EFFECT OF VOIDS ON INTERLAMINAR BEHAVIOUR OF CARBON/EPOXY COMPOSITES

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Abstract

Porosity is a common manufacturing defect in composite materials. It can be induced by trapped air during lay-up, ineffective debulk or an inadequate autoclave curing cycle. It is almost impossible to completely eliminate voids during manufacturing. They can lead to development of other defects such as delamination and have a detrimental effect on matrix-dominated properties of composites. Many researchers have investigated the effect of void content on mechanical properties of composites, but size, shape and location are important parameters often not characterised. In this work destructive and non-destructive techniques were used to identify void content and their parameters. Short beam shear tests (SBS) were performed to measure interlaminar shear strength (ILSS). As expected ILSS reduced with increasing void content. However it was shown that location of the voids in a composite also influences the mechanical performance. Thus maximum void volume fraction in a ply showed the best correlation with the knockdown of ILSS. For better understating of behavior of composites with high level of porosity the void morphology was analysed.

1. Introduction

Manufacturing of composites has the potential to introduce different defects, such as porosity, fibre and ply misalignment, fibre waviness, foreign objects, partially-cured matrix material, resin rich areas and delamination. Arguably the most crucial defects are voids, as they are difficult to eliminate during manufacturing, can induce other defects, such as delamination, and have a detrimental effect on the mechanical properties.

The influence of porosity (void content) on the mechanical properties of composite materials has been widely studied [1]–[10], and has shown that porosity primarily influences the matrix-dominated properties, such as interlaminar shear strength, bending properties, compressive strength and modulus, fatigue and fracture toughness. Since the majority of voids are located between the plies, at the interface [2], [5], a dominant effect on the interlaminar shear strength (ILSS) can be expected. Poor ILSS leads to through-thickness failure which is a major concern for composite structures.

There are a number of theories that describe the relationship between mechanical properties and void content under various loading conditions [3], [11]. However these models are not definitive as they need to consider, for example, different types of material, different stacking sequences and processing parameters that could affect the distribution, location, shape and size of voids. Currently, no model is able to satisfactorily consider all these variables and produce an accurate assessment of the effects upon the properties of the material.

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X-ray computed tomography (CT) is a promising non-destructive technique that can give information about pore location, size and shape in three dimensions [12]–[15]. However the technique is only able to accommodate small sample sizes, so that high resolution of the image can be feasibly obtained. Additionally, care is required during post-processing to identify voids in CT-scan images, as it is difficult to establish the exact void size and shape from these images. Usually, validation of the images is performed by comparison to optical microscope images of slices of the sample, which give high-fidelity measurements of the individual voids captured during CT-imaging. However, this optical technique is restricted to 2D and requires samples to be cut in multiple sections, which leads to a loss of some information; hence it is necessary to adjust the X-ray CT post-processing in order to get accurate quantitative analysis of the voids data.

The aim of this work is develop methodology to assess and characterise porosity defects, with further correlatation of particular features of the voids to the interlaminar behavior of the composite.

2. Experimental procedure

2.1. Material and specimen preparation

The material used is a unidirectional carbon/epoxy (Hexcel™ IM7/8552). Four different panels, with different void content were manufactured. To introduce voids the panels were made using a compaction method with a uniform pressure of 0.3 MPa, which is lower than is recommended by the manufacturer. Hot plates were heated to a temperature of 30 °C (batch 1) and 90 °C (batch 2); further curing of the panels involved post-curing in a hot air oven at 100 °C for 17 hours. One reference panel was manufactured by the compaction method using the cure cycle recommended by the manufacturer (batch 3) and a second reference was cured in the autoclave using the same cure cycle (batch 4). The compaction method involved mounting the hot plates in an Instron™ testing machine and applying a pressure at a constant controlled temperature. The experimental setup is shown in Figure 1. The composite panel consisted of a symmetric cross-ply with a total of 16 plies, i.e. $(0/90)_{4S}$. All specimens were cut to the dimensions of 20 mm length, 8 mm width and 2 mm thickess.

