UNDERSTANDING THE EFFECT OF VOID MORPHOLOGY AND CHARACTERISTICS ON LAMINATE MECHANICAL PROPERTIES

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ABSTRACT

Due to the nature of composite materials, it is almost impossible to manufacture defect-free parts. The presence of micro-defects, such as porosity, is commonly observed, and it is been shown that porosity has a detrimental effect on the matrix-dominated mechanical properties. Most researchers have attempted to evaluate the behaviour of composites with defects by correlation of the average void content to the particular mechanical property. However different materials, stacking sequences and processing parameters can affect the distribution and morphology of the pores. It is crucial to understand the variations in void morphology, such as the pore size, shape or distribution, and how these parameters affect the progressive failure behaviour of composite structures.

In this work, two carbon/epoxy toughened prepreg systems were investigated, namely the Hexcel® IM7/and the Hexcel® IMA/M21. To introduce inter-ply voids, cross-ply laminates were manufactured using a novel controlled heat and pressure curing process.

To characterise the voids, micro X-ray Computed Tomography (CT) was used, giving high resolution information about pore size, location and shape in three dimensions. This information was used in this study to develop a methodology to characterise the effects of porosity on the matrix-dominated material properties.

1 INTRODUCTION

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

The influence of average 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 most voids are located at the interfaces between plies [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.

Micro X-ray computed tomography (CT) scanning 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 produce high resolution images of relatively small samples. 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 microscopy of slices of the sample, which gives high-fidelity measurements of the individual voids captured during CT-imaging. However, this optical technique is restricted to 2D scans and requires samples to be cut along a limited number of sections, which leads to the loss of some information. Hence it is necessary to adjust the X-ray CT post-processing in order to get accurate quantitative analysis of individual void data.

Based on the challenges described above, the aims of this work are:

- to develop an out-of-autoclave method to produce panels with controlled void content;
- to investigate the effects of different material systems on void morphology;
- to understand the effect of void morphology on the interlaminar shear strength of laminates.

2 EXPERIMENTAL PROCEDURE

2.1 Materials and specimen preparation

In this work, two carbon/epoxy toughened prepreg systems, developed by Hexcel, were investigated: namely the Hexcel® IM7/8552, with a cured ply thickness (CPT) of 0.125 mm, and the Hexcel® IMA/M21 with a CPT of 0.184 mm. In the IM7/8552 carbon fibre/epoxy system, a thermoplastic toughening phase is dispersed within plies, throughout the bulk of the resin, whilst in the IMA/M21 system an extra layer of thermoplastic particles is formed as a distinct 'interlayer' between the plies during processing.

One of the challenges in this research work is to be able to manufacture panels with deliberately (but controllably) high void content as well as preferably to get porosity distributed as uniformly as possible. To introduce inter-ply voids, panels were manufactured using a novel controlled temperature and pressure curing method. This involved compaction (consolidation) of the laminates using heated plates, followed by curing in the oven. For this, the heater plates are heated up to the temperature as shown in Table 1, and uniform pressure of 3 bars is applied to the laminates. To reach the appropriate cure degree, composite panels were cured in the oven. The set- up of the experiment is shown in Figure 1.

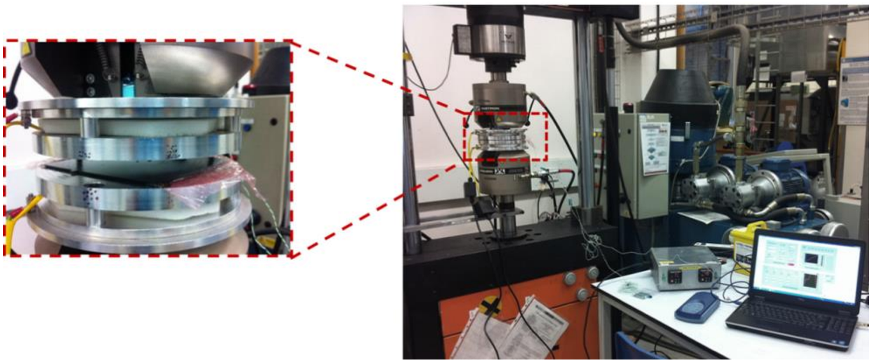


Figure 1: Experimental set-up for manufacturing panels with controlled void content.

