Fatigue damage development in 3D woven glass and glass/carbon composites

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This thesis is submitted for the degree of Doctor of Philosophy 2017
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Abstract

A number of studies have been conducted on 3D non-crimp orthogonal woven composites, but their industrial application is still in its infancy. 3D woven composites show increased through-thickness strength, reducing delamination damage, which is often a key failure mechanism for composites under various loading conditions, especially fatigue. This work investigates the fatigue performance and damage development in a 3D non-crimp orthogonal woven composite consisting of three weft tow layers, two warp tow layers, and a through-thickness z-binder that interlaces along the warp-direction. While the properties of carbon fibres are generally superior to glass fibres, they are more expensive. Therefore, it is of interest to see if the fatigue performance of a glass fibre 3D orthogonal weave can be improved via selective hybridisation using a small amount of carbon fibres.

Initial work began on a commercial all-glass 3D orthogonal weave called 3D-78, which was produced by 3TEX. It was found that quasi-static tensile mechanical properties were the same for both warp and weft loading directions, but when loaded in tension-tension fatigue, the warp direction had longer fatigue lifetimes than the weft-direction. The crack density was lower in warp-direction specimens as a result of greater micro-delamination growth blunting stress concentrations around the tips of matrix cracks. The micro-delamination damage in warp-direction fatigue specimens showed a shield-like shape (not previously observed), i.e. wider along one side and narrowing to a point on the other side; where delamination was restricted (at the pointed end), fibre fractures occurred in the adjacent warp tow. The pointed portion of the micro-delamination corresponded to proximity to a z-binder crown. Other damage that was common to both loading directions (warp and weft) included: transverse cracks in transverse tow and resin-rich regions, z-binder debonding, and longitudinal tow splitting cracks. No obvious failure sites were noted for weft-direction fatigue loading.

The second material used, 3DMG, was manufactured by the University of Manchester. This material was produced with two different z-binder tensions. The initial z-binder tension (3DMG-T1) resulted in a higher tensile modulus and strength-to-failure, and lower strain-to-failure, for the warp-direction, while the tensile fatigue properties of both directions were similar. Increasing the z-binder tension (3DMG-T2) reduced the tensile modulus and increased the strain-to-failure of the warp-direction, with these properties now similar in both loading directions; the tensile strength for both loading directions remained similar. However, the fatigue performance of the warp-direction was observed to increase with increased z-binder tension, while the weft-direction remained the same. The damage that developed in both materials was similar to the damage in 3D-78, and remained practically the same regardless of z-binder tension, though the energy dissipated per cycle for warp-direction specimens was higher in 3DMG-T1, which corresponds well with the lower number of cycles to failure.
The final material tested was a University of Manchester hybrid 3D non-crimp orthogonal woven composite, termed 3DMHyb; here the glass fibre z-binder was replaced with carbon fibre; the z-binder tension used here was the same as 3DMG-T2. Generally, the quasi-static properties of this hybrid material were similar in both loading directions, with the exception of the tensile modulus which was approximately 10% higher, indicating that the carbon fibre z-binder may influence low strain properties. Additionally, the properties of 3DMHyb remained similar to 3DMG-T2. For fatigue performance, however, the fatigue lifetime to failure appeared to increase by a factor of just over 2 at lower peak stress/initial peak strains for the hybrid warp-direction specimens. Again, the energy dissipation per cycle was lower for specimens that had larger number of cycles to failure, in this case the hybrid specimens. Damage development also remained similar between the 3DMG-T2 and 3DMHyb specimens, indicating that the extension of fatigue life noted in 3DMHyb may be the result of the carbon fibre z-binder suppressing the development of damage mechanisms leading to ultimate failure of the specimens.
Acknowledgements

For helping, guiding, and providing me with valuable advice all the through my PhD, I would like to thank Prof. S. L. Ogin. I would also like to thank Mr. Peter Haynes, and Dr. T. Capell for helping me at various stages with my testing and manufacturing of materials. For the manufacture of material, I want to thank Dr. D. Patel, Dr. V. Koncherry, and Prof. P. Potluri from the University of Manchester.

I would like to thank the Engineering and Physical Sciences Research Council (EPSRC) as well as DSTL for sponsoring my PhD, and allowing me to conduct this research.

Finally, I want to thank my friends and family for supporting me through this period of my life.
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ICCM20, Copenhagen, 2015

**M. C. Poole, S. L. Ogin, P. A. Smith, G. M. Wells, P. Potluri, P. J. Withers,** “Warp and weft direction damage development in the late-stage fatigue life of a 3D non-crimp orthogonal weave composite”
ECCM17, Munich, 2016

S. Topal, L. Baiocchi, A. D. Crocombe, S. L. Ogin, P. Potluri, P. J. Withers, M. Quaresimin, P. A. Smith, **M. C. Poole,** A. E. Bogdanovich, “Late-stage fatigue damage in a 3D orthogonal non-crimp woven composite: An experimental and numerical study
Chapter 1
Introduction

1.1. Background

Although composite materials are not a new technology, it is only within recent times that they have begun to find use in a wide range of applications. Application of composite materials have been far reaching from technical industries such as automotive and aerospace, to use in sport equipment, such as tennis rackets. The interest in composites usually comes from the fact that they are lightweight, whilst still maintain load bearing capabilities similar to metallic materials. The desirable properties of composites are usually specific strength and stiffness, and fatigue resistance among others, depending on the composite system. Design of complex shapes is also possible with composite materials since they can be in a dry fabric form, or a pre-impregnated with resin (pre-pregs) form, readily manufactured into a component with a complex shape (the degree of complexity being dependent on the reinforcement architecture and the manufacturing method). These fabrics and pre-pregs can be used to manufacture near net-shape components that may otherwise require much more machining if materials such as metals were to be used.

However, one critical issue for composite materials is their frequent susceptibility to damage under relatively low loading compared with their strength, stiffness, and strain-to-failure. Unlike metals, polymers and ceramics, composites are usually made up of multiple layers of fibre reinforcement in order to achieve the desired properties for various loading cases. The highest strength of a composite is typically along the fibre axis, while the binder, such as a polymer resin, is the weakest component. Most fibre reinforced composites will have the fibres orientated in more than one direction, none of which is through the thickness of the component. This means that through the thickness, the only material keeping the layers together is the matrix surrounding the fibres. Since this is the weakest component, failure can often occur along the interfaces between layers. This type of damage is known as delamination, and it can develop during many different loading conditions including, but not limited to, impact, tension, flexure, compression, and fatigue.

The best way to limit, or remove completely, the effect of delamination damage is by improving the through-thickness strength (the interlaminar strength). The improvement of through-thickness strength can be achieved by the addition of a through-thickness reinforcement to the structure of the composite. There are many ways that a through-thickness reinforcement can be added to a composite, including weaving (composites with fibres in all three principal directions are generally called 3D
composites), braiding, knitting, pinning, stitching, and tufting. Each of these methods provide various levels of improvement to the through-thickness performance. However, they can have detrimental effects on other mechanical properties, which is the subject of continued study.

Additionally, some 3D composites can be quite expensive to manufacture in comparison to 2D varieties. This results from the need for new manufacturing equipment, or modifications to current equipment, and an increase in preparation time/complexity of manufacture. Therefore, it is of interest to develop cheaper composite materials with properties similar, equal, or better than current materials. In particular, the properties of carbon fibre based composites are generally superior to both glass and aramid fibre composites. A question therefore arises as to whether improvements to the properties of the cheaper glass composites can be made by incorporating small quantities of more costly fibres, like carbon. This process, called hybridisation, is in its infancy with regard to 3D reinforced composites, but could be a method for improving the properties of 3D composites without adding significantly to an already relatively expensive material.

1.2. Project aims

For 3D composites to find wider application in various industries, rigorous analysis and understanding of these materials under various loading conditions need to be undertaken. As many composite applications are in aerospace, automotive or marine areas, where the structure is subject to repeated loading, an important feature to be understood is the fatigue response of 3D reinforced composites. There is a significant body of work in the literature on this topic, but a more comprehensive understanding of the damage accumulation mechanisms leading to failure is required in order to be able to design better 3D reinforcement geometries, and hence more fatigue resistant components.

This thesis aims to characterise a particular 3D woven structure with regards to its fatigue performance and the development of damage during fatigue loading. The aim is to understand the performance and damage that develops in 3D orthogonal woven structures consisting of one fibre type. Selective hybridisation will then be used in an attempt to improve the fatigue performance.

1.3. Dissertation outline

The outline of the dissertation is as follows. In the next chapter (Chapter 2), a wide-ranging review of the mechanical behaviour of 3D reinforced composites will be presented, with particular reference to fatigue damage development. In Chapter 3 the experimental procedures followed throughout this work will be provided. This is followed by Chapter 4 which will provide a detailed evaluation of the quasi-static and fatigue properties of the 3D-78 material produced by 3TEX. Chapter 5 will provide a characterisation of the damage development in the 3D-78 material under quasi-static and fatigue loading. Chapter 6 and 7 will focus on the mechanical properties and damage development in an all-glass 3D orthogonal weave called 3DMG subjected to quasi-static and fatigue loading. After
this Chapter 8 focusses on the mechanical properties and damage development of 3DMHyb, a hybrid 3D orthogonal weave. Finally, the overall conclusions, and some suggestions for future work are presented in Chapter 9.
Chapter 2

Literature Review

2.1. Introduction

The work to be presented in this thesis is related to tensile mechanical properties and damage development of 3D orthogonal woven composites. It is useful to survey the studies already published on 3D composites to put this work into context as well as gain some understanding of these materials when subjected to other loading cases. Therefore, this chapter will aim to review much of the literature published on 3D woven composites. However, 3D woven composite structures can be relatively complex, and as such the first few sections will provide a brief introduction to composites and various composite architectures in order to provide the reader with a basic understanding of the 3D structures used in much of the literature.

2.2. Introduction to composites

According to the American Society for Testing and Materials (ASTM) [1], a composite material is defined as two or more materials that when combined produce properties not possessed by each component individually. In addition, each material remains distinct within the structure. The constituents of a composite are typically a matrix and a reinforcing phase/s. Generally, the matrix phase has less desirable properties than the reinforcement, but helps bond the reinforcement into a solid structure since it often lacks a useful form structurally. This is because the reinforcement will have at least one dimension that is very small, often of the order of a few microns [2]. Combining a matrix and reinforcement produces a structure with properties closer to that of the reinforcement, while not losing useful characteristics obtained from the matrix [3].

Reinforcements tend to be of two major varieties: particulate or fibrous. The geometry of each will have a large effect on the mechanical properties. Fibrous reinforcement is defined by the ratio of its length to cross-section as this is much larger than that of the particulate reinforcement. Fibrous reinforcement can be split further into two sub groups: continuous and discontinuous. Continuous fibre reinforced composites are defined as those where the length of the fibres within the starting material exceed 7.5mm [4]. In a large portion of cases continuous fibres will cover a great percentage length and/or breadth of the material. Discontinuous fibres on the other hand tend to be surrounded by matrix. This can lead to the ends playing a much larger role in the fracture of these composites as the stress and strain fields respond differently to those composites with continuous reinforcement [3].
A matrix can be polymeric, ceramic, or metallic. The most common materials used for fibrous reinforcement include: glass, carbon and some natural fibres such as aramid. However, metals have also been used, as in the case of reinforced concrete. In this project only polymeric composites with continuous fibre reinforcement are of interest. Reinforcement material is limited to glass and carbon.

2.3. Fibre-reinforced composite architectures

Unlike many materials, composites are not generally limited in the possible architectures they can possess; there are an infinite number of possible combinations which can be produced. As such, composites are usually designed with a particular application in mind. For fibre-reinforced composites the orientation and layout of the fibres will be optimised to produce the best results for the given task.

Many composites are made up of layers or “plies”. These plies can be laid up in a variety of orientations and are usually chosen based on the required performance of the composite. An example of this would be a quasi-isotropic layup consisting of fibres orientated parallel and perpendicular to loading, as well as at an angle of 45°, written as [0°/90°/+45°/-45°]s; here the number of plies and the orientation of each is clearly displayed – the “s” stands for symmetrical about the mid plane.

Generally, composites are split into one of three groups, UD, 2D and 3D, referring to the number of fibre axes present. UD refers to all the fibres being unidirectional, while 2D plies typically have textile architectures and are usually either woven, knitted, or braided. Similarly, 3D plies are similar to 2D, however there is extra reinforcement through-thickness. 3D plies are generally much thicker than a single UD or 2D ply, containing multiple layers that are bound together to form a single ply.

2.3.1. 2D composites

Since 2D plies tend to follow textile based patterns, the terminology from this industry has been adopted to help avoid confusion when manufacturing these fibre layups. For instance, it is common to name unidirectional (UD) plies after their angle of orientation, with 0° often representing the loading direction. In contrast, 2D textile composites (excluding non-crimp fabrics and braids) have two perpendicular directions, the warp and weft. The warp and weft simply signify the fabric weaving directions, with the warp running lengthways and the weft covering the width. As discussed below, the fibres in the warp and weft directions in textile fabrics interlace, and thus make it difficult to have layers of fibres running in only one orientation. The fabric plies can be orientated at any angle to the loading direction, however the two fibre tows in a single ply will always be perpendicular to each other. The preforms consist of bundles of fibres, or “tows”, with the size of these tows variable to achieve the properties or flexibility desired.

A woven fabric is usually defined by the interlacing of tows in both the warp and weft direction. Unlike other fabric types, woven fabrics can have the highest fibre density. For woven fabrics density
can be measured in many ways. There is the warp and weft density which is determined by the number of ends or picks tows per cm. Ends and picks can be thought of as the individual tows in both the warp and weft directions, respectively [5]. The next density measurement is the linear mass density (LMD). LMD is usually measured in g/km or “tex” and is often treated as a rough representation of the size and/or amount of fibres per tow. “tex” is common to most European countries, whereas the U.S. uses the unit “yield”, which has units of yd/lb. The final density measurement for woven fabrics is the areal density. This is a measure of the fabric mass per unit area, with the unit g/m². This is used as a more reliable method for thickness representations. This is because measured thickness will vary as a result of applied pressure and other feature of the fabric.

For woven fabrics, there are many potential fibre configurations and structures. This is made possible through modifications of density parameters and the interlacing pattern [6]. The most basic 2D woven fabric is the plain weave and is effectively the interlacing of warp and weft in an alternating fashion, i.e. one over and one under in both directions, Figure 2.1. The majority of other 2D fabric structures are based on a change in the interlacing pattern. For instance, in a five-harness satin weave, the warp travels, or “floats”, over four weft tows before going under one (see Figure 2.1).

![Figure 2.1: Some 2D weaves examples. Left to right: Plain weave, twill weave, 5-harness satin weave [7]](image)

In woven structures, crimp can be an issue. Crimp is the effect of bending of fibres around each other and can result in a degradation of various properties. It is usually desirable to have straight fibres as their strength is highest parallel to the longitudinal axis. Properties such as strength and stiffness can be reduced in fabrics with a large crimp factor. It is found that a larger “harness” can provide better strength and stiffness, since there are longer regions of straight fibre before any crimp may occur. Of course, this is very idealised and reducing crimp is not always simple in many composite structures. Crimp can be measured by taking a tow from a length of fabric; the difference between the two, usually expressed as a percentage, is total crimp in the one direction [6].

2.3.2. 3D composites

One major weakness of UD and 2D composite layups is the through-thickness strength. In order to achieve desired properties and component thicknesses, many plies are usually stacked on top of one
another. Under many loading conditions, such as impact or cyclic loading, a stack of plies (known as a laminate) can become susceptible to a damage type known as delamination [7]. A delamination tends to occur at interfaces between different structural phases, such as between individual plies. This type of damage occurs because the strength along an interface between two layers is low, often with properties similar to that of the matrix.

3D composites are classically defined by the use of a through-thickness reinforcement to bind all of the layers within a composite structure together. It can be noted that this does not completely eliminate delamination damage, but does manage to limit its presence as the through-thickness reinforcement reduces major interfacial damage within the structure.