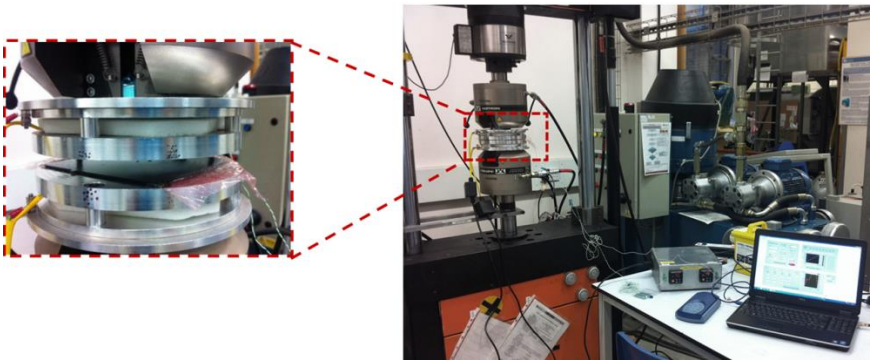

Figure 1. Experimental set-up for the out-of-autoclave curing method with controlled temperature and pressure

2.2 Characterisation of voids

Different techniques were chosen to calculate the porosity content in the specimens. Firstly, the gravimetric method, which is a non-destructive and non-imaging method based on the measurement of density following Archimedes's principle. This test was performed using a modified procedure of ASTM D792, where the weight of the water container was monitored instead of the tension in the

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string to which the sample is attached. Material data was taken from the material certificate for the Hexcel™ IM7/8552 pre-preg system, which states a mass fraction of resin of 33%, density of fibres 1.77 $g \cdot cm^{-3}$ and density of resin 1.3 $g \cdot cm^{-3}$.

This technique does not provide detailed information such as void size, location or distribution so an imaging technique is essential for further characterisation. Scanning Electron Microscopy (SEM) and Optical Microscopy (OM) were also used for void analysis, however these methods are limited to scanning along 2D 'cuts' of the sample, and require a destructive (and time-consuming) procedure to progressively slice as new scans are taken. Thus microscopy was used primarily for detailed analyses of selected portions of the specimens and for validation of the other characterisation methods as described below.

X-ray Micro-Computed Tomography scanning (μCT-scanning) was performed before and after the SBS tests in order to provide information on the shape, size and distribution of voids in three dimensions (3D). A Nikon™ XTH225ST CT-scanner was used in this work. A source voltage of 65 kV and source current of 309 μA were used with two shots per projection, averaged to reduce scattering noise. A scan resolution (voxel size) of 9.4-11.8 μm was possible due to the small size of the samples. The void content and morphology were visualized and analysed further using post-processing software *VG Studio™ MAX* version 2.2 with a porosity analysis plug-in.

2.3 Short Beam Shear Test (SBS)

The Short Beam Shear (SBS) test consists of a three-point bending test on a specimen of small span to thickness ratio. The SBS rig was installed on a Shimadzu testing machine, equipped with a 10 kN load cell, as shown on Figure 2. The loading pin is a 6 mm diameter cylinder in accordance with ASTM D2344. The span length is 10 mm, thus the span-to-thickness ratio is 5 as recommended. The crosshead speed was set to 1.0 $mm \cdot min^{-1}$ in accordance with the standard. The tests were stopped automatically by the testing machine at a load drop-off of 30%.

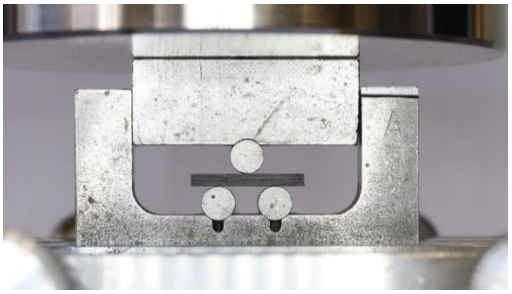

Figure 2. Short beam shear experimental set-up

The out-of-autoclave manufacturing method for controlled void content used a different cure cycle from that recommended by the manufacturer. Hence prior to test cure degree of the samples was assessed. This was done using Differential Scanning Calorimetry (DSC), which involves measuring the heat flow from a sample which is affected by chemical reactions and phase changes.