	Material	Consolidation		Curing in the oven	
		Temperature, °C	Pressure, MPa	Temperature, °C	Time, h
Batch 1	IM7/8552	30	0.3	180	5
Batch 2	IM7/8552	90	0.3	180	5
Batch 4	IM7/8552	120	0.3	180	5
Batch 1	IMA/M21	30	0.3	180	3
Batch 2	IMA/M21	90	0.3	180	3
Batch 4	IMA/M21	120	0.3	180	3

Table 1: Manufacturing process of the laminates

Two plates measuring 120 mm × 120 mm were manufactured using the proposed method for each of the material systems. Each plate of IM7/8552 consisted of cross-plies with a total of 19 plies; whilst for IMA/M21 the total number of plies was 11. Reference plates (batch 3) were made using the manufacturers recommended autoclave curing cycle.

After manufacturing, 10 mm wide samples were cut from the panels using a diamond wheel. Variation of the sample thicknesses was found, especially for the IMA/M21 material system which had a range from 2 to 3 mm. For IM7/8552, the thickness of the samples was more consistent and ranged from 2.3 to 2.6 mm. The length of the samples was chosen based on the span-to-thickness ratio recommended for short beam shear tests; in this work, a value ~4.5 was chosen.

2.3 Void characterisation

Usually voids in laminates are characterised by *void volume fraction*, sometimes referred to as *void content*. Commonly used methods to obtain this information include the gravimetric method (density measurement), ultrasound attenuation and microscopy. The first two methods cannot provide geometric information about individual voids, thus imaging techniques are required for void characterisation. Although microscopy is used for this purpose, it is limited to scanning '2D slices' of the sample, and is a destructive and time-consuming procedure.

Hence, for this research, micro X-ray CT scanning was used as a primary tool for void characterisation. Each sample was scanned prior to testing in order to obtain all necessary information. A Nikon™ XTH225ST CT-scanner was used in this work. A source voltage of 50 kV and source current of 136 μA were used with two images per projection, averaged to reduce the scattering noise. A scan resolution (voxel size) of 12.6-13 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.

2.4 Mechanical testing

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 in Figure 2. The loading pin is a 6 mm diameter cylinder, in accordance with ASTM D2344. The span-to-thickness ratio was 4.5, as recommended. The crosshead speed was set to 0.5 mm·min^{-1} in accordance with the standard. The tests were stopped automatically by the testing machine at a load drop-off of 30%. The interlaminar shear strength of the composite was then obtained by assuming a parabolic through-thickness shear distribution, i.e.:

$$\tau_{\mathrm{SBS}} = \frac{3\,P_{\max}}{4\,w\,t} \tag{1}$$

where $P_{\max}$ is maximum load, obtained during the test, and w and t are the specimen width and thickness respectively.