The main types of 3D fabrics are woven, braided, knitted, stitched and z-pinned. Z-pinning and stitching are the simplest methods of improving the through-thickness strength and producing a 3D composite. These work on the basis of inserting thin pins, or stitching fibres (i.e. non-crimp fabrics), into various locations within an already established composite structure; both of these methods have to be used before the composite is cured. However, these methods, especially z-pinning, will often have a knockdown effect on the in-plane properties of the original laminate [8, 9, 10, 11, 12].

In contrast to z-pinning and stitching, 3D woven preforms are produced on a loom as a self-contained preform. Unlike 2D woven, for 3D through-thickness reinforcement, the warp and weft tows are not interlaced in the conventional way. Instead, the warp and weft tows are generally kept straight, while the through-thickness reinforcement interlaces through the thickness. A 3D fabric can either be warp or weft interlaced. It is more common for warp interlacing, and as such all patterns discussed will assume this convention, unless otherwise stated. Warp and weft layers almost always alternate. This means there will always be one more weft, or warp, layer.

For the moment, the effect of structural changes to properties will be ignored as much of this will be discussed later. The use of through-thickness reinforcement (z-binders) for structural purposes has advantages and disadvantages. Compared to a traditional woven fabric, there is a greater freedom in the number of warp and weft layers. This is useful as it can result in a large reduction in crimp within the fabric since the warp and weft tows can remain straight.

For 3D woven fabrics there are three main categories of fabric structure: layer-to-layer, angle interlock, and orthogonal. Like the 2D weaves mentioned above, each of these categories can use similar methods of adjustment from their basic patterns, i.e. harnesses etc. It can be noted that angle interlock can be split down into two main categories which utilise components from each of the other two 3D woven fabric types.

- **Orthogonal:** In this case the z-binder travels vertically through the entire thickness of the fabric, passing over the surface weft tows. Ideally z-binder tows follow a square wave-like path Figure 2.2. However, z-binder tows tend to conform more closely to a sinusoidal wave-like pattern.
Along the transverse tows the pattern alternate, i.e. as binder yarn passes over the top of the fabric, neighbouring binders will pass under.

- Layer-to-layer: Similar to orthogonal weave, however the z-tow does not completely travel through the thickness of the fabric. In this case multiple binders overlap. In its most basic arrangement each z-tow will interlace between two weft tows. Each z-tow will begin its pattern a weft tow down from the previous. Like the orthogonal weave the pattern can alternate along the width; however, it can also alternate through the thickness.

- Angle Interlock:
  
  - Layer-to-layer angle interlock: This works in a similar manner to the layer-to-layer fabrics, except each z-tow follows a step like pattern. The number of tows covered by this type of pattern in the weft will always be an odd number, with the smallest equal to three.
  
  - Through-thickness angle interlock: This pattern follows the same step structure as the layer-to-layer angle interlock, but travels through the entire thickness of the material like the orthogonal weave pattern.

![Figure 2.2: Different 3D woven fabrics; a) layer-to-layer interlock; b) through-thickness angle interlock; c) orthogonal woven [5]](image)

2.4. 3D Composites – manufacture and properties

2.4.1. Weaving and weaving damage in 3D orthogonal woven fabrics

The manufacturing of 2D fabrics can be carried out on traditional or modified weaving looms. There are many types of looms, though the Jacquard loom is the most popular providing automation, high weaving speeds and the ability to control the structure of the fabric. The process used by the Jacquard looms to weave a fabric can be split into three main parts: shedding, weft (or “filling”) insertion, and beat up [13]. In a loom set up, warp yarns traverse the length, whilst the insertion of weft yarns occurs across the width. Each warp yarn is passed through a tensioning device and drawn through
a heddle eye, which controls vertical motion during weaving. Ahead of the heddles, warp tows proceed to pass through a comb-like component called a reed, which has the ability to move forward or back. To insert a weft yarn, warp yarns must be separated apart in an action called shedding (Figure 2.3a). The weft yarns are then inserted in front of the reed between the separated warp yarns (Figure 2.3b). Finally, the reed is moved forward to “beat-up” the weft yarns, or push into the correct position of the fabric, whilst maintaining separation of the warp yarns (Figure 2.3c) [14].

![Figure 2.3: Main processes for traditional 2D weaving](image)

Traditional weaving machines can be used to produce multilayer 3D preforms. However, according to Bogdanovich and Mohamed [13] manufacturing preforms using this method can reduce productivity as layers have to be built one weft tow layer at time. The biggest drawback of this method is the amount of damage that can be done to the warp tows. All the warp tows are drawn through heddles, and move up and down during each weaving cycle (shedding) regardless of the weaving pattern design. To avoid many of the issues involved in using 2D weaving machines, a fully automated 3D weaving machine was developed at North Carolina State University (NCSU), College of Textiles [13]. Unlike modified 2D weaving machines, this machine uses no traditional 2D weaving equipment and can produce an entire column of multilayer fabric every cycle. Weaving of 3D preforms using this machine only requires the z-yarns to be drawn through heddles to form a shed, while each warp tow layer is held in tension with no movement. In addition, the machine can insert multiple weft tows simultaneously enabling the production of entire columns of 3D preform to be produced [14]. Consequently, 3D preforms produced using this method have very little, or no, crimp in both the warp and weft tows, and damage to the fibre tows during weaving is reduced.

While dedicated 3D weaving machines are used by some technical textile weaving manufactures, many companies still use traditional 2D weaving machines to produce some multilayer 3D woven reinforcement. As a result, many studies analyse the amount of damage incurred when using modified 2D weaving equipment to produce 3D woven preforms, since the movement of the warp tows during shedding can lead to fibre damage. For instance, two investigations were undertaken by the Royal Melbourne Institute of Technology (RMIT) to assess the effects of the weaving damage on 3D orthogonal woven preforms using traditional weaving apparatus; one focussed on 3D carbon fibre preforms [15], while the other looked at 3D glass fibre preforms [16]. In both studies, in addition to the
final preform, samples from various stages along the weaving process were examined to determine weaving damage progression.

During the weaving of a multi-layered 3D carbon fibre orthogonal woven fabric preform (Figure 2.4a), damage to the warp yarns increased progressively with each stage in the weaving process [15]. This was shown to be the result of yarn abrasion against various loom components such as warp beams, tensioning devices, heddle eyes, and reed dents (comb-like spacing on the reed that warp tows pass through). Abrasion can lead to breakage of fibres within a yarn, with warp yarns seen to become increasingly “hairy” as the broken fibres cling to each other, in turn exacerbating damage. The damage to fibres were noticeably more severe by the beat-up stage than the tensioning or shedding stage. Although the damage to individual warp yarns increased at each stage of weaving, dry yarn property degradation was not affected beyond an initial knockdown of tensile strength and strain-to-failure from the as-received material by approximately 12%.

![Figure 2.4: 3D orthogonal weave architecture used in [15] (left) and [16] (right). Carbon fibre was used in [15], while glass fibres were used in [16]](image)

A similar damage progression was seen during weaving of a 3D glass fibre orthogonal woven fabric preform (Figure 2.4b) in a study by Lee et al. [16]; however, there was an increase in damage sustained during weaving, and loss of strength when compared to carbon fibre weaves. Lengths of dry yarns were removed from various stages of weaving and loaded in tension, where it was found that there is a progressive loss of strength during each stage. The greatest loss of strength occurred during the tensioning stage and was attributed to yarns sliding forward and backward a number of times, thus abrading and fracturing fibres. By the final stage of weaving, the total loss of strength of dry glass fibre warp yarns was approximately 30%, while these yarns only lost 20% when consolidated with resin. Additional work by Rudov-Clark et al. [17] looked at the effect of weaving on Z-yarns noting a loss of dry yarn strength of up 50%. For Z-yarns it was suggested that the severe bending, as they are forced to follow an orthogonal path, and smaller tow size, made them more sensitive to weaving induced damage. In both studies, it was not made clear how samples from each weaving stage were removed without further damaging the yarns. While damage to fibres were clearly observable during weaving, it
is possible that some of the loss of strength, particularly toward the final weaving stage, could have occurred during the removal and handling of yarns for testing.

Figure 2.5: Cross sections of a layer-to-layer and an angle interlock 3D weave both in an off-the-loom condition and after compaction with a pressure of 100 kPa – each were infused with an epoxy resin system; a) layer-to-layer off-the-loom; b) layer-to-layer compacted; c) angle interlock off-the-loom; d) angle interlock compacted [18] – the top and bottom images in each of a-d represent cross-sections along the longitudinal and transverse directions respectively.

A study by Archer et al. [18], looked at the effects of weaving, using traditional weaving techniques, and subsequent consolidation on two different carbon fibre reinforced 3D woven architectures; layer-to-layer and angle interlock. In a similar manner to studies presented above, warp
and z-binder yarns were removed at various stages of weaving and tested to see if there was any property degradation of the dry fibre yarns when compared to the as-received yarns. According to the authors, care was taken when removing warp and z-binder yarns from the fabric, and during subsequent testing to avoid any further degradation of fibre yarns. However, even careful extraction could have resulted in more damage to the fibre yarns, especially for any woven z-binder tows, since these will probably be difficult to remove. Nonetheless, it was reported that approximately 9-10% of the dry yarn strength was lost from as-received to fully woven for both warp and z-binder tows when subjected to tensile loading. The loss of strength measured in these layer-to-layer and angle interlock weaves is similar to the loss of strength in carbon fibre 3D orthogonal weaves reported by Lee et al. [15].

When Archer et al [18] Examined the structure of the layer-to-layer and angle interlock weave in the off-loom state, both the warp and weft tow layers were observed to be generally straight, but vertical alignment of warp tows in the layer-to-layer is less ordered than in the angle interlock (see Figure 2.5a and c). In addition, cross-sections of the angle interlock show that the warp and weft tows were able to maintain a regular geometry. In contrast, the path of each z-binder in the layer-to-layer weave causes natural structural compaction via nesting and lateral movement of tows, which resulted in thin and lenticular shaped warp and weft tows. Under consolidation, using vacuum assisted resin transfer moulding (VARTM), a compressive pressure of 100kPa was applied to the preforms. For both architectures, a large reduction in thickness was observed resulting in increased nesting of yarns in both directions (see Figure 2.5). This was most notable in the angle interlock weave, where the cross-section of individual yarns in each direction become thinner and wider. One feature of the structure common to both architectures was the presence of resin-rich regions; with z-binders present at each layer in the layer-to-layer weave, these regions were smaller than those in the angle-interlock weave.

While little damage may be sustained by warp tows when using a dedicated 3D weaving machine, the z-binders are still moved using heddles. According to 3TEX (the company that initially commercialised the system), this may still result in substantial damage being imparted to the z-binders [13]. However, the amount of damage that could be produced has not been quantified and this remains speculative. One fabric produced by the now defunct 3TEX had its internal geometry examined by Karahan et al. [19]; this fabric showed a high degree of fibre tow straightness and geometric uniformity. The variation in measurements of geometric components was found to be much lower than that measured in typical 2D woven fabrics with a measured variance of: 3-4% for yarn spacing; 4-5% for the tow width; 6-8% for yarn thickness; and 2-6% for yarn fibre volume fraction.

In summary, weaving of 3D woven preforms results in some knockdown in dry yarn strength during weaving. This knockdown will affect warp tows and z-yarns, if traditional weaving techniques are used, or mostly z-yarns, if a dedicated 3D weaving machine is used, as a result of abrasion of the fibre tows against various moving components on the loom. In addition, less damage is imparted at various weaving stages when using carbon fibres, when compared to glass fibres. However, it can be
noted that the damage sustained becomes less important when the final preforms are consolidated with a resin system as load redistribution around any damaged fibres will occur throughout the structure likely making any knockdown appear small.

2.4.2. Delamination resistance of 3D woven composites

In traditional 2D textile and unidirectional composites, the through-thickness properties are typically much lower than the in-plane properties. Under various loading conditions, such as cyclic loading and impact, delamination cracks can initiate through the rise of interlaminar stresses. Interlaminar stresses are induced by the increase of local out-of-plane loading, which are commonly the result of material and/or structural discontinuities [20]. Examples of these discontinuities include, but are not limited to, a mismatch of properties between interfaces at free edges, out-of-plane bending, stress concentration at tips of matrix cracks, ply-drops, and loading of bonded or bolted joints. There are three fracture modes that, combined, can be used to describe the deformation of any crack face; mode I, mode II, and mode III [21]. In relation to the crack faces, mode I refers to surface parting under tension, while mode II and III are shearing motions in the form of sliding (in-plane shear) and tearing (out-of-plane shear) respectively. There have been a number of suggestions for improving damage tolerance, and in turn resistance to delamination crack growth including: toughened resins, interleaving, and through-thickness reinforcement [20]. In the present work, only studies related to through-thickness reinforcement will be discussed.

With the addition of through-thickness reinforcement, it is often useful to quantify its influence on various properties such as interlaminar fracture toughness. Delamination resistance and fracture toughness of various 3D weaves under mode I and mode II loading has been the subject of many studies. Most of these studies tend to use 3D orthogonal weaves with glass or carbon fibres, though some have also considered layer-to-layer and angle interlock weaves. The results of these investigation are discussed below and share many similarities that can be used to provide a general overview of the performance of 3D composites with regards to delamination resistance.

In mode I loading, the load-deflection response of a 3D woven composite typically begins with a linear increase until crack propagation, followed by a load drop (Figure 2.6a) [22]. The load then rises again until another load drop occurs. This pattern repeats until total failure of the specimen, and is the result of a “stick-slip” action. Stick-slip is the unstable growth of a crack front as it comes into contact with a z-binder. Load increase is representative of the crack front crossing the z-binders, with a rapid drop in load occurring as the crack propagates through the matrix between binders [23]. As indicated in Figure 2.6b, these load drops are more pronounced and abrupt with increased z-binder content [22]. This can be further explained by discussing the interlaminar fracture toughness.

It was shown by both Mourtiz et al [24] and Tamuzs et al [25] that the magnitude of the initial mode I interlaminar fracture toughness during DCB testing of 3D woven composites tends to be similar
to both unidirectional and 2D textile composites. This value of the fracture toughness remains relatively constant until the crack front encounters the z-binders, upon which it increases rapidly [26]. The initially low fracture toughness will only occur if a crack begins some distance ahead of a z-binder, else the initial fracture toughness will appear high as the z-binder makes it harder for the crack front to propagate. Eventually, the interlaminar fracture toughness begins to level off, reaching a “steady-state” value (Figure 2.7). This is usually reached at a condition where z-binders are being encountered along the crack front while those behind it begin to fracture. Compared to multi-layered 2D composites, continued propagation of an interlaminar crack in a 3D weave requires an increase in input energy as a result of the z-binders. As the crack face opens, energy is absorbed by the composite and used to debond, pull-out, and fracture the z-binders [27]. In 3D orthogonal woven composites, z-binder fracture is found to initiate around the binder region near the surfaces [23]. In many of the 3D weaves tested it was not uncommon to see the formation of secondary cracks, via crack branching, which would generally run parallel to the main crack. The combination of each of these mechanisms corresponds to an increase in fracture toughness. The development of secondary cracks may suggest that other loading modes are present during the loading of 3D weaves, contributing to the measured fracture toughness. However, while this was not discussed by any of the authors in these works, these values were treated as “apparent” toughness values.

Figure 2.6: Effect of z-binder content on a carbon fibre/epoxy 3D orthogonal weaving subjected to mode I DCB loading conditions. a) load-displacement response, and b) interlaminar fracture toughness initiation and steady-state values [22]
Figure 2.7: Delamination resistance (R-curve) of various carbon fibre/epoxy 3D woven composites subjected to mode I loading indicating the behaviour of interlaminar fracture toughness with crack length. a) standard layer-to-layer; b) toughened layer-to-layer; c) orthogonal with 6\% z-binder content; d) orthogonal with 3\% z-binder content; e) angle interlock [26]

One study by Tanzawa et al. looked at the effect of z-binder tension on mode I delamination resistance, consistently noting an increase in the fracture toughness in specimens with a lower z-binder tension [23]. Consequently, the lower tension specimens were found to be able to maintain a higher load with crack extension, while the load held by high tension specimens gradually decreased. In the lower tension specimens there is “slack” in the z-binder which delays fracture by allowing greater pull-out to occur during mode I loading. This effect was also observed by Stegschuster et al. in a thin 3D orthogonal woven composite where the z-binder incline angle is high relative to the orthogonal direction [22]. In this case, the length of z-binder incline is greater than the thickness of the specimen, which results in a greater length of z-binder to be debonded and pulled out during mode I loading. Unlike orthogonal binders which are subjected to axial tension, the stresses acting on the inclined binders are axial shear and through-thickness tension.