3. Results and discussion

3.1. Characterisation of porosity

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In this work**,** two non-destructive techiques (i.e. gravimetric method and μCT-scanning) were used to measure the void percentage within samples. The gravimetric test is a much quicker, easier and inexpensive method; however, a number of technical challenges were identified that could reduce the accuracy of the method, such as the small size of the samples (relative to the recommended dimensions), air bubbles on the surface of the specimens and the reliance on accurate knowledge of the density and fibre volume fractions**.**

μCT-scanning requires small samples to provide a good resolution of the scanning. The quality of the CT-scanning process is dependant partly on the aspect ratio of the specimen, so it was improved by creating an assembly of four stacked specimens scanned simultaneously; individual specimens could be 'separated' digitally during post-processing. μCT-scanning (Figure 3) showed needle shape voids with various volume through the thickness of the laminate.

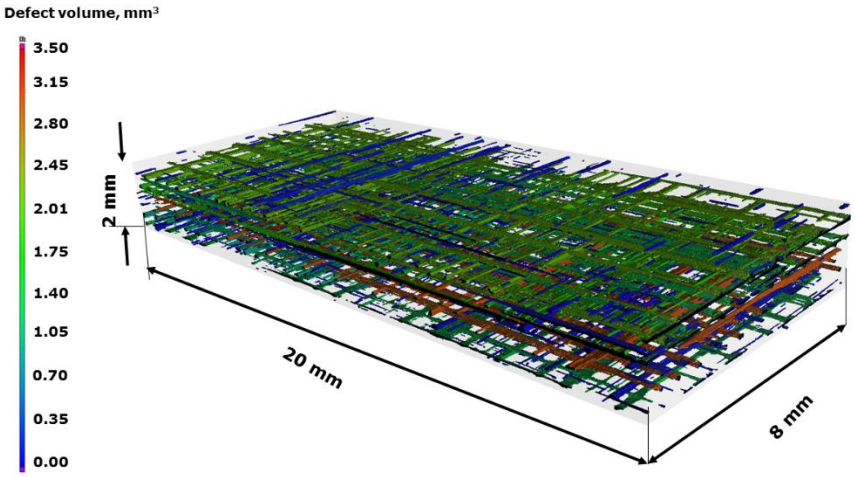

Figure 3. Void distribution as detected using μCT-scanning

The two methods of determining void content are compared in Table 1. The differences between the gravimetric method and the μCT-scanning are significant. As mentioned previously the presence of air bubbles around small SBS specimens is believed to result in an overestimation of the void content. Since the gravimetric method had these limitations and cannot provide geometrical data about voids, the μCT-scanning technique was chosen as the main experimental tool in the investigation of voids.

Table 1. Comparation of methods by void content

	Void content, %			
	Batch 1		Batch 2	
	Gravimetry	μCT-scanning	Gravimetry	μCT-scanning
Average (±SD)	9.17 (±0.50)	4.87 (±0.68)	6.85 (±0.50)	1.82 (±0.36)

Optical microscopy was used to analyse the morphology of voids in detail and to validate the μCT-scanning technique. Figure 4 shows a series of micrographs of several voids within a single SBS specimen. Although different shapes and sizes are present, the majority of the voids are located *at the*

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interface between plies or appear *within plies* and are usually elongated in a direction parallel to the fibres of the plies.

