening, resin film infusion, RFI, voids/porosity, 3D void distribution, distance map.

1 Introduction
The properties of a composite are detrimentally affected by voids introduced during the manufacturing process [1-4]. It is therefore of interest to characterise the voids in terms of size, shape and spatial distribution. The common method of determining the void volume fraction is acid digestion for carbon fibre reinforced composites [5] and matrix burn off for glass fibre reinforced composites [6]. Both are destructive techniques that can only provide data from a limited volume of the panel. The void size, shape and spatial distribution are usually determined by means of two-dimensional analysis techniques, i.e. optical or electron microscopy [7]. However, it is very difficult to adequately assess the number, position and morphology of these defects with these techniques. X-ray computed tomography (XCT) is emerging as an alternative technique for porosity measurements for composites [8, 9] and can potentially provide a new impetus to void distribution characteristics in 3D. This is important because the influence of the void spatial distribution on the properties of a composite changes according to the test performed. For example, in a mode I interlaminar fracture test; the presence of voids in the vicinity of the crack affects its propagation [10]. As a result it is important to determine the void distribution as a function of the manufacturing technique and the associated processing parameters in order to improve quality [3, 4]. That is the aim of this paper for resin film infusion (RFI).
2 Experimental

2.1 Materials

Two composite panels (labeled L and H) were investigated in this study. The epoxy resins used in both panels were triglycidylaminophenol; TGAP (Araldite® MY0510, Huntsman) and tetraglycidyl-4,4’-diaminodiphenylmethane; TGDDM (Araldite® MY721, Huntsman) and the hardener was 4,4’-diaminophenyl sulfone; DDS (Aradur® 976-1, Huntsman). One panel (designated L) was made from unmodified resin whereas the other panel (designated H) used the same resin modified with polyethersulfone; PES (Virantage® VW-10300 FP, Solvay) to improve fracture toughness, which results in higher viscosity. The reinforcement of the composite was a unidirectional carbon fibre fabric, which was supplied by Sigmatex, UK in which 12 k carbon tows were bound with a fine glass fibre yarn with a spacing of approximately 6 mm to give a fabric of 445 g.m⁻².

Composite laminates were manufactured by resin film infusion (RFI) which is suitable for high viscosity resins [11, 12]. Resin films were degassed at 130 °C for 1 hour and then were immediately frozen. The infusion setup is presented in Figure 1. The laminates were cured at 130 °C, 165 °C and 200 °C for 2 hours at each temperature. Vacuum was applied throughout the cycle for the infusion to take place.

Each panel (approximately 5 mm thick) was cut into five 25 mm wide strips, along the 0° direction. The middle three specimens of each panel were used for the study and the middle section of each specimen was scanned.

![Figure 1: Resin film infusion setup.](image)

2.2 Techniques

2.2.1 X-ray computed tomography

The composite specimens were scanned at the Henry Moseley X-ray Imaging Facility on the Nikon Metrology 225/320 kV Custom Bay. The system was equipped with a 225 kV static multi-metal anode source (Cu, Mo, Ag, and W) with a minimum focal spot size of 3 μm and a PerkinElmer 2000 × 2000 pixels 16-bit amorphous silicon flat panel detector.

The scanning was performed with the copper target using a voltage of 75 kV and a current of 130 μA. The acquisition software was Nikon Metrology proprietary software InspectX (version XT 2.2 service pack 5.5). The data acquisition was carried out with an exposure time of 2000 ms, and no filtration. The number of projections was set to 3142 and the number of frames per projection was 1, resulting in an acquisition time of 1 hour and 45 minutes. The correction images were acquired by averaging 128 frames using the exact X-ray conditions as used for the acquisition.
The reconstruction was performed with Nikon Metrology proprietary software CT-Pro. The beam hardening correction setting was set to 1 (out of a range of 1–6) whereas the noise reduction setting was set to 2 (out of a range of 1–6) and interpolation was used. The entire volume (2000 × 2000 × 2000 voxels) was reconstructed at full resolution with a voxel size of 17.3 μm along the x, y, and z directions. The resulting reconstruction time was 30 minutes. In order to visualise and process the data, the data set had to be reduced. The data were loaded into Volume Graphics proprietary software VGStudio MAX®, and then converted from 32 bits to 8 bits and the grey scale was remapped from [0,150] to [0,255]. An example of reconstructed 2D slice is given in Figure 2.