Figure 2.8: Typical load-displacement response for 3D orthogonal woven composite (left), with the development of damage shown schematically (right) [28]
3D woven composites loaded in mode II using End Notch Flexure (ENF) conditions have been shown to exhibit trends which differ to those presented in mode I loading. Analysis of the load-deflection response indicates initial loading is linear with no crack growth, with a slope reduction associated with the initial propagation of the crack front. This slope remains linear while both the main crack, and the subsequent secondary crack, develop (Figure 2.8). It can be noted that the secondary crack does not initiate until the first crack becomes arrested; both cracks stop growing once a point below the loading head is reached. Following the complete propagation of both cracks, a kink band from compression under the loading head and matrix cracking on the tensile surface form, represented by an abrupt drop in load [28]. Whilst the interlaminar fracture toughness in mode I has been shown to reach a steady state value, studies in mode II have not observed this. Two studies [26] [28], both of which tested different 3D weave architectures, found that the interlaminar fracture toughness followed a continually increasing trend with crack length, with no obvious levelling off (Figure 2.9a). As indicated by the authors, this increasing trend suggests an increase in load is required for continued crack propagation.

In [28], Pankow et al. impacted ENF specimens at low velocity, noting that increased loading rate increased the measured interlaminar fracture toughness of the 3D weaves tested. Observations of damage progression during loading showed that at the higher loading rates, the crack front took longer to develop to the same point as was seen during quasi-static loading. The issue with the comparison of low velocity impact and quasi-static loading of ENF specimens is that there is a large difference in the loading speeds. The highest quasi-static loading speed was 50.8 mm/s, while the lowest impact speed used was 2.8 m/s. It is possible that under very rapid loading, mechanisms enabling crack growth may not have the time to develop and an “apparent” increase in mode II toughness could occur. However, the investigation of more loading rates is needed to prove that the increase in mode II fracture toughness
is not just related to impact loading rates. The effect of loading rate on delamination growth was more noticeable in specimens loaded along the warp direction (Figure 2.9b).

While there appears to be an increase in mode I fracture toughness of 3D woven composites, it must be noted that none of the authors cited above indicated whether there was any influence of any other fracture modes. For instance, it was shown in many of the mode I studies that when the crack front reaches the z-binders, it slows down and many other damage mechanism, including crack branching occur. This kind of damage should not develop during pure mode I loading, and therefore may be indicative of other failure modes contributing to the improved performance. In contrast, no direct comparison of mode II loading has been made between 2D and 3D woven composites, and it is unclear whether the mode II fracture toughness improves with a z-binder. However, stable crack propagation and a higher load have been observed in z-pinned composites when compared with their unpinned counterparts, which may indicate some improvement [29]. Nevertheless, the fracture toughness values measured in the studies above can only be treated as “apparent” toughness values since the influence of the z-binder on the measured values needs to be quantified.

2.4.3. Impact resistance of 3D woven composites

With the increasing application of composite materials to component designs in a number of different industries, their ability to withstand and/or resist the development of damage is important. This is of particular note in cases where components may become subjected to various forms of impact loading. Due to the weak through-thickness properties of traditional layered composites, out-of-plane loading (e.g. impact) can potentially lead to damage, ultimately, reducing the designed performance of the component. As indicated in previous sections, the addition of through-thickness reinforcement, through weaving and pinning, can improve through-thickness properties; this is seen from the substantial increase in both modes I and II fracture toughness when compared to their traditional composite counterparts.

Some studies by Baucom and colleagues have investigated the effect of repeated impact on various 3D weaves, often comparing performance to 2D fabric preforms [30, 31]. In each of these studies the 3D orthogonal and 2D plain weave composites were impacted at a fixed velocity and impact energy multiple times until perforation of the specimens occurred. It was generally noted that the number of strikes needed to perforate each 3D orthogonal weave was greater, by at least two times, than strikes required for the 2D plain weave [30] – see Figure 2.10. This trend was also mimicked in the total cumulative energy dissipated by perforation, determined from the incident and residual velocity of the impactor using translational kinetic energy [31]. The greater amount of energy absorbed in the 3D weaves during multiple strikes was translated into more extensive damage in the forms of matrix cracking and debonding/delamination of the z-binders and surface weft tows.
Figure 2.10: Comparison of a 3D orthogonal weave, 2D plain weave, and a biaxially reinforced warp knit subjected to multiple impact strikes until perforation where (a) shows the peak force per strike, and b) shows energy dissipated per strike. Each material was infused with a vinyl ester resin system. [30]

For low-velocity impact testing, many studies made use of a Split Hopkinson Pressure Bar (SHPB) apparatus under a variety of different velocities [32]-[33]-[34]. In each of these works, increased velocity resulted in an increase in the energy absorbed. Gerlach et al. [34] noted that the increase in energy absorbed was the result of the initiation and development of different damage mechanisms which absorbed energy. The authors also showed that the lowest impact velocity used resulted in the most dominate failure mechanism to be debonding and delamination. By increasing the impact velocity, significant fibre rupture as well as the addition of intra-ply failure occurred. Under ballistic velocities [35], damage sustained has been found to be significant and more extensive than under low-velocity impact. Bulging at each surface has been observed with extensive separation of layers noted around the impacted pathway.

Fibre architecture of 3D woven composites also appears to play a role in the extent of damage and energy absorption. Work by Gerlach et al [34] compared the performance of two 3D angle interlock weaves with different z-binder volume contents. In this study, impact resulted in smaller delaminated areas in specimens with a higher binder content. This effect was also noted to occur in a study by Potluri et al [36] that tested a variety of different 3D weaves including modified and standard layer-to-layer, angle interlock and orthogonal. Here, it was noted that the damage area surrounding the impact site would increase as a linear function with increasing impact energy. When compared with a 2D plain weave and UD cross-ply, the damage area was noted to be considerably smaller in all of the 3D weaves tested (see Figure 2.11). The damage resistance of the 3D weaves, from highest to lowest, was found to be in the order: modified layer-to-layer, standard layer-to-layer, angle interlock, and finally orthogonal.
Hart et al. studied the impact damage of 2D plain weave and 3D orthogonal woven beam specimens subjected to different impact energies [37]. The loading response of specimens impacted with energies of 10 J or more resulted in a regular load drop occurring in both materials. This was found to be representative of inter-ply delamination formation and propagation. The load responsible for the initiation of delamination was defined as the delamination threshold load (DTL). It was found that DTL is approximately constant across both materials and impact energies, indicating that it is independent of fibre architecture and is controlled by both the matrix properties and the strength of the fibre-matrix interface. Higher impact energy results in the development of more delaminations, until delamination development and growth becomes saturated; the load now exceeds the DTL, with a build-up of internal strain energy due to the absence of delamination fracture absorbing the energy. For both materials, the load profiles are very similar until delamination saturation, after which the through-thickness reinforcement provides stiffening of the material.

Another study by Seltzer et al [38] investigated damage mechanisms in 3D orthogonal weave composites under low velocity impact, and compared how the various damage modes present in impacted glass and carbon panels influenced the performance of a hybrid version consisting of layers of glass and carbon. The hybrid preform had three warp layers and four weft layers; the top two and a half layers were carbon, while the remaining four and half layers were glass. Both the glass and hybrid weaves used polyethylene fibre (PE) as the z-binder, while the carbon fabric had carbon fibre z-binders. It was generally found that the 3D glass specimens could withstand higher measured impact loads than the 3D carbon specimens. It was suggested that this increase in load response is the result of the higher strain-to-failure of glass fibres as compared to carbon fibres. In absolute terms, the hybrid performed worse than both the carbon and glass weaves. However, while the areal density of the glass and carbon preforms are similar, that of the hybrid is much lower. As such the authors normalised the peak load
with respect to the areal density and noted that the hybrid was therefore superior to the carbon weave, but still inferior to the glass weave.

Using x-ray tomography, damage progression away from the impacted surface was studied by Seltzer et al. [38]. At a small distance from the impact centre, damage in the all specimens tested consisted of z-binder debonding, surface resin cracking, fibre tow kinking, tow splitting, and tensile fracture toward the back surface. In the carbon specimens, the presence of fibre tow kinking was greater and more prevalent toward the upper surface than in the glass specimens. Carbon z-binders were also observed to have fractured at a small distance from the impact site, which often led to the development of delamination cracks; in glass specimens, the PE z-binders were only noted to begin failing at distances much closer to the impact centre. Cross-sections closer to the impact centre revealed damage emanating from the impacted surface in a cone type shape. Within this region, tensile fracture of both the warp and weft tows is seen toward the interior of the specimens. Additional damage present here includes micro-buckling and tow bending, severe tow splitting, and the fracture/debonding of the z-binder. Under the impact centre, complete failure of the composite is observed in the formation of a plug type shape from the impactor; material surrounding this region is relatively intact. An example of the damage present in an impacted glass-fibre 3D orthogonal weave scanned using x-ray tomography can be seen in Figure 2.12.

In [33], Hao et al manufactured a T-beam from a 3D orthogonal weave using three pieces of the preform and impacted the structure using the SHPB method. Two pieces were bent into L-shapes to produce the web and part of the flange, while a single piece was attached to the front as the flange. All three pieces were stitched together using aramid filaments. It was indicated by the authors that more of the impact energy was absorbed by the T-beam than by a single ply thick plate of the same material tested by Ji et al. [39]. The comparison of a T-beam and a flat plate made of the same material does not work as the structures are clearly very different. In addition, while in absolute values the T-beam did absorb more energy from the impact, the authors made no suggestion to the fact that the webs of the T-beam were actually two plies thick, while the plate was just a single ply. Both differences do not make this a very good comparison. However, Baucom and Zikry [40] observed that stacking multiple 3D orthogonal woven plies provides greater energy absorption over that of a single 3D orthogonal ply of the same thickness. This observation was made by comparing the performance of a thick and thin single-ply 3D orthogonal weave with a 3D laminate consisting of three plies of the thinner 3D orthogonal weave. Testing showed that the energy absorption was greater in the three-ply 3D, followed by the thick single-ply 3D, followed by the thin single-ply 3D. Greater energy absorption with three plies of 3D weave seems counter intuitive since the purpose of the z-binders is to increase through-thickness strength, and having three plies of 3D reintroduces some of this inherent weakness between plies. It was mentioned that having two ply-level interfaces means there are more z-binder crowns in the overall composite structure, suggesting that the authors believe that the z-binder crowns are uniquely
responsible for increasing the energy absorption in 3D orthogonal weaves through their interaction with the rest of the structure.

In addition to understanding how impact may affect the immediate condition of a component, it is also important to determine what level of load carrying capacity is retained after impact. In [36], Potluri et al impacted a variety of different 3D weaves at various impact energies, including: a modified layer-to-layer (i.e. extra warp tows interlacing along the top and bottom surface like a plain weave), a standard layer-to-layer, an angle interlock, and finally an orthogonal weave. Each of these 3D woven composites were loaded in compression after impact (CAI). It was observed (Figure 2.13) that each of the 3D weaves had a similar rate of loss of compressive strength with increasing impact energy. Differences in the CAI strength from weave to weave were mainly found to be a function of the undamaged compressive strength, which in turn was a function of the tow waviness along the loading direction. As such, whilst being the most damage resistant, the modified and standard layer-to-layer architectures had the highest tow waviness and thus lowest undamaged compressive strength.

A study by Hart et al on CAI and flexure-after-impact (FAI) investigated the behaviour of a 3D orthogonal woven composite compared with a 2D plain weave composite of equivalent areal density [41]. For CAI testing of the 3D architecture, no significant loss of strength and modulus was seen at all impact energies tested. This was attributed to the through-thickness reinforcement restricting localised buckling by binding all the layers together. It is interesting to note that the impact energies used in this study were higher than those used by Potluri et al [36], and yet no changes in compressive strength were seen. The specimens used by Hart et al were approximately 70% thicker than those used by Potluri et al, so it is possible that a higher impact energy is required to produce an equivalent amount of damage and therefore loss of compressive strength. In contrast, a large reduction in post-impact flexural strength and modulus was observed in the 3D weave with increasing impact energy during FAI testing. In the 2D plain woven composites, there was a greater reduction in post-impact strength than the 3D orthogonal weave for both CAI and FAI. However, the modulus remained higher in the 2D weave as a result of its higher fibre volume fraction. A comparison between CAI and FAI was attempted by normalising the impact energy by the unclamped specimen volume to produce a “energy density” (see Figure 2.14). However, this is not very useful since the “energy density” does not seem like a clear measurement comparison technique. Additionally, the sample geometries are very different, with CAI performed on plate specimens and FAI on beam specimens; this causes vastly different amounts of damage, thus not making them easily comparable.
In the works presented above, it has been suggested that 3D woven composites have a higher impact resistance than both 2D and UC composites. The increase in impact performance is related to mechanisms by which the 3D fibre architectures absorbs and dissipate energy; this is mostly in the form of extensive damage, via matrix cracking, debonding and delamination of the z-binder and in-plane tows.
There is some conflict in the reported post-impact compression performance of 3D orthogonal weaves, and it is believed further work in this area is required to resolve this understanding.

Figure 2.13: Graphs showing a comparison of a) normalised CAI strength against impact energy, and b) normalised CAI strength against damage width for a variety of different 3D woven preforms [36]
2.4.4. Mechanical characterisation of 3D woven composites

2.4.4.1. Compression

For compression loading of 3D woven composites, Cox et al. [42] examined the compressive failure mechanisms of a carbon fibre reinforced layer-to-layer and a through-thickness angle interlock composite. In comparison, the loading response of both 3D composites became non-linear on approach to the maximum load. Upon reaching the maximum load, a sudden drop in load occurred to slightly above 50% of the max load. This load was maintained until around 15% strain. Cross-sectional examination of early damage development was seen to consist of the debonding of the z-binders from surrounding material, with much of this debonding extending along warp/weft tow interfaces. Debonding of warp tows continued until a critical load was reached where some buckle and fail, resulting in a load drop. As mentioned above, some load was maintained, with further buckling of entire layers occurring, causing the specimen to become barrel-shaped. However, the z-binders continue to constrain the layers from buckling further. Instead kink bands became a key damage mechanism that developed along each of the warp tows, eventually leading to shear bands that separated the specimen into large pieces and led to final failure. Development of kink bands occurred as a result of initial misalignment of warp tows in the fibre architecture and proximity to the z-binder.

Another study by Cox and colleagues looked at the effect of compaction during consolidation on the mechanical performance and damage development of a variety of 3D composites subjected to tension, compression and flexure [43]. Materials consolidated with a minimal compaction pressure, known as light compaction composites (LC), had a low fibre volume fraction and an irregular structure with relatively distorted warp tows. Materials compacted with a substantial pressure of 1.5 MPa were called high compaction composites (HC) and had a relatively high fibre volume fraction with heavily distorted weft tows and z-binders, but straighter warp tows; these were also half as thick as LC specimens. For both LC and HC specimens subjected to compression loading, the strains to failure were quite high, similarly to those found in [42]. HC specimens were seen to have 2-3 times the strength of
LC specimens along the warp-direction, but a lower strain-to-failure. Failure of HC and LC specimens were different, with HC prone to buckling and delamination failure, whereas LC specimens rarely buckled, instead suffering kink band formation on all warp tows. The increase in compressive strength with reduced thickness of these specimens seems reasonable since the warp tows are straighter in HC than LC specimens. Spatial distribution of geometric features in HC specimens, i.e. fibre tows, was much closer than in LC specimens, thus failure of a warp tow will probably cascade through the other tows much quicker as a result of load transfer, thus failing the composite with a lower strain-to-failure.