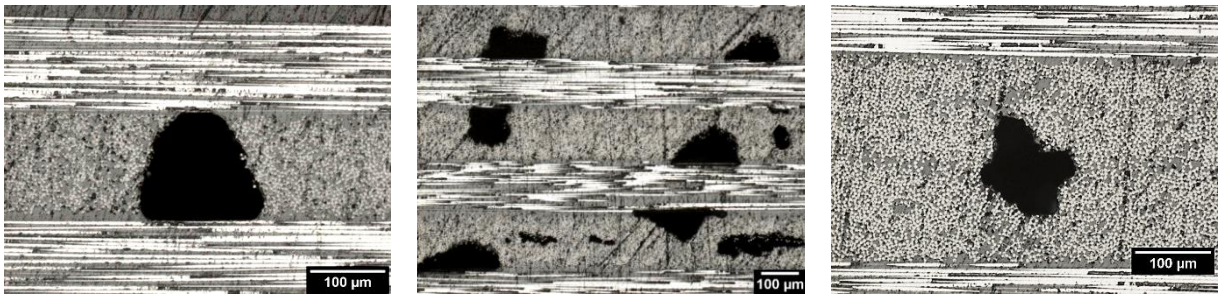

Figure 4. Optical micrographs of the sample 24, batch 1 (V_v=5.86%), showing variety of voids shape, size and position within a sample.

In order to validate the µCT-scanning technique for void characterisation, high-magnification optical microscopy images were compared with µCT-scans for the same location within the specimen as shown on the Figure 5. Excellent correlation is observed for void analysis both qualitatively (distribution and shape) and quantitatively (dimensions). This indicates that µCT-scanning is an effective and accurate method for analysis of void morphology and location. The small differences observed in the extracted dimensions are attributed to the challenges in accurately defining the boundaries of the voids, particularly for the µCT-scans.

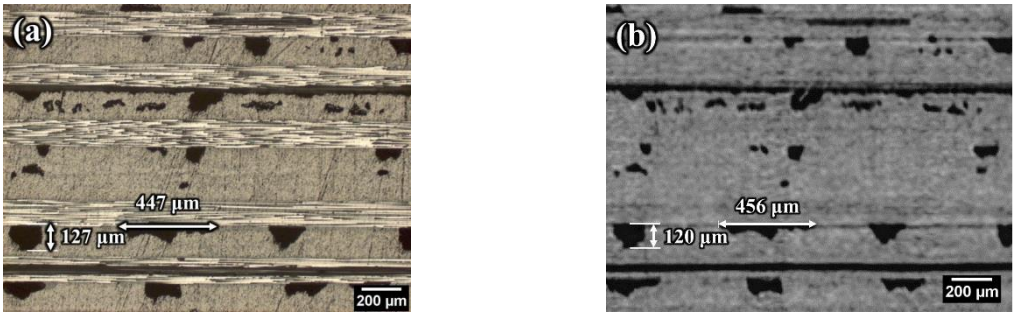

Figure 5. Comparison analysis of: (a) microscopy; (b) CT-scanning

To investigate the influence of voids on crack initiation and propagation, tested samples were also characterised by SEM as shown in Figure 6. These images suggest that voids promote the development of the large number of small cracks in the material, and that propagation involves the coalescence of some of these cracks. Moreover, there is some evidence that the shape of the voids may influence the initiation and propagation of cracks. For example, voids of triangular cross-section appear to show cracks at the vertices where the radius of curvature is smaller – and the stress concentration should be higher. However, crack initiation is difficult to track using *post mortem* techniques and some of these observations are still speculative. In order to characterise true *damage initiation* from voids, the author suggests the use of *in situ* µCT-scanning conducted *during* mechanical tests.

3.2 Short beam shear test results

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Twelve samples from batch 1 and batch 2 have been tested and showed failure via multiple crack initiation events. This was confirmed by the small load drop in the recorded load-displacement curves, as well as an audible cracking noise during test. This behaviour could be explained by the presence of numerous small voids which act as cracks starters and coalescence points.