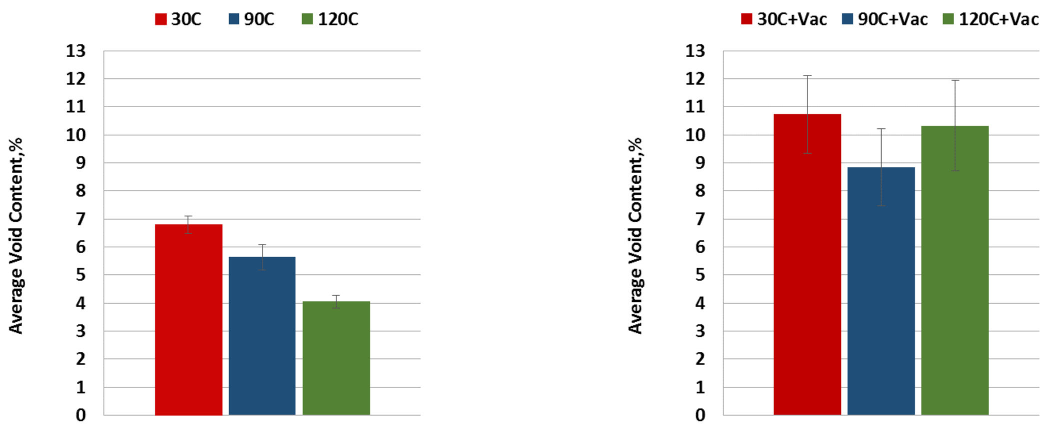
3. RESULTS AND DISCUSSION

3.1 Manufacturing process and average void content of the panel

In this work, a bespoke manufacturing process was developed to produce panels with induced voids. The same procedure was applied to both material systems. The pressure of the heated plates was kept constant for all batches, whilst the temperature of 'debulking' (consolidation) was varied from batch to batch, as shown in Table 1. Further curing was completed free standing in the hot air oven. The effect of temperature during consolidation on the void content was investigated.

The chosen manufacturing process led to an extremely high void content (30-35%) in IMA/M21 laminates. Due to the presence of the toughening elements, the surface of the IMA/M21 prepreg is rough and less tacky, thus it can increase the amount of entrapped air during the layup process. Moreover, during cure in the oven, the void pressure increases as the temperature rises, which leads to further void formation and expansion. When the viscosity increases or gelation occurs, voids are locked into the resin matrix. However, there is no pressure applied during curing, to collapse the voids. Hence it led to an excessive voidage in the composite panels. To mitigate this problem, it was decided that a vacuum pressure would be applied during the oven cure. This allowed the average void content to be reduced within the samples to a comparable level (8-11%).

The temperature during compaction showed a strong effect on the void content for the IM7/8552 material system. Figure 2a shows the reduction of the average void content with increasing of the heater plate temperature. However, the porosity in the IMA/M21 panels did not depend on the temperature during debulk (Figure 2b). It can be suggested that the pressure might have a stronger effect on the void content for this material system due to the presence of the toughening interlayer.



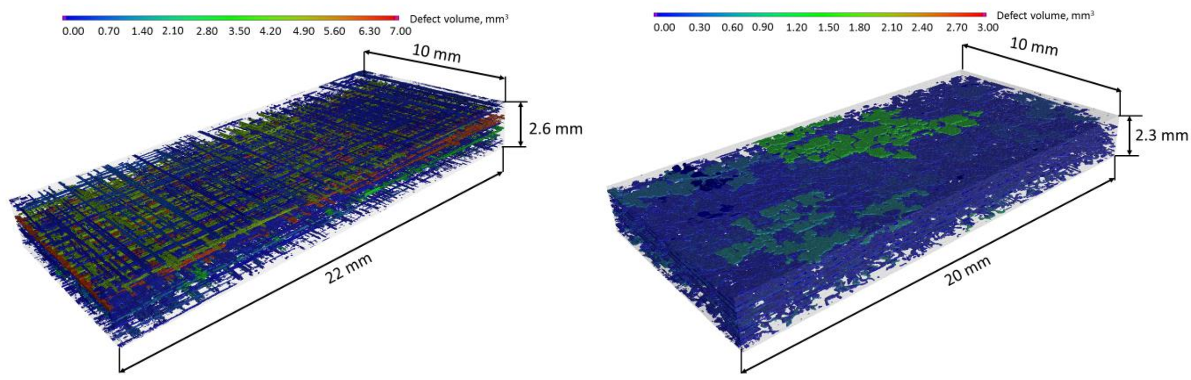
(a) IM7/8552 (b) IMA/M21

Figure 2: Consolidation temperature effect on the average void content for (a) IM7/8552 and (b) IMA/M21.