In [45], Warren et al tested three carbon fibre 3D weaves (orthogonal and two layer-to-layer) in compression, with mechanical performance and failure examined. In both layer-to-layer preforms the weft tows had 24K weft tows, while the warp tows in each fabric were either 12K or 24K. For all three preforms it was noted that there was a greater amount of weft tow waviness than warp tow waviness. This seemed to influence the properties in both directions as the strength and stiffness of the weft direction was always worse than the warp direction. The strength and stiffness of the orthogonal weave was better than the layer-to-layer fabrics due to this weave having straighter tows in both directions.

Fibre tow waviness in non-crimp fabric preforms is typically detrimental to the performance of a structure under compression, which is not easily avoidable in 3D woven structures such as layer-to-layer and angle interlock. This is because they weave round multiple layers rather than travelling through the thickness vertically like orthogonal weaves, and therefore will influence the relative straightness of the warp and weft tow layers, especially when consolidated into a composite. Under compression loading, failure mechanism in the 3D orthogonal weave was observed along the warp and
weft-direction using cross-sections. It was noted that failure along the warp direction consisted of mild brooming and kink band formation, while the weft direction had more fibre tow microbuckling. In both layer-to-layer fabrics loaded along the warp and weft direction, failure consisted of more kink band, fibre tow micro-buckling, and delamination damage than the orthogonal weave. A higher proportion of kink band and micro-buckling in the layer-to-layer fabrics seem reasonable as a result of the fibre tow misalignment.

Figure 2.16: Comparison of compressive strength and modulus of six different 3D woven architectures [44]

The compressive properties and damage developed in four different orthogonal weaves and two angle interlock weaves was investigated by Dai et al [44]; the architecture of each material can be seen in Figure 2.15. It can be seen in Figure 2.15 that the orthogonal weaves can be split into two groups, the first is a traditional orthogonal weave where the z-binder interlaces one weft tow column at a time (1-by-1), while the other three interlace three weft tow columns (3-by-3). In each of the 3-by-3 orthogonal weaves, the pattern of z-binders was adjusted to see what effect it had on the performance. Similarly, the second angle interlock preform did not have any warp tows, only weft tows and z-binders. Under compressive loading, each of the orthogonal weaves had a similar strength and stiffness as shown in Figure 2.16. In contrast, the properties of both angle interlock were drastically different from each other, with the architecture known as W-3 having a superior strength and stiffness due to the presence of warp tows. Observations from specimen cross-sections show common damage features in each preform included matrix cracking, delamination, and warp tow fracture, except for W-4 with regards to warp tow fracture. Due to different z-binder paths, delamination crack lengths during compression loading were different for each preform. The shortest delaminations occurred in W-1 specimens, occurring between every two layers and only extending two unit cells before being arrested. The z-binder sequence in W-2.1 produced the longest delaminations when compared to W-2.2 and W-2.3. In W-3, the angle interlock z-binder sequence causes large distances between through-thickness portions of z-binders at different layers. As such, delamination growth is less inhibited for greater distances
before being arrested. Finally, in W-4 there are no warp tows, so no delamination growth occurred. However, the through-thickness interlacing tows did become debonded from the weft tows, which reduced its load-bearing capability.

Studies on compression loading of 3D woven composites have shown that the mechanical properties and damage development are generally dependent on the degree of fibre tow misalignment along the loading direction. Damage often resulting in longitudinal fibre tow kink band formation as well as debonding and delamination leading to buckling. It is interesting to note in one study a layer-to-layer was susceptible to fibre misalignment because of the z-binder tows weaving between each weft tow layer, whereas an angle interlock fabric in another study, which weaves in a reasonably similar pattern through the thickness, does not have much crimp. This difference is probably due to layer-to-layer fabrics having multiple z-binders inline vertically, while the angle interlock does not. Generally, orthogonal weaves remain quite consistent regardless of the z-binder path, since the z-binder does not interfere with the in-plane fibre tows as much as other weaves.

2.4.4.2. Tension

There is a relatively large literature on the tensile behaviour of 3D composites. Lomov and colleagues [46] [47] examined the quasi-static tensile performance and damage development of two E-glass single ply non-crimp 3D orthogonal woven composites (3D-96 and 3D-78), with comparisons made to an E-glass 2D plain weave composite (2D-24). One of the main differences between the two 3D orthogonal weaves was the number of warp and weft layers. 3D-96 had three warp tows and four weft tow layers, while there were only two warp tow and three weft tow layers in 3D-78. The fabric names were related to their areal density, i.e. 3D-96 has an areal density of 96oz/yd². Through intensive testing and analysis, it was concluded that both 3D fabrics performed better under tensile loading conditions that the 2D plain weave (see Table 1). Not only did both non-crimp 3D orthogonal woven composites have higher in-plane ultimate strength and strain, but the damage initiation threshold (DIT) was also significantly higher than that of the 2D plain weave. It was suggested by the authors that the differences between 3D and 2D in-plane properties may be two-fold; (1) related to some combination of inherent crimp and interlaminar shear stresses found within the 2D composites – both are negligible in 3D orthogonal woven composites due to the in-plane yarn straightness; and (2) weaving damage may be more severe in 2D fabric preforms – this is because 3D weaves are produced on modified Dornier looms, operating at lower speeds than used for 2D fabric, and as such less damage may be imparted on fabrics at lower weaving speeds [46].

Table 1: Tensile property comparison between two non-crimp 3D orthogonal woven composites and one four ply 2D plain weave composite taken from [46] [47]

<table>
<thead>
<tr>
<th>Type</th>
<th>Direction</th>
<th>E@50%Vf</th>
<th>v</th>
<th>σult@50%Vf</th>
<th>εult(%)</th>
<th>εmin(%)</th>
<th>ε1(%)</th>
<th>ε2(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3D-96</td>
<td>Warp</td>
<td>24.6±1.2</td>
<td>0.14±0.16</td>
<td>435±34</td>
<td>2.74±0.29</td>
<td>0.26±0.11</td>
<td>0.43±0.04</td>
<td>0.54±0.04</td>
</tr>
</tbody>
</table>
It was mentioned in [46] that damage in the 2D-24 composite initiated at lower strains than its 3D counterparts. Damage in the 2D-24 seems to initiate as high-density transverse matrix cracks, whereas in the 3D composites damage initiation locations depend upon the loading direction [47]. For the warp loaded 3D composites, the z-binders appeared to be stress concentrators; damage initiation seemed to be localised toward the weft tow situated below the z-crown. Weft loading, on the other hand, produced transverse matrix cracks in both the warp and z-binders. How these damage mechanisms affect or aid in the ultimate failure of these specimens was not investigated.

Similar work was conducted on three different glass fibre preforms by Callus et al [48]: orthogonal, layer-to-layer interlock, and offset interlock. These authors noted that for all three 3D composites, cracking tended to initiate preferentially at the z-binders, though unlike in the work of Lomov et al [46] this was in the form of interfacial damage between the binders and the resin. In addition, in [48], Callus et al showed cracking within the resin rich regions between tows. It was suggested that in glass-fibre composites cracking may initiate in the resin rich regions as the maximum tensile strains present are higher than the bulk strain. As a result, lower bulk strains would be required to cause cracking to occur along the resin-rich regions, and examples of this damage can be seen in Figure 2.17.

Cox et al investigated carbon fibre reinforced 3D layer-to-layer interlock and 3D through-thickness angle interlock woven composites and observed three distinct stages during tensile loading (shown in Figure 2.18) [49]. These stages were; (1) elastic loading up to approximately 0.5% strain; (2) a hardening phase, represented by a non-linearity in loading between strains of 0.5-2.5% - this was suggested to be due to the straightening of misaligned tows, though the strain-to-failure of the carbon fibres was 1.5% strain, so misalignment is unlikely to provide an extra 1% strain increase. Damage investigations found that large portions of warp tows had fractured during this loading stage. A
combination of tow waviness, and through-thickness compression provided by the z-binders, were suggested as methods enabling load transfer from the regions of fibre fracture enabling continued loading. If, as the authors have noted, fibre fractures have developed during this loading stage, then some form of load transfer must occur in order for loading to continue, else the specimens would have fractured sooner. However, it was not mentioned how near to the peak load that the fibre fractures were observed. Therefore, it is possible that these fractures occurred near to the peak load, thus load transfer would be limited. Finally, (3) is the pull-out phase – at strains greater than 2.5%, a large load drop occurs. Strains here become quite significant and it was suggested that failing z-binders caused the load drop and allowed fractured warp tows to move past each other with relative ease. It can be noted that the final pull-out stage of loading does not appear to have been reported in other 3D papers, likely because this information is not really useful to specimen loading as a component reaching this level of strain would not be able to continue functioning as intended.

![Figure 2.18: 3D layer-to-layer angle interlock stress-strain curve. Taken from the work of Cox et al [49]](image)

The tension properties of an orthogonal and layer-to-layer preform were examined in [45]. The stress-strain response for the layer-to-layer composite was clearly bilinear, while the orthogonal was more linear to failure; a linear response for orthogonal weaves was also noted in [50]. The bilinear response of the layer-to-layer was more pronounced for weft-direction loading, resulting in a lower strength-to-failure as well as reduced linear elastic properties. In addition, the strain-to-failure for the layer-to-layer was greater for the weft direction loaded specimens. Since there was more crimp in the weft tows, it was suggested that the higher strain-to-failure along the weft-direction was related to tow straightening. For the orthogonal preform, the strength and stiffness was found to be greater than both layer-to-layer preforms. Like the layer-to-layer, the strength and stiffness of the weft direction was lower than the warp direction properties. The difference in modulus was attributed to a mismatch in in-plane fibres, while it was suggested that the difference in strength was a result of tow waviness and the addition of z-binders along the warp direction.

In addition to studying compressive properties of four different orthogonal and two angle interlock weaves (see Figure 2.15), a study mentioned previously also analysed the tensile performance
of these preforms loaded along the warp direction [45]. With regards to stress-strain response, each exhibited linear behaviour initially becoming more non-linear toward failure. Non-linear behaviour was due to damage development and load redistribution for all of the preforms with the exception of W-4; for W-4, the non-linear behaviour was a combination of damage development and straightening of the z-binder tows since there were no straight warp tows in this preform. The strength and modulus for each of these weaves has been normalised to their warp fibre volume fraction and plotted in a bar chart for comparison (see Figure 2.19). The tensile modulus is influenced by the amount of fibre tow waviness, with a higher waviness producing a lower modulus. It was also found that resin-rich regions, high strain between surface weft tows, as a result of the matrix having a lower modulus than the fibres, led to development of transverse cracking. In Figure 2.19, W-3 (an angle interlock weave) can be seen to have the highest strength and stiffness. It was indicated from cross-sections of this weave that there was less crimp in the warp tows due to compact weft tows and angle z-binders, and this weave had the smallest resin-rich regions. In contrast, the z-binder path in W-2.1, W-2.2, and W-2.3 resulted in large resin-rich regions, and the strength-to-failure was lower.

Figure 2.19: Comparison of tensile strength and modulus of six different 3D woven architectures [44]

Using the weaves W-1 and W-3 (see Figure 2.15), one study looked at the effect of notches on the tensile properties [51]. Each specimen had a width of 25 mm, and a notch diameter of either 4.1 mm or 12.5 mm drilled into the centre of the specimen gauge. With a notch of 4.1 mm, the net section strength of W-1 and W-3 specimens was 15% and 2% lower than the un-notched specimens respectively. With an enlarged hole diameter of 12.7 mm, the loss of strength compared to the un-notched strength became 20% for W-1 specimens and 7% for W-3 specimens. From surface strain mapping via digital image correlation (DIC), it was seen that the strain distribution for W-3 was more uniform across the whole length as a result of smaller resin-rich regions, whereas there were larger resin-rich regions in W-1 specimens and so the strain distribution reflected this. Failure modes in the notched specimens were similar to those seen in un-notched specimens and included matrix cracking,
debonding, and warp tow fracture. In both W-1 and W-3 notched specimens, longitudinal tow splitting cracks developed from the notch edge where portions of warp tows had been cut by the notch. Due to the stress concentration around the hole, warp tows debonded, causing a relaxation in the stress and thus reducing the notch sensitivity.

Munoz et al investigated the tensile behaviour of a hybrid 3D orthogonal weave, looking at both the notched and un-notched properties [52]. This preform had three warp tow layers and four weft tow layers. The top four layers of this hybrid preform were composed of S-glass fibre tow, the bottom two layers had carbon fibre tows, and a hybrid weft tow layer consisting of glass and carbon fibres separated these two sections. The z-binders were ultra-high molecular weight polyethylene. Additionally, every other carbon warp tow was removed from the structure. The structure of this fabric can be seen in Figure 2.20. It can be noted that the hybrid layer did not have mixed glass and carbon fibres, instead they were split into two distinct regions. Due to the asymmetry of this fabric, strain measurements were made along both surfaces during tensile loading. It was found that there was little difference in strains on both surfaces as shown in Figure 2.21e, indicating that any extension-bending coupling induced as a result of the asymmetry did not play a significant role during loading. The stress-strain response of the un-notched hybrid preforms loaded in tension along the weft and warp directions can be seen in Figure 2.21a and Figure 2.21b respectively. Weft direction loading was initially linear up to approximately 1.2-1.6% strain, after which there was a load drop associated with the failure of carbon fibre tows. A slight increase in load until final failure was associated with a take up of load by the glass fibre tows until failure. For the warp direction, a similar initial loading occurs until the carbon fibre tow failure. Unlike the weft direction, loading of the warp-direction continued after the load drop to its maximum strength, and was connected to glass fibre tow failure. For both loading directions there were two load peaks with a load drop between. For weft-direction loading, the first peak was related to the maximum strength of the weft direction, while the maximum strength in warp direction was reached at the second peak. The maximum strength of the weft direction specimens was higher than the warp direction and was probably because of the greater proportion of carbon fibres along the weft direction. Similar trends in the stress-strain response were noted for specimens with a notch of 4.1 mm and 11 mm. However, increasing the notch size resulted in more non-linearity in each loading response, as can be seen in Figure 2.21c and Figure 2.21d. In Figure 2.21f the normalised strength (normalised to average failure strength of un-notched specimens) is plotted against the ratio of hole diameter to specimen width. Here it can be seen that the loss of strength with increasing hole size in these specimens is essentially linear. This implies that the stress concentration induced by the hole does not influence the failure strength of notched specimens, and as such these specimens are notch insensitive.
Figure 2.20: Hybrid 3D orthogonal woven structure. Top four layers (red) are glass fibre tows and the bottom two layers (grey) are carbon fibre layers. The yellowish layer is a hybrid layer containing glass and carbon fibres. The z-binder (blue) is polyethylene. [52]

Several articles have also been written about the effects of binder path on tensile properties and failure mechanisms. One article studied two 3D orthogonal woven carbon composite preforms with the same layup, but different z-binder path lengths [53]. It should be noted that in these fabrics the z-binder interlaced in the weft direction. The first fabric, termed “normal”, had the z-binder following the standard sinusoidal pattern. The other preform was modified such that the z-binder path was longer, causing it to follow a squarer path. Cross-sectional images are shown in Figure 2.22. When comparing Figure 2.22a with Figure 2.22b a large difference can be immediately noted. In the “normal” version, large resin rich regions and areas of high fibre density are created when the z-binder follows its sinusoidal path. When z-binders follow this path, they are said to ‘pinch’ the surface tows. By extending the z-binder path, much of the pinching of the surface tows is alleviated. Through tensile loading in the warp direction it was found that the modified version had significantly better strength and strain to failure than the standard “normal” fabric by 40% and 90% respectively. The strength along the weft direction in the modified fabric was also shown to be better by about 30% compared with the normal fabric, whereas the strain-to-failure remained practically the same. It was noted that in the “normal version”, the pinching of the surface warp tows causes crimping to occur in the weft tows. As mentioned before, extension of the z-binder path length reduces much of the force acting on the warp yarns, thus allowing the weft tows to be straighter. It was stated that near failure, the modified 3D woven preform began to experience splitting; this was not seen to occur in the “normal” preform. The mechanisms of failure for both the normal and modified composites were not provided.
Figure 2.21: Stress-strain responses for a hybrid 3D orthogonal weave: a) un-notched weft direction; b) un-notched warp direction; c) notched weft direction; d) notched warp direction; e) warp and weft direction strains on either specimen surface; f) normalised strength vs ratio of hole diameter to coupon width [52]
A study by Quinn et al. [54] looking at the effect of through-thickness z-binder harness on the tensile properties of 3D carbon orthogonal woven composites, as well as their failure mechanisms, noting that increasing the float length improved the strength and stiffness. It was suggested that this was due to the increased fibre content in the loading direction, as much of the z-binder float would lie in the longitudinal plane. Like previous work, it was noted here that the presence of a z-binder created large resin rich regions between neighbouring weft tows, as well as pinching of the surface weft tows. Failure during tensile loading was shown to occur in the region where the z-binder entered the fabric. It was hypothesised that increased localised strains in the resin rich region due to lower modulus of the matrix may explain the failure in this location. This is similar to that reported in [48] with regards to the initiation of cracking in these resin rich regions. Using Electronic Speckle Pattern Interferometry (ESPI), the variation in strain over a specimen gauge length was mapped. It was shown that the regions of highest strain corresponded to the regions where the z-binders entered the fabric. As can be seen in Figure 2.23a, there are distinct regions of higher strain that appear at regular intervals and correspond to areas where the z-binders enter the fabric. Figure 2.23b is a line profile taken along the length of a z-binder – the regularity noted is clear. In Figure 2.23b there are four line profiles for four loading phases, each of these phases a higher load, and hence a higher strain, applied to the specimen. The average strain in loading phase 4 is approximately 0.29%, while the strains around the resin-rich regions can be seen to be greater than 1%. Therefore, it is possible that cracking may eventually develop in this region if the strains increase enough to reach the failure strains of the matrix. Digital image correlation (DIC) shown in [45] and [44] also noted a presence of higher strains around these same regions on similarly structured orthogonal weaves.