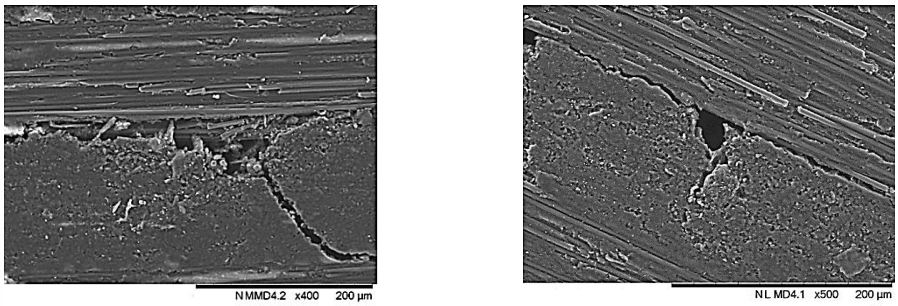

Figure 6. Scanning electron micrograph of tested samples, showing cracks emanating from (or propagating through) voids

As expected, the ILSS decreases with an increasing void content (Figure 7a). More precisely, a 20% decrease in ILSS is observed between specimens with void contents (by volume) of 1.5% and 6%. Figure 10 shows that a reasonable correlation (r=0.858) is found for a linear relationship between the interlaminar shear strength and average void volume fraction.

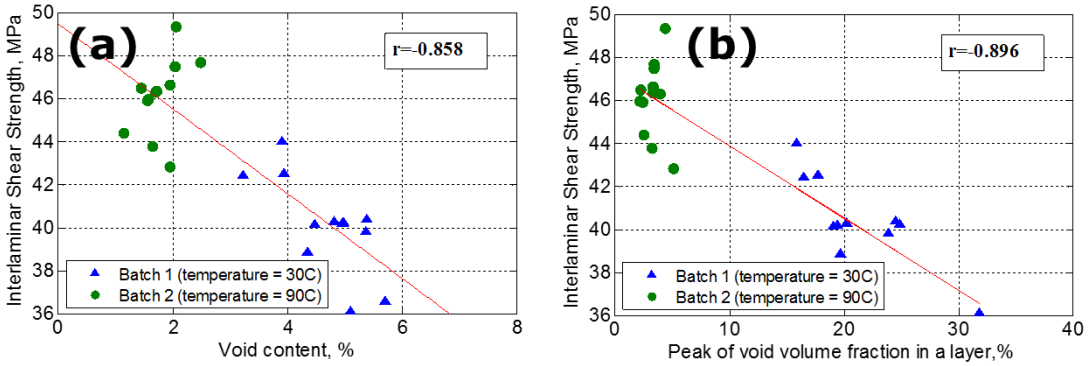

Figure 7. Intelaminar shear strength of the sample vs (a) average void content; (b) peak of void volume content in a layer

However, the 'ultimate' ILSS depends on the combination of void shape, size and location, and meaningful results could only be obtained at this stage by averaging over a number of samples. In an effort to capture more detailed data on the influence of porosity, the ILSS is plotted against the peak void content among all layers in the specimens in Figure 7b. The results showed better correlation in comparison with the average void content in the whole sample (Figure 7a), and a slightly better 'clustering' of points for the two separate batches tested (1-5% for batch 2 and 16-30% for batch 1). It is expected that analyses that take into account more detailed spatial information will improve further the correlation of the results. For example, the stress distribution in the SBS specimen is not homogeneous – it follows a parabolic relation through the thickness and is also affected by contact stresses under the loading pins – so the precise location of each void will also determine its criticality.
Further analyses were carried out based on different parameters. Figure 8a shows the ILSS plotted against the 'maximum dimension' of the largest void in the specimen. It is noticeable that within batch

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2 the maximum dimension varies from 10 to 17 mm, whilst for batch 1 the spread is very small around a mean value of 19 mm. Since the voids are long and thin, the maximum dimension is not always a very meaningful parameter, so maximum *void volume* might have a more significant effect on the failure properties. In order to facilitate visualisation, the volume can be stated as an 'effective radius', i.e. the radius of a sphere with the same volume given by:

$$r_{eff} = \sqrt[3]{3V/4\pi} \quad (1)$$

where V is the volume of the void. Figure 8b shows the ILSS plotted against the effective radii found in each specimen. This measure provided a slightly higher correlation with a linear fit (r=0.863) which suggests that this is a better morphological characteristic to be used when assessing the influence of voids on the interlaminar shear strength.