3.2 Void morphology

Different types of materials, different stacking sequences and processing parameters could affect the distribution, location, shape and size of voids. Moreover, two types of voids that can be found in the samples: *intra-ply* voids, that are located within the plies of the laminates, and *inter-ply* voids, that are based on the interface in between the plies.

For both material systems, intra-ply voids are needle- shaped and elongated in the fibre direction. However, inter-ply voids show different characteristics: for IM7/8552 they have the same shape as intra-ply voids, whilst for IMA/M21 the voids are more circular. This is clearly seen in CT-scans of both material systems in Figure 3.



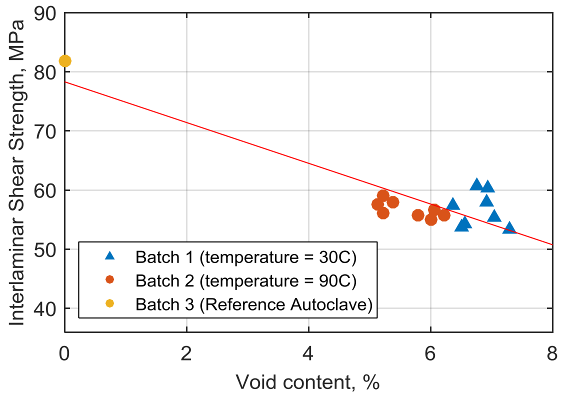
(a) IM7/8552 (b) IMA/M21

Figure 3: Void morphology for (a) IM7/8552 and (b) IMA/M21.

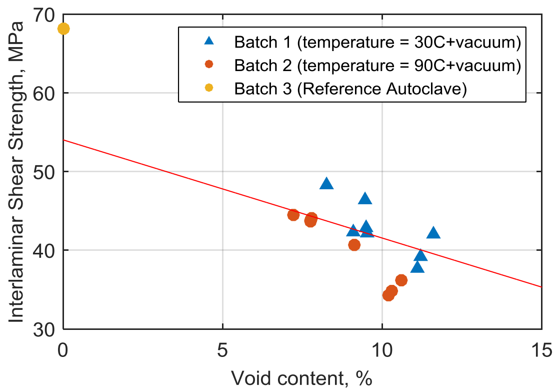
3.3 Void features analysis

Eight samples of each batch (except batch 4) have been tested using the SBS configuration, after they underwent X-ray CT-scanning. It was observed that the specimens failed via multiple crack initiation events; as opposed to the usual sudden failure behaviour of pristine laminates. This was confirmed by the small load drop in the recorded load-displacement curves, as well as an audible cracking noise during the tests.

As expected, the ILSS decreases with an increasing average void content as shown in Figure 4. However, the results did not show a strong linear correlation between the average void content and ILSS, neither for the reference samples nor samples with introduced porosity. Moreover, significant scatter was observed for batches 1 and 2 for both material systems. Some of the samples with different void content showed the same ILSS, and conversely samples with the same void content failed at the different ILSS levels. To assess the reasons behind these observations, a void statistical analysis was conducted.



(a) IM7/8552



(b) IMA/M21

Figure 4: Reduction of the ILSS with the increasing of the average void content for (a) IM7/8552 and (b) IMA/M21.

The reduction in the mechanical properties of a laminate with voids depends on the detailed features and parameters of individual voids. These features can be divided into three categories, (i) void size, (ii) void shape and (iii) void location. Void size can be characterised by *volume*, *diameter* or *length* in a particular direction. Void shape can be represented by *aspect ratio*, *compactness*[1] or *sphericity*[2]. The critical locations for voids were assumed to be the regions of maximum stress within samples where failure is most likely to occur.

For the proposed statistical analysis, voids with volume lower than 0.005 mm^3 were neglected, as such small volumes are unlikely to affect the strength of the material considerably. Concerning the critical location, the regions within the specimen with the highest shear stresses (as determined by 3D Finite Element analysis) were chosen. Hence, only the voids that were located in these areas were included in the statistical analysis. Moreover, just the 20 largest voids (by volume) were included in the investigation, as these are the most likely to promote damage initiation.