Acoustic emission (AE) monitoring is a useful method of defining and monitoring the damage developed in composite materials. It works by recording the release of energy, in the form of stress waves, from events within the structure that relate to a redistribution of stress within the material. In composite materials, these energy events can be the result of the development of various damage mechanisms, such as matrix cracking, interfacial debonding, and fibre fracture. In one study, this
technique was used to determine damage thresholds and to examine whether certain AE event parameters could be correlated with various damage types in a carbon fibre 3D orthogonal weave [55]. During quasi-static tensile loading, the accumulation of energy events could be used to determine damage thresholds. The first indicator of damage initiation $\varepsilon_{\text{min}}$ was represented by low energy events, occurring at strains of approximately 0.4% strain for both the warp and weft direction loading. Two further damage thresholds, $\varepsilon_1$ and $\varepsilon_2$, corresponded to the introduction of cumulative higher energy events occurring at strains of approximately 0.62% and 0.72% respectively. Damage development in this material was described in detail in [56]. Initial damage begins in the form of matrix cracks along the edge of transverse tows. This is then followed by the formation of cracks within the transverse tows. Finally, the development of local debonding along various tow interfaces occurs. By failure, a well-developed network of transverse cracks within tows and along tow boundaries has occurred. Cracks within the resin-rich regions were found to be quite sporadic. Using the known damage progression and the AE event information, [55] attempts were made to characterise the frequency range associated with each damage mechanism; a frequency range of 50 kHz to 500 kHz was split down into four smaller ranges as proposed in [57]. However, it was found that the frequency ranges suggested do not provide a clear enough distinction for each damage type, especially with regards to fibre fracture. It was therefore determined that the energy of the AE events is a much better way of defining damage development thresholds.

Figure 2.23: Example of recorded strain mapping data from ESPI on 4 harness 3D orthogonal weave composite; a) strain map and photograph for data correlation; b) line profile taken across a warp binder at different loading stages. Take from the work of Quinn et al (2008) [54]

In summary, the structure of various 3D woven composites appears to influence their respective tensile properties. Large resin-rich regions in these structures appear to be of higher strains than the bulk material as a result of the lower modulus of the resin compared with fibres. This results in a damage in terms of transverse cracking developing along these regions. In addition, the z-binder path has a noticeable effect on the size of the delamination damage, with structures such as 3D orthogonal weaves having short, more constrained delamination growth compared with angle interlock weaves. Z-binder path also influences the mechanical properties, with a more idealised (square-shaped) z-binder enabling
the improvement of tensile results significantly over conventional sinusoidal z-binder paths. This will be due to the increased amount of straight fibre tows.

2.4.5. Characterisation of 3D composites subjected to fatigue loading

Characterisation of the tensile fatigue performance of an E-glass/vinyl ester 3D orthogonal weave was conducted by Carvelli et al. [58] and compared to a 2D plain weave. The 2D and 3D preforms used were identical to those described in studies by Lomov and colleagues [46] [47], and were termed 3D-96 and 2D-24. Tensile fatigue tests were conducted along both the warp and weft direction at a variety of peak stress levels (60 MPa to 350 MPa). The lowest peak stress of 60 MPa was treated as a fatigue limit as many specimens tested had not failed after five million cycles. In this work it was generally found that the weft direction performed better at all stress levels than both the warp direction and the 2D-24 specimens; the S-N curve for each loading direction, as well as the average number of fatigue cycles, along with the standard deviation and covariance for each stress, can be seen in Figure 2.24 and Table 2. For peak stresses 150 MPa to 350 MPa, the slope of the S-N curve for both the warp and weft direction loading, as well as for 2D-24, is practically the same. However, at the lower peak stresses (i.e. < 150 MPa) a difference greater than a factor of two in fatigue life between warp and weft direction occurs. A number of suggestions were made to account for the difference in warp and weft direction fatigue lifetimes, including: (1) weaving damage – warp tows sustain more damage during weaving than weft tows; (2) the addition of resin-rich regions caused by the presence of z-binders increases the local stress concentrations and ultimately reduces a composites resistance to initial damage - there are only small resin-rich pockets between adjacent weft tows compared with the through-thickness resin-rich regions between warp tows; and (3) for warp-direction loading the z-binders have direct in-plane loading, whereas this does not happen for weft-direction loading – it is possible that Poisson’s effects cause a reduction in proximity between warp tows and z-binder, causing cyclic frictional contact and thus reducing fatigue life.

Figure 2.24: S-N curves for E-glass 3D-96 along the a) weft direction and b) warp direction; c) is the S-N curve for a plain weave fabric [58]
Table 2: Number of cycles to failure at various stresses shown for 3D-96 warp and weft, and 2D-24 plain weave composites. Taken from the work of Carvelli et al [58]

<table>
<thead>
<tr>
<th>σ, MPa</th>
<th>3D-Weft</th>
<th>3D-Warp</th>
<th>PW</th>
</tr>
</thead>
<tbody>
<tr>
<td>55</td>
<td>5,000,000</td>
<td>n/a</td>
<td>n/a</td>
</tr>
<tr>
<td>60</td>
<td>5,000,000</td>
<td>n/a</td>
<td>n/a</td>
</tr>
<tr>
<td>70</td>
<td>2,263,028</td>
<td>104,676</td>
<td>4.6%</td>
</tr>
<tr>
<td>80</td>
<td>771,452</td>
<td>184,724</td>
<td>23.9%</td>
</tr>
<tr>
<td>95</td>
<td>192,313</td>
<td>34,368</td>
<td>17.9%</td>
</tr>
<tr>
<td>135</td>
<td>13,595</td>
<td>995</td>
<td>7.3%</td>
</tr>
<tr>
<td>200</td>
<td>3372</td>
<td>118</td>
<td>3.5%</td>
</tr>
<tr>
<td>300</td>
<td>322</td>
<td>6</td>
<td>1.7%</td>
</tr>
<tr>
<td>350</td>
<td>118</td>
<td>13</td>
<td>11.1%</td>
</tr>
</tbody>
</table>

Another study by Karahan et al. investigated the tension-tension fatigue performance of a carbon fibre/ epoxy 3D orthogonal weave [59]. This orthogonal weave had seven layers, four weft tow layers and three warp tow layers. Fatigue loading was conducted along both the warp and weft direction with a frequency of 6 Hz and an R-value equal to 0.1. Data collected from testing at a variety of peak stress levels were plotted as an S-N curve and represented by a tri-linear curve (see Figure 2.25). The tri-linear curve was split into three regions: Region I consists of low cycle fatigue at high stress amplitudes where behaviour is strongly influenced by static strength and non-progressive fibre damage; Region II consists of progressive matrix cracking at intermediate peak fatigue stresses; and finally, Region III has matrix cracking that becomes arrested by fibres at low stresses. Along Region II, the warp and weft direction seem to have the same linear slope, but the fatigue performance of the warp direction was shown to be superior to the weft direction by more than a factor of three. For the same number of cycles to failure, the difference in peak stress for warp and weft direction loading was very similar to the difference in quasi-static strengths. These results directly contrast with those presented by Carvelli et al. [58] where, for an E-glass/ vinyl ester 3D orthogonal weave of similar construction to that used here, the weft direction fatigue performance was noticeably better than the warp direction. However, for the E-glass orthogonal weave, weaving damage and the presence of resin-rich regions were suggested as a reason for the warp-direction performing worse that the weft-direction in fatigue. However, as it was shown in Section 2.4.1, carbon fibres are not as susceptible to weaving damage as glass fibres, and the resin-rich regions probably do not influence the overall performance due to the superiority of carbon fibres compared with glass fibres. Therefore, a knockdown in warp-direction fatigue properties of carbon fibre 3D weaves may be limited compared with glass fibre 3D weaves.
Rudov-Clark and Mouritz studied the effect of z-binder volume fraction on the tensile fatigue properties of an E-glass/vinyl ester 3D orthogonal woven composite [60]. Z-binder volume fractions of 0.3%, 0.5%, and 1.1% were used, with all 3D geometries and compared against a 2D plain weave fabric. With regards to interlaminar fracture toughness it was noted that even a small z-binder volume fraction caused a large increase. Addition of 1.1% z-binder into the laminate improved the interlaminar fracture toughness by approximately 400% in comparison to the plain weave fabric. It has been suggested by the authors that this increase is due to a combination of elastic stretching, crack bridging and frictional resistance against the pull-out of z-binders. Although interlaminar fracture toughness increased, fatigue performance did not. It was clearly shown (see Figure 2.26) that an increased z-binder content caused a decrease in fatigue life. In fact, compared to a 2D plain weave the addition of z-binders caused a reduction of fatigue life. This is in direct contrast to [58] and [61] where improvements in fatigue performance between 3D orthogonal over 2D plain weaves were seen. In [60] it was suggested that this is due to the increase resin rich regions as a result of the inclusion of z-binders. However, it should be noted that the 3D orthogonal layup used between these three papers are different and it is currently unknown how the increase in thickness affects the fatigue properties.

![Figure 2.25: S-N curve for carbon fibre 3D orthogonal weave [59]](image_url)

The damage development during tension fatigue of an E-glass 3D orthogonal weave in a study by Carvelli et al [58] was seen to follow a similar trend to the work described in [46, 47]. At low stresses (55 MPa for warp, and 60 MPa for weft) it was shown that although much of the damage produced in the 3D-96 was similar in both the warp and weft directions, there were still noticeable differences. For instance, for the warp direction loading, transverse cracks grew in both length and width at a faster rate than for the weft loading. Additionally, in the weft direction, large numbers of transverse cracks grew and began to mutually interconnect. At higher stresses (200 MPa) damage initiated much quicker than at the lower stresses, with it noted that crack saturation in the 3D-96 weft direction occurred after only 100 cycles. From this point longitudinal cracks, as well as cracks on the surface of z-binders, were
shown to initiate rapidly. The precursor to failure was observed to be an intensive formation of transverse and longitudinal cracks within the yarns leading to macro-splitting, weft tow fibre breakage, and separation. As with the lower stresses, damage in the 3D-96 warp direction occurred more rapidly than the weft. The precursor to failure in this case was determined to be longitudinal cracks and local debonding of the z-binder from the matrix.

![Figure 2.26: S-N curve comparing a 3D orthogonal weave with increasing z-binder content with a 2D plain weave composite [60]](image)

It was noted by Karahan et al [59] that there is a direct correlation between the second damage threshold (similar in meaning the that presented by Lomov et al previously [55]) measured from acoustic emissions (AE) monitoring during quasi-static loading and the low stresses required for a fatigue limit of three million cycle. Micrographs of quasi-static and fatigue damage at peak fatigue stresses giving a lifetime of over $3 \times 10^6$ cycles show transverse cracks within fibre tows and debonding along various tow interfaces. It was suggested from this correlation that it may be possible to determine a “safe” maximum cycle stress just from monitoring high energy AE events during quasi-static loading, which would result in a large reduction in the amount of fatigue testing required for a given architecture. However, it was noted that more rigorous testing, both quasi-statically using AE monitoring and fatigue testing, would be required for this correlation to be proven conclusively.

Two studies investigated the evolution of damage in 3D woven GFRP composites subjected to tension-tension fatigue utilising XCT as a method of investigating the damage developed [62] [63]. Yu et al. compared the damage in both angle interlock and modified layer-to-layer 3D weave composites [62]. Fatigue loading of these specimens was conducted at 45% UTS (ultimate tensile strength) with an R-value of 0.1 and a frequency of 5Hz. To see damage more readily when scanned used XCT, a zinc iodide dye penetrant was used; it was stated that the increase in contrast provided by the dye penetrant means that damage less than 5% of a voxel size could be seen upon reconstruction of the image slices; a single voxel in this work was 10.7µm. However, the dye penetrant is only useful if the damage within
the composite links to the surface of the specimen, otherwise various regions of damage may not be able to be easily resolved. This will most likely be more useful for assessing the damage developed in a fatigued specimen once large scale damage has been induced; low load, early stage damage may not be observed effectively using this technique. Nonetheless, the various damage mechanisms noted in the XCT reconstructed volume, i.e. large-scale transverse cracking in weft tows and resin-rich regions, as well as debonding and delamination, show good correlation to the damage seen in SEM images. This technique was employed for further investigation in [63] for the modified layer-to-layer 3D weave where XCT scans were taken at various percentages of the fatigue life. The increasing amount of damage over the fatigue life was resolved and to some extent quantified by using image analysis tools to segregate individual damage mechanisms as shown in Figure 2.27.

Figure 2.27: XCT scan of 3D modified layer-to-layer subjected to tension-tension fatigue. Here the scan has been segmented to display the various damage mechanisms that develop during tension-tension fatigue, each highlighted in a difference colour. For reference, B is for binder, W is for weft, and R is for resin. [63]

Dai et al studied the [51], open-hole fatigue performance of two carbon fibre reinforced 3D weaves; an orthogonal (W-1) and angle interlock (W-3) weave identical in structure to the materials used in [44] – see Figure 2.15 for the architectures. Specimens were machined with a large notch (specimen width of 25 mm) of diameter 12.7 mm and tested with a peak fatigue stress of approx. 62% of the ultimate tensile strength in order to induce damage without loading for a large number of cycles. A frequency of 20 Hz was used to reduce testing time and enable loading to be treated as adiabatic; this allows thermal imaging to relate temperature change to stress changes within the specimen. Thermal images were taken after various numbers of fatigue cycles and normalised to the initial images by
plotting the difference in temperature in order to highlight the damage developed; some of these images can be seen in Figure 2.28 for W-1 and W-3 specimens. Both W-1 and W-3 specimens were cycles for approximately 1,000,000 cycles before failure. From surface imaging, it was found that during early loading W-1 and W-3 had similar size damage areas, but by 100,000 cycles the damage growth in W-3 specimens slowed and became practically saturated by 200,000 cycles. W-1 specimens had a much greater number of binding points (locations of z-crowns due to being an orthogonal weave), creating many large resin-rich regions from which matrix cracks developed. In comparison, the resin-rich regions were small around binding points for W-3 specimens, and no cracks were seen around these regions. In W-1 specimens there was clear debonding of portions of warp tows cut by the notch. This debonding was caused by the tensioning of the uncut portion of warp tows around the notch edge. The uncut warp tows around the edge of the notch are place in tension, enabling the development of longitudinal cracks through the warp tows; off these many transverse cracks were noted. Longitudinal cracks were also seen in W-3 specimens, but other damage was not as easily seen as in W-1 specimens.