![Figure 2: Reconstructed 2D slice and corresponding line profiles.](image)

The visualisation software employed was Visualisation Science Group proprietary software Avizo® Fire version 7.0.1. The segmentation strategy adopted was to first segment the outside of the specimen using the magic wand tool for grey values in the range [0,60]. The selection was then inverted and applied to a material labelled “Matrix”. Within the Matrix selection, a threshold was applied to segment the binder yarns and the voids. The respective threshold intervals were [150,255] and [0,60], and the selections were applied to materials labelled “Yarns” and “Voids”. An example of the segmentation obtained is given in Figure 3.

![Figure 3: Examples of segmentation: matrix (blue), binder yarns (yellow), and pores (red) (10 mm scale bar).](image)

Our approach to porosity distribution measurements in 3D is focussed on the position of the voids relative to both the edges of the panel and the yarns. The void-to-yarn distance and the void-to-edge distance can be obtained from Avizo® by combining the distance maps (respectively yarn distance map and matrix distance map) with the thresholded voids as Figure 4 shows. As a result, the distance of every voxel segmented as void to the closest yarn and the closest edge can be measured over the entire 3D volume (small distances are dark, large distances are light in Figure 4e and f).
Figure 4: Workflow to obtain the void-to-yarn and void-to-edge distance measurements on a virtual slice.

### 2.2.2 Statistical analysis

A statistical analysis has been performed in MATLAB® using the `kstest2` function. This function performs a two-sample Kolmogorov-Smirnov test to compare the distributions of the values in the two input data vectors [13]. The null hypothesis is that the two vectors are from the same continuous distribution. The alternative hypothesis is that they are from different continuous distributions. The significance level $\alpha$ was chosen to be 0.05. Therefore, the two-sample Kolmogorov-Smirnov test result is 1 if the test rejects the null hypothesis at the 5% significance level, 0 otherwise.

### 3 X-ray CT Results

X-ray computed tomography allows a full non-destructive characterisation of the porosity contained within the composite specimens. The quantification performed on the voids for all specimens is summarised in Table 1.

<table>
<thead>
<tr>
<th>Specimen label</th>
<th>Minimum mm</th>
<th>Maximum mm</th>
<th>Mean mm</th>
<th>Standard deviation mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>L-1</td>
<td>0.027</td>
<td>0.398</td>
<td>0.078</td>
<td>0.055</td>
</tr>
<tr>
<td>L-2</td>
<td>0.027</td>
<td>0.404</td>
<td>0.075</td>
<td>0.050</td>
</tr>
<tr>
<td>L-3</td>
<td>0.027</td>
<td>0.290</td>
<td>0.073</td>
<td>0.049</td>
</tr>
<tr>
<td>H-1</td>
<td>0.027</td>
<td>0.406</td>
<td>0.078</td>
<td>0.055</td>
</tr>
<tr>
<td>H-2</td>
<td>0.027</td>
<td>0.458</td>
<td>0.074</td>
<td>0.051</td>
</tr>
<tr>
<td>H-3</td>
<td>0.027</td>
<td>0.419</td>
<td>0.084</td>
<td>0.059</td>
</tr>
</tbody>
</table>

The summary of the void equivalent diameters indicates that the void dimensions are similar for both panels (L and H). For all specimens, the minimum equivalent diameter is just below 0.03 mm and limited by the resolution of the X-ray CT analysis. The values of the maximum equivalent diameter are around 0.45 mm whereas the mean void diameter ranges from 0.075 mm up to 0.084 mm. The standard deviation values are on the same order of magnitude as the mean values which indicates that the equivalent diameters are relatively spread out over the diameter range.

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1 The equivalent diameter is the diameter the void would have if it was perfectly spherical.
3.1 Sample L (low viscosity resin)

Figure 5 summarises the void equivalent diameter distribution as well as the void-to-edge and void-to-yarn distance distributions. From the equivalent diameter distribution (5a), no clear differences can be seen between the three specimens. The two-sample Kolmogorov-Smirnov tests performed between the three equivalent diameter distributions suggests that the data are from the same continuous distribution.

The large peak in the void-to-yarn distance suggests that the voids are predominantly centred at a distance of around 0.12 mm from the yarns; this is also reflected in the evenly spaced maxima in the void-to-edge distance which reflect the regular positions of the yarn within the sample.