Detailed analyses were carried out on CT-scans of pairs of specimens which yielded similar ILSS values. The results for one pair of IM7/8552 specimens are shown in Figure 5. It was found that the samples with the highest void content had a small number of voids (4 to 5) with large volume but low sphericity, which means that these large voids were thin and elongated. Samples with lower void content tended to have more spherical voids of almost constant volume. Because these pairs of specimens exhibited similar ILSS, the data suggests that both morphology combinations – few large and elongated voids, versus many smaller and spherical voids – result in the same strength reduction.

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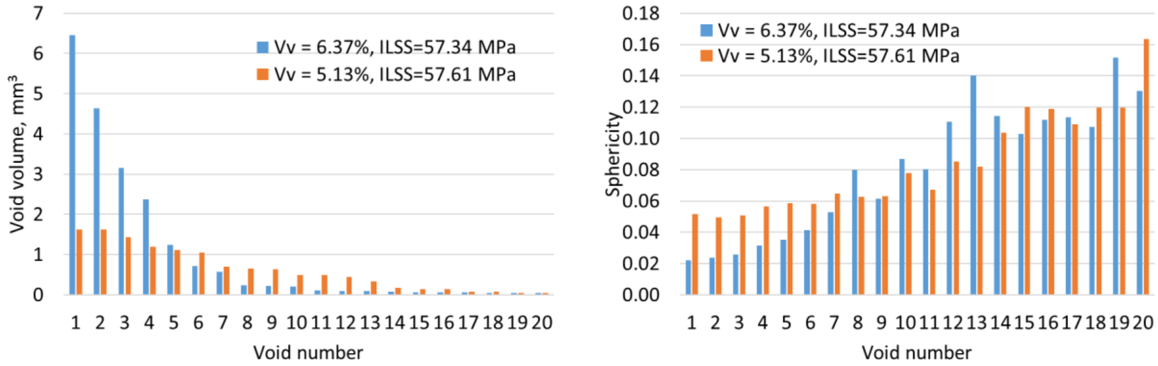
[1] *Compactness* = ratio between surface area and volume.
[2] *Sphericity* = ratio between the surface area of a perfect sphere and the surface area of the void, both with the same volume.

Similar analyses and conclusions were obtained for both material systems, the only difference being that the majority of the voids in the IMA/M21 laminates had in general greater sphericity.



(a) (b)

Figure 5: Comparison of the void volume (a) and sphericity (b) for the 20 largest voids in specimens of both material systems.

4 CONCLUSIONS

In this work two carbon/epoxy toughened prepreg systems with controlled porosity were investigated, namely the Hexcel® IM7/8552 (dispersed toughening phase) and the Hexcel® IMA/M21 (interlayer toughening phase). A proposed out-of-autoclave method was used to manufacture flat cross-ply laminates with consistent levels of porosity. Pressure during compaction was kept constant, and the effect of the temperature was studied. A higher compaction temperature was found to reduce the average void content of the IM7/8552 laminates, however it did not affect the IMA/M21 material to the same extent. The morphology of the intra-ply voids for these two materials was found to be significantly different. The voids were more circular for IMA/M21 and more needle-like (elongated) for IM7/8552. In order to investigate the effects of the void morphology on the interlaminar shear strength, a statistical analysis was used. Three different void characteristics – void size, shape and location - were investigated. The analysis showed that smaller spherical voids with constant volume, centred in highly stressed regions, have a similar effect on the interlaminar shear strength compared to fewer, larger and elongated voids. However, this analysis is not yet conclusive. Further morphology parameter distributions are currently under investigation. Tests with in-situ X-ray scanning and detailed numerical modelling are currently being conducted to fully understand the complex failure mechanisms in laminates containing voids.

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