Figure 2.28: Thermal imaging of notched 3D woven specimens at various fatigue cycle. These have been normalised to an early cycle; a) W-1, 3D orthogonal weave; b) W-3, angle interlock [51]

Another notch fatigue study looked at two carbon fibre reinforced layer-to-layer angle interlock composites each with a different number of layers [64]. The structure of these preforms was such that the warp tows were kept straight and the weft tows were woven through the thickness around them (see
The notch diameter was 4 mm, and the tensile fatigue performance was compared to that of the un-notched specimen. The two fabrics used were termed 3-layer and 5-layer as a result of the number of weft tows present in each cross-section however, from the schematics in Figure 2.29, the use of the terms 3-layer and 5-layer is confusing. The fatigue limit of both the un-notched and notched 3-layer and 5-layer specimens were determined to be 60% and 70% of the ultimate tensile strength, respectively. Beyond the knockdown in tensile properties, these architectures are observed to be notch insensitive; it is interesting to note that this was speculated theoretically in [43]. When normalised to the ultimate tensile strength, the line of best-fit for the notched and un-notched specimens of each material were practically the same. The 5-layer was seen to be the less sensitive to fatigue loading.

Damage in the un-notched and notched specimens was essentially the same and consisted of transverse cracks in the warp tows and matrix, debonding of warp and weft tows, matrix shear cracking, and weft tow fibre failure. However, the damage that developed around the edge of the hole in a notched specimen occurred at a much faster rate than the un-notched specimen as a result of the stress concentration. A schematic of the fatigue damage accumulation process in these specimens can be seen in Figure 2.30, showing that it initiates as transverse cracks in the resin-rich regions developing into the transverse warp tow, followed by debonding along the weft tow/warp interfaces as a result of the transverse cracks, with eventual failure of the weft tows down the plane of a transverse crack.

![Schematic of three-layer interlock woven structure](image1)

![Schematic of five-layer interlock woven structure](image2)

**Figure 2.29: Structure of two layer-to-layer weaves, a) three-layer and b) five-layer. The fabrics terms come from the number of weft tows interlocking the warp tows. [64]**

With regard to fatigue loading in compression, Dadkah et al looked at the performance of various 3D woven composites when fatigue loaded in compression [65]; the 3D weaves used included layer-to-layer, through-thickness angle interlock, and orthogonal. It was found that for each 3D weave tested, final failure was the result of kink band formation across most of the longitudinal warp tows. In
the lead up to failure, some transverse cracking was seen, though to a lesser extent than was observed in quasi-static compression loading. In addition, the delamination damage seen in quasi-static compressive loading was notably absent during fatigue loading of these specimens. For all specimens toward the end of the fatigue life, kink bands developed in individual warp tows. When the hysteresis loops were examined, little change in modulus was noted to occur until the last 20% of fatigue life; this was assumed to be related to the formation of kink bands in the warp tows. It was suggested that the z-binders force the weft tows into the warp tows, causing out-of-plane misalignment that enables buckling, and eventually the development of kink bands. The number of cycles to kink band formation was shown to decrease with increasing misalignment angle. It was speculated by the authors that as kink bands form, the axial stress in the warp tows reduces towards zero, and the warp tows debond from the surrounding material over a characteristic length. Load transfer by shear lag finally restores the axial load back into the warp tow beyond the debonded and failed region. When enough of the warp tows have developed kink bands, the specimen fails completely.

Figure 2.30: Schematic showing the damage developed during fatigue loading of layer-to-layer composites; (1) matrix cracking from surface of specimen; (2) matrix cracks developing into transverse tows; (3) more transverse cracking and start of some debonding; (4) further debonding between weft weavers and warp tows; (5) final fracture through weft weaver tows [64]

There are two studies that have focussed on the bending fatigue performance of 3D woven composites; the first looked at a silicon dioxide fibre layer-to-layer angle interlock [66], and the second studied a hybrid, ultra-thick orthogonal weave [67], where the warp and weft tows used E-glass fibres and the z-binder used aramid fibres. In [66], the specimens were fatigued with a peak stress equivalent to 60% of the ultimate tensile strength, which also corresponded to the fatigue limit of the material. It was noted that there was an initial sharp drop in stiffness, followed by a more gradual loss of stiffness that lasted for most of the fatigue life. Over the last 10% of the fatigue life a more rapid decline in stiffness occurred. A similar trend was seen in [67], but it was noted here that there was a performance difference between the warp and weft loading directions. In this ultra-thick orthogonal woven composite, the weft direction had a better bending fatigue performance. No explanation was provided
for this difference. During bending fatigue, damage initiated in the form of resin cracks along both the tensile and compressive surfaces. Continued loading saw transverse cracking within the transverse fibre tows and resin-rich pockets dominate the compressive surface, while resin/fibre tow debonding and fibre fractures dominated the tensile surface.

From the studies conducted to date it appears that the choice of fibrous material influences the directional tensile fatigue performance, in that the weft direction outperformed the warp direction in a glass fibre 3D orthogonal woven composite, and vice versa for a carbon fibre 3D orthogonal weave. In addition, while increasing the z-binder content in a 3D orthogonal weave increases the fracture toughness, the fatigue performance is degraded. It is assumed that the presence of resin-rich regions within these structures play a crucial role in the fatigue performance of these composites, since increasing z-binder content increases the size of these regions. Interestingly, the tensile fatigue performance of various 3D woven structures generally appears to be notch insensitive with any knockdown in performance found to be directly related to the knockdown in static strength. The damage mechanisms present during tensile fatigue loading appear to be similar for both loading directions, though the extent of these damage mechanisms seems to differ slightly. For instance, longer and quicker growing transverse cracks were observed in warp-direction loading of a carbon fibre/epoxy 3D orthogonal weave, whereas shorter and more numerous transverse cracks that eventually began to interconnect developed in weft-direction loading. XCT has been shown as a powerful tool for analysing the damage developed during fatigue loading; however, a dye penetrant is required in order to highlight the various damage mechanisms and this is only effective if all the damage links up through to the specimen surface. In contrast to failure under tensile fatigue, compression fatigue failure was shown to be mostly the result of kink band formation, with a notable absence of transverse cracking and delamination damage when compared to quasi-static compressive failure. It was reasoned that the tension on the z-binder forced the weft tows into the warp tows, increasing the misalignment angle, and thus facilitate the development of kink band failure.

2.5. Concluding remarks

In this chapter, a review of 3D composite materials subjected to a variety of different loading conditions has been presented. As presented here, there is a relatively large body of work that has been undertaken in the literature for a wide range of different 3D woven architectures subjected to different loading conditions, including: interlaminar fracture toughness, impact resistance, compression, tension, and fatigue. The literature has shown that the loading mechanisms of 3D woven structures can be complex, with small changes to z-binder path greatly influencing the mechanical properties and damage developed. Of particular interest is the fatigue performance of 3D composites, from which one study showed the increase in z-binder content will result in a reduction in the cycles to failure at different peak stresses. In addition, the fatigue performance is usually better along one direction than another. In
an E-glass 3D orthogonal weave the fatigue performance was worse along the warp direction that the weft direction. It was suggested that weaving damage to the warp tows, and the presence of resin-rich regions ultimately resulted in a reduced warp-direction fatigue performance. In contrast, a carbon fibre 3D orthogonal weave with the same structure showed that the warp-direction performed better in fatigue. As mentioned in Section 2.4.1. carbon fibres are less susceptible to weaving damage. Added to this, the superior performance of carbon fibres are probably not influenced by the presence of high-strain resin-rich regions to the same extent as glass-fibre weaves.

While a few studies have investigated the fatigue performance of 3D woven composites, each of these studies has focussed on carbon or glass fibre 3D woven structures. No fatigue work has been focussed on the hybridisation of 3D woven composites, and the potential cost benefits where small amounts of expensive and strong carbon fibres are selectively installed into a cheap and relatively weak glass-fibre 3D weave. The focus of this work will be to initially understand the mechanical performance and damage development in an all-glass 3D orthogonal woven composite subject to quasi-static tension and tension-tension fatigue loading. After this, using selective hybridisation a hybrid glass/carbon 3D orthogonal weave will be manufactured and tested, with comparison made to an all-glass 3D orthogonal weave of the same structure. This will allow a direct comparison to be made, as well as a determination of the influence of selective hybridisation.

The next chapter is related to the experimental methodologies used during this work. After that, Chapters 4-8 will detail the quasi-static tensile and tension-tension fatigue performance, as well as the damage development for three 3D orthogonal weaves of the same structure; two all-glass fibre/epoxy composites, and one selectively hybridised glass/carbon epoxy composite.
Chapter 3

Experimental Methods

3.1. Introduction

In this chapter, the experimental methods used during this work will be described. Three different 3D fabrics were used, each consisting of a similar geometry, but provided by two different suppliers. These fabrics were consolidated with resin using mostly a wet-layup method, though a more industrial process known as Vacuum Assisted Resin Transfer Moulding (VARTM) was also used. Specimens cut from the manufactured laminates were tested in both quasi-static tension and tension-tension fatigue, with images taken during testing for damage analysis. Finally, tested specimens were sectioned and polished at regions of interest, with optical microscopy used to observe damage. Each of the techniques used will be described below.

3.2. Materials

In this work, several different 3D orthogonal woven fabrics [3D-78, 3DMG, 3DMHyb] were used. Each of these consisted of three weft tow layers, two warp tow layers, and a z-binder that interlaces along the warp direction. A 3D representation of this structure can be seen in Figure 3.1, taken from a paper by Lomov et al. [46]. In this schematic, the warp tows are represented by the red tow layers, the weft tows are in blue, and the z-binder in green. Details of each preform used are described below.

![Figure 3.1: Structure of the 3D orthogonal weaves used in this work [46]](image-url)
3.2.1. 3D-78 material

The fibre reinforcement, known as 3D-78 is a commercial 3D orthogonal woven fabric that was manufactured by 3TEX (now TexTech Industries). In this work, the structure is woven using E-glass as the fibre reinforcement. The fabric used in this work is identical to one used and described by Lomov et al. in [46]. Details of this fabric materials and structure are given in Table 3.1. In the table below, areal density refers to fabric mass per unit area. Additionally, “yield” is a U.S. term generally used to indicate how big a fibre tow is, whereas in Europe this is measured using the unit “tex”. The warp end and weft pick density refers to the number of warp or weft tows along each length of a fabric, usually taken from a single layer of fibre tows; however, the number can include all tows vertically as well as horizontally.

Table 3.1: Fabric details of the 3D orthogonal woven structure 3D-78

<table>
<thead>
<tr>
<th>Fabric details</th>
<th>3D-78</th>
</tr>
</thead>
<tbody>
<tr>
<td>Areal density</td>
<td>77.9oz/yard$^2$ (2.64kg/m$^2$)</td>
</tr>
<tr>
<td>Fibre rovings</td>
<td></td>
</tr>
<tr>
<td>Hybon 2022 [E-glass fibre]</td>
<td></td>
</tr>
<tr>
<td>[Yield (yd/lb)]</td>
<td>Warp 218 yield (2275 tex)</td>
</tr>
<tr>
<td></td>
<td>Weft 330 yield (1500 tex)</td>
</tr>
<tr>
<td></td>
<td>Z-binder 1800 yield (276 tex)</td>
</tr>
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<td>Warp end density (ends/cm)</td>
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<tr>
<td>Weft pick density (picks/cm)</td>
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</tr>
<tr>
<td>Z insertion density (ends/cm)</td>
<td>2.75</td>
</tr>
</tbody>
</table>

3.2.2. 3DMG material

The fabric 3DMG (short for 3D Manchester glass) is an E-glass 3D orthogonal weave that was manufactured by the University of Manchester using fibre tows from the PPG Hybon 2002 series. In this fabric, both warp tow layers used were 2400 tex fibre tows, whereas only the central weft tow used 2400 tex fibre tows, and the surface weft tows used 1200 tex fibre tows. Additionally, this fabric was woven with two different z-binder tensions. The original z-binder tension was achieved by attaching weights, equivalent to 80 gf, to the end of each z-binder tow; this fabric was called 3DMG-T1. For the second version of this fabric, the weavers maintained the same structural setup as 3DMG-T1, but increased the weight on each z-binder to 120 gf; this fabric was termed 3DMG-T2. Despite the weave setup remaining the same for both fabrics, it was found that the size of the unit cell reduced slightly along both directions. The warp-direction unit cell was 6.49 ± 0.17 mm in 3DMG-T1 and 6.26 ± 0.15 mm in 3DMG-T2; a similar decrease was noted for the weft direction with the unit cell being 6.51 ± 0.14 mm and 6.17 ± 0.11 mm in 3DMG-T1 and -T2 respectively. Details of this fibre architecture can be seen in Table 3.2 below.
3.2.3. 3DMHyb material

Similar to the 3DMG fabrics described above, 3DMHyb was manufactured by the University of Manchester. The main difference between 3DMHyb and 3DMG is that the PPG Hybon 2002 300 tex E-glass z-binders were replaced with Tenax-E HTS45 E13 200 tex carbon fibre z-binders. The tension placed on the z-binders remained the same as the higher z-binder tension used in the 3DMG-T2 material (i.e. 120 gf). The unit cell dimensions along the warp and weft-direction for 3DMHyb were found to be 6.18 ± 0.04 mm and 6.33 ± 0.09 mm, respectively. Details of the 3DMHyb fibre architecture can be found in Table 3.2.

### Table 3.2: Comparison of 3DMG and 3DMHyb fabric details

<table>
<thead>
<tr>
<th></th>
<th>3DMG</th>
<th>3DMHyb</th>
</tr>
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<tbody>
<tr>
<td></td>
<td>T1</td>
<td>T2</td>
</tr>
<tr>
<td>Fibre rovings (warp and weft)</td>
<td></td>
<td>Hybon 2002</td>
</tr>
<tr>
<td>Fibre rovings (Z-binder)</td>
<td>Hybon 2002</td>
<td>Tenax-E HTS45 E13 [z-binder]</td>
</tr>
<tr>
<td>Warp (tex)</td>
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<td></td>
</tr>
<tr>
<td>Weft (tex)</td>
<td>1200 [surface layers]; 2400 [central layer]</td>
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<td>Z-binder (tex)</td>
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<td>Z-binder tension (gf)</td>
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<tr>
<td>Z insertion density (ends/cm)</td>
<td>3.08</td>
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</table>

3.3. Manufacture

During this work, two different methods of composite manufacture was employed. The first method, used for the majority of testing presented in this thesis, was a wet-layup method, which is relatively easy to implement and generally appropriate for 2D UD and textile composite materials. The second method is known as VARTM (vacuum assisted resin transfer moulding) and is a common method used in industry. VARTM was employed at a later stage in the project because using the wet lay-up technique unexpectedly led to a very small volume fraction of small voids which could not be removed; also, it was valuable to determine whether altering the manufacturing route would produce differences in material characteristics. The resin system used here enabled the production of fairly transparent specimens when combined with glass fibres as a result of similar refractive indices between the resin and the fibres. This was useful for monitoring damage in specimens under various loading conditions.
3.3.1. Wet-layup laminate manufacture

A three-part resin system consisting of Epoxide Resin 300, MNA (Methyl nadic anhydride) hardener, and Ancamine K61B curing agent, was used in this work. Each component was measured by weight into a plastic container in the ratio of 100:60:4. The container was placed inside a warm bath of water so that the viscosity of the resin was lowered, making mixing easier. Mixing of the resin was done within a fume cupboard until homogeneous (no streaks). It would take up to 15 minutes to sufficiently mix the components so that no individual parts were still present, and to avoid the adding excess air into the resin. Once mixed the resin was put into a vacuum oven at 60°C with a pressure of -1000mbar and left for approximately an hour to degas.