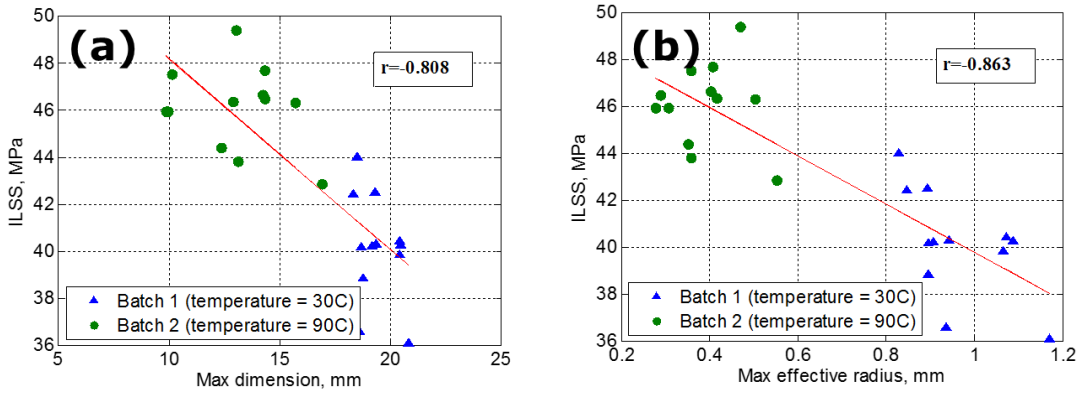

Figure 8. Effect of (a) maximum dimension and (b) maximum effective radius of the void on ILSS

4. Conclusions

In this work, an out-of-autoclave curing technique using hot plates installed in a universal testing machine successufully produced flat cross-ply laminates with consistent levels of porosity. By controlling the parameters of this process – i.e. pressure and temperature – it is possible to control the porosity content in the composite very accurately. However, this compaction method resulted in different temperature profiles from the ones recommended by the manufacturer, and the degree of cure in the samples was at times excessively low. Hence the process must be improved by conducting a 'post-cure' stage in a hot air oven. The μCT-scanning technique was used to characterise voids in composites, providing full geometrical information in 3D about these defects within a laminate. A comparison with high-resolution optical microscopy was used to validate this method in the qualitative and quantitative assessment of void size, morphology and distribution. Voids are found to be elongated along the fibre direction and of ellipsoidal/spherical shape for this particular material (Hexcel™ IM7/8552) in this particular layup, i.e. $(0/90)_{4S}$. However, it is known that void sizes and shapes are affected by several factors (including fibre architecture, matrix material, stacking sequences etc.). In order to gain a deeper understanding of void formation in composites, further tests must be carried out on different material systems. The influence of different void characteristics on the interlaminar shear strength (ILSS) was investigated. As expected, the strength is reduced with an increase in porosity content. However, it was shown that the location of these voids within the laminate also influences its mechanical properties. The *maximum void volume fraction in a ply* was found to provide the best correlation with the knockdown in ILSS, performing considerably better than the *average void content* which is the current industry standard. To better understand the behaviour of composites with high levels of porosity, and also to collect appropriate information for future modelling, the void morphology was analysed; initial results show a strong correlation with the *effective radius*. *Post mortem* examinations using μCT-scanning and SEM suggest that matrix cracks tend to propagate from void to void, altering their direction several times along the crack path, and

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also appear to be influenced by void shape. In addition, multiple cracks were found to initiate from voids depending on their position and size. However, the effects of void morphology and location in the failure mechanisms of composites are not yet conclusive. Tests with *in situ* x-ray scanning and detailed numerical modelling will be required for a full understanding of this complex material behaviour.

Acknowledgments

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