3.2 Sample H (high viscosity resin)

Figure 6 shows the void equivalent diameter distribution as well as the void-to-edge and void-to-yarn distance distributions. From the equivalent diameter distribution (6a), no significant differences can be seen between the three specimens. The two-sample Kolmogorov-Smirnov tests performed between the three equivalent diameter distributions reveals that the data are from the same continuous distribution.
As for the low viscosity resin samples the void-to-yarn distance plots show a single sharp and intense peak at a distance close to 0.12 mm which indicates that the voids are mainly located around the yarns. The maxima in the void-to-edge distance plots in Figure 6b are regularly spaced, but in contrast to those for the low viscosity resin, the intensities decrease with decreasing distance from the panel edge, indicating a higher distribution of voids close to the centre of the panel.

### 3.3 Sample comparison

The distributions obtained for the three specimens of each sample have been combined to compare void size, distance to edge and distance to yarn distributions between samples L and H (Figure 7 and Figure 8 respectively). Although no major differences are evident from the void equivalent diameter distributions (Figure 7a), they are sufficiently different that the two-sample Kolmogorov-Smirnov test rejects the hypotheses that the two void distributions are from the same distribution.

![Figure 7: Statistical distribution for L and H samples.](image)

The same result was obtained for the void-to-yarn distance distributions (Figure 7b) even though similar sharp peaks around 0.12 mm are obtained for both specimens. The differences between the two specimens are emphasised when plotting the corresponding cumulative distributions functions as shown in Figure 8.

![Figure 8: Cumulative distribution functions of the void-to-edge and void-to-yarn distances.](image)
The cumulative distribution functions of the void-to-yarn distance demonstrate that the voids in specimen L are located significantly closer to the yarns than for specimen H. For example, 90% of the voids are located within 0.25 mm from the yarns in sample L whereas the same percentage of voids is located within 0.50 mm from the yarns in sample H. Regarding the void to edge distances, the cumulative distributions functions demonstrate that the voids are more uniformly distributed throughout the thickness of the panels for specimen L than for specimen H: 50% of the voids lie within the middle 2.5 mm of panel L whereas 50% of the voids are concentrated within the central 0.5 mm of panel H.

4 Discussion

X-ray computed tomography allows a full non-destructive characterisation of the porosity contained within the composite specimens. The summary of the void equivalent diameters (Table 1) shows that the characteristics of the voids (minimum, maximum, and mean values) are consistent between panel L and panel H. The two-sample Kolmogorov-Smirnov tests performed between each of the three equivalent diameter distributions reveal that the 3 samples within each type are consistent. However, the Kolmogorov-Smirnov test demonstrates that the void equivalent diameter distributions from L and H are statistically different with a 95% confidence level.

The new approach to 3D void distribution characterisation, namely the measurements of the void-to-edge distance and void-to-yarn distance distributions, has given new quantitative insights on the resin film infusion process. First it demonstrated that for both the high and low viscosity systems, most of the voids were located around the binder yarns. An example of the void-to-yarn relationship is given in Figure 9.

Figure 9: 3D rendering of voids (red) around binder yarns (yellow).

It is noteworthy that 90% of the voids were located within 0.2 mm of the yarns for the low viscosity system (panel L) whereas this distance was increased to 0.5 mm for the high viscosity system (panel H). The increase in viscosity limits resin flow during manufacturing resulting in larger gaps around the binder yarns. Secondly, it has been shown that the resin viscosity has a significant impact on the void distribution through the panels. For the low viscosity panel, although located around the binder yarns, the voids are fairly uniformly distributed throughout the thickness of the panel (50% of the voids are in the central half (2.5 mm) of panel L), whereas for the high viscosity samples, the voids are concentrated very close to the middle of the panel (50% of the voids lie within the middle 10% (0.5 mm) of panel H).
5 Conclusion

This study has presented a new approach to void characterisation in 3 dimensions applied to polymer-fibre composites. The standard quantification of voids, with measurements such as minimum, maximum and mean equivalent diameter values, did not yield significant differences between the two panels. By contrast, both qualitative and quantitative results could be obtained from the void-to-yarn and void-to-edge distance distributions. For the high viscosity resin, the resulting panel showed a much higher void distribution towards the centre of the panel. This suggests that either the resin stays towards the centre due to its inability to flow through the layers or the resin itself contains voids which are retained in the centre whilst the resin flows. In the first case it suggests that higher processing temperatures are needed during RFI to reduce the resin viscosity so that the flow is improved. In the latter case, the degassing of the resin needs to be further optimised.

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References