Figure 3.2: Some components used in wet-layup manufacture – two glass plates, 3D woven fabric, frame and resin

Two metal plates of dimensions 400 x 400 x 6 mm³ were placed in an oven at approximately 100°C while the resin degassed; these were used during wetting of the fabric preform with resin. While these plates were being heated, a PTFE frame was prepared to house the fabric preform for wetting. The frame had an inner area of about 330 x 330 mm². A silicone coated Melinex sheet, approximately 500 x 500 mm², was attached to one side of the frame via the use of vacuum tacky tape. It is important to ensure that this Melinex sheet is taut and there are no air gaps between the sheet, tape, and frame.

The fabric was cut down to the required size and place on the Melinex sheet within the frame. It can be noted that the 3D-78 fabric preforms were generally cut to a size of 300 x 300 mm². However, for both the 3DMG and 3DMHyb, the width of the fabric provided was limited to just over 200 mm when the edges were trimmed down. Therefore, these fabrics were cut to smaller sizes.

The final stage before fabric wetting was the preparation of two glass plates. These glass plates were used to provide a flat surface for the fabric to rest against. One plate was bigger than 380mm² (the outer frame dimension), while the other glass plate was approximately the same size as the fabric. These plates were waxed using a mould release wax on one surface to make sure any resin that may end up
on the glass plate can be removed easily after curing. The plates used can be seen in Figure 3.2 along with the cut fabric and frame.

![Figure 3.2: Some images at various stages during wet-layup manufacture: a) Melinex sheet attached to a frame with the fabric sitting inside; b) hot metal plate and glass plate in vacuum box; c) pouring resin over the fabric in the vacuum box; d) squeezing out excess resin; e) weights placed on top of fabric during curing.](image)

Once the resin was degassed, each of the prepared components were placed within a vacuum chamber. The component ordering includes: metal plates, large glass plate, frame and fabric. The degassed resin was then poured over the fabric. Sealing the vacuum chamber and turning on the vacuum pump, the fabric was left for 1 - 1 ½ hours in order for the resin to wet the fabric successfully. Once the fabric was removed from the vacuum chamber, another sheet of Melinex was placed over the newly wetted fabric. Using a flat edged tool, excess resin and air bubbles were pushed out to the edge of the frame. A small glass plate was then placed onto the top Melinex sheet, wax face down. Finally, the glass plates and frame were placed into an oven to cure. Weights equal to approximately 91 kg (200 lbs) were placed on the glass plate. Curing takes just over four hours. The cure cycle was as follows:

- Heat to 100°C at a rate of 2.5°C/minute
- Constant heating at 100°C for three hours
- Natural cooling to room temperature

Figure 3.3 shows some images of some of the stages during wet-layup manufacture as described above.

3.3.2. Vacuum Assisted Resin Transfer Moulding (VARTM)

VARTM manufacture is a method that utilises vacuum bagging in the consolidation of composite preforms. It is a cheaper version of resin transfer moulding (RTM) as it only requires a single mould surface and bagging material. It is often used with prepreg material, but can also be used to infuse resin into dry preforms. Unlike RTM manufacture where the thickness of the laminate is dictated by the cavity in the RTM rig, VARTM thicknesses are dependent on the compressibility of a composite preform under vacuum pressure. Infusion is achieved via the drawing of resin from one side of a component to the other. This generally ensures that voids are removed as the resin passes through the structure, guiding any voids to the outlet. It can be noted that voids appeared unavoidable during wet-layup manufacture of 3D woven composites, so VARTM was used to make sure they were removed.

VARTM manufacturing starts with the preparation of two glass plates, one 400 x 400 mm² and the other 250 x 250 mm². Layers of mould release agent were applied to both glass plates. The mould release agent stopped any resin bonding with the glass plates. The release agent had to be applied to all surfaces on the smaller glass plate, including the sides. For the larger glass plate, the mould release was only put on a single surface, and of that surface it was only be applied to the area that was to be bagged.

Once a few layers of mould release had been applied to the glass plates, vacuum bag (tacky) tape was applied around the outside edge of the large glass plate. The dry preform was then cut slightly bigger than 250 x 250 mm² and place into the centre of the glass plate. Two small strips of peel ply and infusion mesh were cut out with a length of at least 250 mm and width of approximately 100 mm. A strip of peel ply was placed along opposite sides of the dry preform, overlapping the preform slightly. The strips of infusion mesh were placed on top of the peel ply strips, and both of these were taped down to the glass plate along each edge. Two lengths of spiral tubing were cut to the same length as the preform, placed about midway on top of the infusion mesh and then stuck down at each end. A resin infusion connector was then placed along the centre of each length of spiral tubing. The small 250 x 250 mm² glass plate was then laid on top of the fabric.
After this had been completed, the vacuum bag was stuck down to the tape around the edge of the base plate. It was important that there was enough slack in the bag for it to form around the small glass plate. This was achieved by adding pleats into the corners of the plate using more tacky tape. Once the bag was sealed a small cut was placed into the bag around each of the resin infusion connectors and the inlet and outlet tubes were attached. Tacky tape was wrapped around the tubing and stuck to the vacuum bag to seal the bag completely.

The outlet tube was attached to a resin catch pot with a vacuum gauge and then clamped shut. The vacuum pump was attached to the catch pot and turned on. Any air within the bag was evacuated and leaks detected. After producing a vacuum of -1 bar, it was useful to remove the vacuum pump and leave the setup for 30 mins or longer in order to see if there any leaks in the bag. Once a sufficient vacuum was maintained resin was infused through the dry preform.

It should be noted that the resin system used for VARTM was the same as the resin system described in Section 3.3.1. After the resin had been mixed and degassed, the fabric was ready to be infused. A heated metal plate was placed under the base plate to help with maintaining a low viscosity flow of the resin during infusion. Additionally, the resin container was placed within a warm bath of water. With the vacuum pump switched on, the inlet tube was placed into the resin allowing the resin to flow through the fabric preform. The flow of resin was maintained until no air bubbles could be seen in the inlet tube. Both inlet and outlet tubes were then clamped shut and any remaining tube cut away. The mould was then placed in an oven and cured using the procedure described in Section 3.3.1 was used. A schematic of the VARTM setup can be seen Figure 3.4. Additionally, a photograph taken during setup can be seen in Figure 3.5.
3.3.3. Specimen preparation – quasi-static and fatigue test coupons

Test coupons were cut from the cured laminate using a water-cooled diamond saw produced by Diamant Boart. The width of coupons along the warp direction were typically cut such that three z-binders were within the width; since adjacent z-binder crowns are situated along the opposite specimen surface, three z-binders were thought to keep the structure balanced from possible twist during loading. This meant that the coupon widths were generally between 10 mm and 13 mm depending on which material was used. Weft direction specimens were a similar width to warp-direction specimens. The length of the specimens were 230 mm for all 3D-78 specimens. By contrast, the length of 3DMG and 3DMHyb specimens were 200 mm because the width of the fabric produced meant only 200 mm lengths could be produced for weft-direction specimens; for consistency, the length of warp direction specimens were kept the same. It can be noted that the width and thickness of each specimen were measured using Vernier callipers, measured at three places along the length of each specimen, with the final value being an average of all three.

All specimens were end tabbed using a GFRP material known commercially as Tufnol. These tabs were approximately 2 mm thick, and had a length of 50 mm. For each of the 3D-78 specimens, the tabs provided a gauge length of 130 mm, while the gauge length was 100 mm for both 3DMG and 3DMHyb specimens. Each end, and both sides, of a specimen was sanded with 320 grit sand paper, while only one side of each end tab was sanded. The end tabs were bonded to each specimen using the
adhesive DP 190, produced by 3M [68]. The adhesive took approximately 24 hours to cure to a tack free state when left at room temperature; full cure took seven days. The specimens were clamped together in a vice-like rig, with sheets of Melinex between them to stop them from sticking together if any excess adhesive were to be expelled when pressure is applied.

3.4. Mechanical Testing

3.4.1. Testing methods

In this work, quasi-static and fatigue tests were carried out. All tests were performed on an Instron 1341 servohydraulic fatigue machine with a load cell rated at 100 kN static and 50 kN dynamic loading, and wedge grips for clamping specimens. Strain was measured using the Instron 2620 602 extensometer with varying gauge length depending on the test. The extensometer has a standard gauge length of 12.5 mm and total travel of +/- 2.5 mm.

The quasi-static tests were all performed in tension with an extension rate of 1 mm/min using the machines standard displacement control. Strain was measured using an extensometer with a gauge length of 12.5 mm. Load, strain and position data were collected by the Instron dataloggger at intervals of 0.05 s.

Tensile fatigue testing was conducted in load control with a stress ratio value, R, equal to 0.1, and a frequency of 5 Hz. Amplitude and mean values for the cyclic waveform used in fatigue testing were calculated using the following standard equations:

\[
Amplitude = \frac{F_{\text{max}} - F_{\text{min}}}{2}
\]

\[
\text{mean} = Amplitude + F_{\text{min}}
\]

Where Amplitude is the force corresponding to half the height of the cyclic waveform used, \(F_{\text{max}}\) is the maximum loaded of the cyclic test, \(F_{\text{min}}\) is the minimum load in cycle test, and the mean is the load half way between \(F_{\text{max}}\) and \(F_{\text{min}}\).

Strain was measured using an extensometer with a gauge length of 96 mm for the 3D-78 specimens, and 75 mm for 3DMG and 3DMHyb specimens. These long gauge-length extensometers were used for enabling the capture of energy dissipation in specimens during fatigue loading and also for the possibility of capturing the development of final failures occurring within the gauge length. Load, position and strain data were collected every 0.002 seconds, equivalent to 100 points per cycle at a frequency of 5 Hz, in order to get a clear representation of a single cycle. Depending on the expected number of cycles to failure, every \(n\)th cycle was collected. In addition, the maximum and minimum points in a cycle were recorded separately.
In order to characterise the damage development, *in-situ* photographs of damage development were taken using a Canon EOS 550D SLR camera during both quasi-static and fatigue loading. The camera was attached to a tripod for consistent imaging of specimens. The photographs were taken at intervals of 5 s and used to monitor damage during tensile loading of specimens.

3.4.2. Data Analysis

For the quasi-static tests, the tensile modulus was calculated from the stress-strain data between the strain limits of 0.1% and 0.3% strain as loading was linear over this range. For the fatigue tests, recording the stress-strain data enabled hysteresis loops to be monitored during the test. A number of parameters were extracted from the stress-strain data of the hysteresis loops. Firstly, the tangent modulus of each hysteresis loop recorded during fatigue loading was calculated by plotting a line of best fit through the linear portion of the initial loading section of the hysteresis, as depicted in Figure 3.6. The minimum and maximum strain in each hysteresis increased with each cycle until failure, and as such the line of best fit used to determine the tangent modulus could not be fixed to a set range of strains like quasi-static loading. The next piece of data extracted from the hysteresis loops was the secant modulus, determined simply by determining the slope between the minimum and maximum point in each hysteresis loop. These generally have a more dramatic change over time as the cycles become more non-linear with increasing damage. Finally, the area of each hysteresis loop was calculated by determining the area under both the stress/strain loading and unloading curves, and subtracting the unloading curve area from the loading curve area (see Figure 3.7). The hysteresis loop area is indicative of energy dissipated during cyclic loading in various forms, most notably by the development of damage. It is calculated from the data collected using the trapezium rule shown in Equation 3; here, stress1, stress2, strain1, and strain2 are sequential data points along the loading and unloading curve.

\[
\text{Area} = \frac{(\text{stress1} + \text{stress2})}{2} \times (\text{strain2} - \text{strain1})
\]

Figure 3.6: A schematic showing two methods of calculating the loss of stiffness per cycle using a tangent to the loading portion of the hysteresis curve and a line between the maximum and minimum points (secant)

Since a large number of cycles were recorded during each fatigue test, a macro was written in MS Excel using VBA (Visual Basic for Applications) code. The macro was written to work with the layout the Instron software Wave Matrix sets up when a .csv file is opened in MS Excel. The macro
was verified by doing the same calculations analytically and comparing with the values produced by the macro. Additionally, depending on the peak stress used, some strain limits within the code must be adjusted manually before the code is run; these limits are based on points within each cycle that are used to calculate the tangent modulus. Finally, when the code was run, the macro required the width and thickness of each specimen to be input so the stress could be calculated from the load cell data. It should be noted that load and strain data are recorded by Wave Matrix with the units of Newtons (N) and micro-strain (µsn), respectively.

Figure 3.7: Schematic depicting the method used to calculate the energy dissipation from cyclic hysteresis

\[
\text{Loading (or unloading)} = \int_{\min}^{\max} \frac{\text{stress}_1 + \text{stress}_2}{2}, (\text{strain}_2 - \text{strain}_1)
\]

Equation 3

3.5. Composite characterisation

Due to the amount of testing required to characterise each of the 3D orthogonal woven composites used in this work, both in terms of tensile mechanical properties and damage development, many laminates were produced. Since many panels were produced, it is useful to characterise differences in quality to aid any comparative analysis. This type of characterisation for composite materials can be achieved in several ways, using both non-destructive examination (NDE) and destructive examination techniques. NDE techniques for this type of characterisation can include, but are not limited to, visual inspection, ultrasonic C-scanning, scanning acoustic microscopy and x-ray computed tomography. Whereas, destructive characterisation can be accomplished using various microscopy techniques, resin burn-off or acid digestion, amongst others. Except for resin burn-off and acid digestion, all the techniques mentioned can be used to assess the structural quality of a composite. Resin burn-off and acid digestion are used to determine the fibre volume content of a composite. In this work, quality control was achieved using visual inspection, optical microscopy, and resin burn-off.
3.5.1. Microscopy

To characterise the damage in greater detail, many of the test specimens were sectioned and polished so that a close examination could be made using optical microscopy. Regions of interest were cut from specimens, having a length no more than 20 mm since they needed to be smaller than the cast size of 30 mm, with excess room. The samples were then embedded in an epoxy resin in a cylindrical cast with a diameter of 30 mm. Sample cross-sections along the loading direction and perpendicular to the loading direction were embedded in order to assess damage along both directions after testing. The resin used was a two-part epoxy provided by Struers, called EpoFix; this requires a ratio of 15 parts epoxy to 2 parts hardener measured by volume, and cures in 8-12 hours at room temperature.

Once the epoxy mounts were cured, the samples were ground and polished using the test procedure shown in Table 3.3. Between each stage, the samples were cleaned with cold water and left to dry in front of a small fan heater. Due to the transparency of the specimens, regions of interest could be determined and the distance from the edge of the specimen noted before the sectioned specimens were embedded in resin. During grinding, Vernier callipers were used to determine the approximate amount of material to be removed, so it was possible to polish down precisely to the region of interest. After polishing and grinding, samples were observed using a Zeiss optical microscope.

<table>
<thead>
<tr>
<th>Table 3.3: Specimen grinding and polishing procedure for GFRP composites</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grinding Stage</td>
</tr>
<tr>
<td>Cloth/Paper</td>
</tr>
<tr>
<td>Grit size</td>
</tr>
<tr>
<td>Lubricant</td>
</tr>
<tr>
<td>Speed (rpm)</td>
</tr>
<tr>
<td>Force (N)</td>
</tr>
<tr>
<td>Time (s)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Polishing Stage</th>
<th>1</th>
<th>2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cloth/Paper</td>
<td>MD Dur</td>
<td></td>
</tr>
<tr>
<td>Grit size</td>
<td>3µ</td>
<td>1µ</td>
</tr>
<tr>
<td>Lubricant</td>
<td>Blue</td>
<td></td>
</tr>
<tr>
<td>Speed (rpm)</td>
<td>150</td>
<td></td>
</tr>
<tr>
<td>Force (N)</td>
<td>90</td>
<td></td>
</tr>
<tr>
<td>Time (s)</td>
<td>300</td>
<td>300</td>
</tr>
</tbody>
</table>

3.5.2. Fibre volume fraction measurements

For glass-fibre based composites, the typical method of determining the fibre volume fraction is to use resin burn-off. This method works on the principle of burning off the resin surrounding the
glass fibres, weighing the material before and after burn-off in order to determine the weight and volume of each constituent component. At least six resin burn-off tests are conducted per laminate produced in order to determine a fibre volume fraction.

Small specimens, usually weighing less than 1 gram, were cut from each of the laminates produced. Each of these specimens were weighed to 0.001 g. Additionally, six ceramic crucibles were weighed, and each of the specimens were then placed within them. Each crucible was placed into an oven that was then heated to a temperature of 600°C for 6 hours, and then allowed to cool naturally down to room temperature over a number of hours. With the resin now burnt off the composite, the glass fibres were reweighed within the crucible. Subtracting the mass of the crucible from the final mass measured provided the mass of glass fibres present in the specimen. Further, subtracting the mass of glass fibres from the initial mass provided the mass of the resin. With the density of the fibres and resin being 2.54 g/cm$^3$ and 1.2 g/cm$^3$ respectively, these masses could be converted into volumes and the total fibre volume fraction calculated.

For determination of the fibre volume fraction of the hybrid 3D orthogonal weave (3DHyb), resin burn-off was also used. However, it is generally suggested that resin burn-off is not used with carbon fibres as they can degrade with over time when exposed to the typical temperatures used in burn-off testing. However, work by McDonough et al. [69] suggested that resin burn-off may be used on carbon/glass hybrid composites by adjusting the temperature used and the exposure time. For this method two temperature cycles were used, with weighing of the crucible and specimen occurring between each stage. The initial burn-off was done at temperature of 600°C, but was held for only 30 minutes as compared to the 6 hours previously. The crucible and specimen were then air cooled and weighed; this stage was considered long enough to burn off the resin component. The next stage involved the burn-off of the carbon fibres, utilising a temperature of 900°C for a further 30 minutes; again, these were air cooled and weighed. Then, using the same calculations as previous for plain GFRP composites, but extended to include carbon fibres (with density 1.77 g/cm$^3$), the individual fibre volume fractions for both glass and carbon components were calculated. In the work of McDonough et al. [69] the mass loss of carbon that occurred when this procedure was followed was small and often very similar to the expected mass loss according to TGA (Thermogravimetric analysis). Therefore, it was concluded that this method would be suitable for the hybrid composites used in this work.

3.6. Concluding remarks

In this chapter, details of the 3D orthogonal woven fabrics [3D-78, 3DMG, 3DMHyb] used in this work were provided. In addition, the two manufacturing methods used for composite consolidation, the mechanical testing procedures, the cross-sectional polishing method, and the data analysis techniques used were described. Apart from the weaving of the various fabrics used, all other procedures described in this chapter were performed by the author, with attempts made to reduce variability in all
the experimental work carried out. However, this was challenging, especially in areas such as composite manufacture, where the use of wet-layup technique resulted in voids within the composite structures test. Compared with previous studies, this work attempts to enhance the understanding of how 3D orthogonal woven composites perform under load, especially with regards to tensile fatigue loading. This is achieved by close inspection of the damage developed during cyclic loading, as well as analysis of the data with regards to loss of stiffness and energy dissipated per cycle.
Chapter 4

Mechanical properties of the glass fibre non-crimp
3D orthogonal woven composite – 3D-78

4.1. Introduction

In this chapter, the mechanical performance of a glass fibre 3D non-crimp orthogonal woven (3DNCOW) composite will be evaluated. The 3DNCOW material used in this work was manufactured by 3TEX and is termed 3D-78 due to its areal density of 78 oz/yd² (2.64 kg/m²). It has three weft tow layers, two warp tow layers, and z-binders that interlace orthogonally through the thickness along the warp direction; some basic detail of the composite architecture were provided in the previous chapter. Mechanical performance of this material is evaluated from quasi-static tensile and tension-tension fatigue testing of coupon specimens. It can be noted that quasi-static tensile testing of this material has been conducted in the literature [46]. However, no evaluation of the tensile fatigue properties have been made.

4.2. Quasi-static tensile mechanical properties of 3D-78

In order to determine the mechanical properties of the 3DNCOW 3D-78 composite, quasi-static tensile tests were conducted in both principal directions (warp and weft). Six tests were carried out along the warp direction and eight tests along the weft with the main properties summarised in Table 4.1.

When comparing the warp and weft directions, it can be seen that the properties in both directions are essentially the same, though the modulus in the weft direction is slightly lower than that measured for the warp direction. These results seem reasonable since the fibre volume content is very similar in both directions; of the total fibre volume, the warp occupied 47.6%, 47.8% for the weft, and the remainder was the z-binder. Comparing these results with those in [46], it is found that the modulus and ultimate strengths reported here are higher by approximately 10% and 20%, respectively. The ultimate strains measured in [46] for the warp and weft direction were 2.96 ± 0.51 % and 3.14 ± 0.44%, which are slightly higher than those measured here, but still within statistical scatter. The differences may be the result of the different resin matrix and manufacturing method (a vinyl ester resin matrix and VARTM were used in [46]).
Table 4.1: Quasi-static tensile mechanical properties for 3D-78; includes total fibre volume fraction (Vf)

<table>
<thead>
<tr>
<th></th>
<th>E (GPa)</th>
<th>σ_{ULT} (MPa)</th>
<th>ε_{MAX} (%)</th>
<th>Vf (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Warp</td>
<td>26.1 ± 0.7</td>
<td>519 ± 33</td>
<td>2.8 ± 0.2</td>
<td>47 ± 2</td>
</tr>
<tr>
<td>Weft</td>
<td>25.3 ± 0.7</td>
<td>514 ± 28</td>
<td>2.8 ± 0.3</td>
<td></td>
</tr>
</tbody>
</table>

Figure 4.1 and Figure 4.2 show the stress-strain plots for each of the warp and weft-direction loaded quasi-static tensile tests conducted on the 3D-78 material. It can be seen here that there is good repeatability between each of the warp-direction tests conducted. A similar level of repeatability is also true for each of the weft-direction tests, though there is a slight divergence of the curves beyond a strain of approximately 1% (see Figure 2). For both loading directions it is clear that the curves are non-linear, generally following the same trend up to failure. From the general trend in each loading direction, each curve appears to become non-linear when loading has reached approximately 0.4% strain. The initiation of non-linearity in each curve during the early loading stage is possibly related to the development of permanent damage to each specimen. Damage observations (see Chapter 5) during loading have shown that there is generally little to no damage in the forms of matrix cracking by 0.4% strain for these specimens. From acoustic emission measurements [46, 47] found a minimum damage threshold at strains slightly lower than this (0.37% and 0.27% for warp and weft-direction), observing only very small matrix cracks of limited size. In addition, it was shown in [54] that at low bulk strains of 0.29%, local strains of more than 1% were measured in the resin-rich regions of a 3D orthogonal weave. Since there are many resin-rich regions with this 3D woven structure, it is possible that plastic deformation of these regions due to the increased local strains initiates the observed non-linearity.

Around 0.6-0.8% strain, the curves presented in both Figure 4.1 and Figure 4.2 begin to show step changes in measured strain. The step changes in strain can either appear as an increase or decrease in strain, and such changes occur throughout the remaining loading of the specimens. These changes in strain are the result of damage development in the form of matrix cracking causing localised asymmetric bending of each specimen. In 3D woven structures each layer alternates in orientation by 90°. For a warp-direction loaded specimen the layers follow the pattern 90/0/90/0/90. Due to the separation of layers, transverse matrix cracks can develop in each layer individually and not necessarily through the thickness. Due to the presence of resin-rich regions and pockets, crack growth is complex and transverse cracks that develop in one of the surface weft tows and do not necessarily propagate through the thickness of the specimen. However, the crack opening on one surface causes an increase in the strain along that surface more than the opposite surface; seen as an apparent increase in strain for little, or no, change in load. This type of jump in strain is usually seen in the loading of specimens under load control, though all static specimens in this work were tested under displacement control. In addition, it can be noted that these changes in strain were only observed in the strain data recorded directly from the extensometer, and are not present in the displacement data from the servo-hydraulic testing machine.
Since there are 90° layers at the surface of the warp-direction loaded specimens, the rapid changes in strain are more noticeable during warp-direction loading than weft-direction loading. The rapid changes in strain can still be seen in weft-direction loaded specimen, but these changes are smaller since there are no transverse (90°) layers at the surface of these specimens, and the majority of transverse cracks do not reach the surface of the specimen.

Figure 4.1: Stress strain response of multiple 3D-78 specimens loaded along the warp-direction

Figure 4.2: Stress strain response of multiple 3D-78 specimens loaded along the weft-direction
4.3. Tension-tension fatigue properties of 3D-78

To characterise the fatigue performance of the glass-fibre 3D-78 material, a number of tests were conducted for each loading direction. Testing was mostly conducted using five peak stress levels ranging from 175 MPa to 350 MPa, representing 0.33-0.68% of the ultimate tensile strength. Figure 4.3 shows an S-N curve comparison of the warp and weft loading direction. While the mechanical properties are similar for both loading directions when loaded quasi-statically, in tension-tension fatigue the warp direction clearly performs better. At the higher peak stresses, the number of cycles required to fail a warp-direction loaded specimen is approximately two times higher than a weft-direction loaded specimen. As the peak stress is reduced, the average numbers of cycles to failure for the warp and weft directions appear to diverge; at a peak stress of 175 MPa, the number of cycles to failure for a warp-direction loaded specimen is approximately three times that of a weft-direction loaded specimen. As the peak stress is reduced the scatter in the number of cycles to failure increases, and some overlap in the warp and weft scatter can be seen. Since the fibre volume fractions for each of the principal directions is approximately equal, it is believed that the increase in warp-direction fatigue life compared to the weft-direction is related to the influence of the z-binder on the damage developed during loading; further discussion of the damage developed during fatigue loading is in Chapter 5.

Figure 4.3: S-N curve comparison for warp and weft-direction specimens loaded in tension-tension fatigue. There is a singular point at 100 MPa that has an arrow to indicate this is a specimen that did not fail before the test was stopped – also known as a run-out specimen.
For each specimen fatigue loaded with a peak stress of 175 MPa or high, easily observable damage began to develop during the first fatigue cycle. At these stress levels, the initial loading is similar to quasi-static loading past the onset of matric cracking damage. A single warp-direction loaded specimen was loaded with a peak stress of 100 MPa, and was stopped after 1.8 million cycles. It seems possible that the fatigue limit for this material is somewhere around this peak stress, although after 1.8 million cycles, there was quite extensive damage to the specimen (i.e. large-scale matrix cracking, delamination damage and interfacial debonding). While only one warp-direction 100 MPa peak stress fatigue test was loaded to over a million cycles, many warp and weft direction specimens were loaded at 100 MPa to a relatively low number of cycles in order to evaluate the early stage damage development in these specimens. Chapter 5 contains a description of the damage developed during the fatigue loading of the 3D-78 material.

Figure 4.4: Image showing a representation of secant and tangent modulus calculation and change over time

In this work, the load and strain data collected during fatigue loading was used to determine the loss in tensile modulus and energy dissipated per cycle. Two methods were used to calculate the loss of tensile modulus, as shown in Figure 4.4. For the first method, a tangent line was drawn along a linear portion of the initial loading during each cycle, while the second method measured the change in slope between the maximum and minimum points (secant line) of each cycle; the stiffness measurements taken using each method are called tangent stiffness and secant stiffness. Of these two methods, the secant modulus shows the largest changes in stiffness during fatigue cycling. With the development of damage within a specimen, there is a corresponding increase in length, which leaves the specimen permanently deformed upon unloading. During load controlled fatigue cycling, the increasing amount of damage produces an increase in both the maximum and minimum strains; an example from a warp-direction loaded 175MPa peak stress test can be seen in Figure 4.5. However, the maximum strain was found to increase at a much faster rate than the minimum strain, while still maintaining the same loads. This is because the loading and unloading stages have become non-linear. By measuring the slope between the maximum and minimum points in this hysteresis (secant modulus), the tensile modulus changes quite significantly as a result of the damage developed. In contrast, the slope measured along
the first part of the loading stage (tangent modulus) is affected by the non-linearity to a lesser extent and the changes are smaller. Both measurements have been used in this work.

![Figure 4.5: Example of changes in hysteresis area at various cycles throughout the fatigue life of a specimen fatigued with a peak stress of 175 MPa along the warp-direction](image)

Figure 4.6 and Figure 4.7 show examples of the loss of tangent and secant modulus, normalised by the initial tangent and secant modulus, per cycle for specimen’s fatigue loaded with a peak stress of 175 MPa along the warp and weft direction respectively; the energy dissipated per cycle is discussed below. While the tangent and secant modulus in the first two stages of the fatigue life are not quite the same, the trends are very similar. However, during the third stage, where the specimen is approaching final failure, the secant modulus can be seen to be more affected by any damage developed during this stage. It should be noted that the larger loss of stiffness measured by the secant modulus during stage three is only possible if final failure, and the damage leading to it, occur within the gauge length of the extensometer used.

In Figure 4.8 and Figure 4.9, the normalised tangent modulus for all specimens tested at different peak stresses are plotted against normalised cycle number (i.e. cycle number divided by the number of cycles to failure). This is useful for comparing the reduction of modulus over the specimen lifetimes for different peak stresses, as well as comparing scatter between specimens loaded to the same peak stress. For both the warp and weft direction loaded specimens, the loss of tensile modulus for test specimens fatigue loaded with a peak stress of 250 MPa or higher was quite consistent, whereas the scatter was much greater for those fatigue loaded with a peak stress of 175 MPa and 200 MPa.
Figure 4.6: Example of measurements made using the data collected during fatigue loading of a warp-direction specimen with a peak stress of 175 MPa. Included here is the reduction of secant and tangent stiffness normalised by the initial secant and tangent stiffness, as well as the amount of energy dissipated per cycle.

For the warp direction loaded specimens in Figure 4.8, the average loss of tensile modulus up to specimen failure can be seen to reduce with increasing peak stress (see Table 4.2). For specimens loaded with a peak stress of 175 MPa the average loss of tensile modulus by failure is approximately 26%, while there is only a 18% loss of tensile modulus for specimens loaded with a peak stress of 350 MPa. This suggests that by failure there is more extensive damage in specimens fatigued with a lower peak stress. At higher peak stresses, the build-up of stress concentrations leading to fibre failure occurs more quickly, thus limiting the development of other damage mechanisms.

To a lesser extent a similar trend can be seen in Figure 4.9 for weft-direction fatigue loaded specimens. In Table 4.2, the average loss of stiffness at failure for the weft-direction specimens fatigue loaded with a peak stress of 175 MPa is approximately 24%, compared with the 26% loss for warp-direction specimens. However, it appears that for all other peak stresses used, the average loss of modulus is consistently 20%. This indicates that the overall damage before failure is greater in warp specimens when fatigued at low loads. Since the weft-direction specimens generally fail earlier than the warp-direction specimens, the increased total loss of stiffness must be related to a more extensive development of damage, such as delaminations, that blunt stress concentrations and enable the specimen to be fatigued for longer before failure; a detailed discussion of the damage development is discussed in the next chapter.
Figure 4.7: Example of measurements made using the data collected during fatigue loading of a weft-direction specimen with a peak stress of 175 MPa. Included here is the reduction of secant and tangent stiffness normalised by the initial secant and tangent stiffness, as well as the amount of energy dissipated per cycle.

Another method of analysing the fatigue data is the energy dissipated per cycle. This is done by determining the area within the hysteresis produced during each loading and unloading cycle. When damage is developed within a specimen, the loading and unloading becomes inelastic due to permanent deformation of a specimen. Plotting the loading and unloading data as stress against strain, hysteresis loops such as those in Figure 4.5 are produced; the area of the hysteresis loop represents the energy dissipated in various forms, such as heat and friction. In Figure 4.5 it can be seen that the area of the hysteresis loops changes at different cycles. The initially large hysteresis is indicative of the relatively large damage input into the specimen during early loading. As the damage begins to saturate within a specimen, the energy dissipated per cycle reduces as indicated by the reduced hysteresis area. The energy dissipated per cycle eventually increases very slowly, a stage which lasts for the majority of the fatigue life. During the final stage, damage leading to failure causes the energy dissipated to begin increasing rapidly again. The three stages described above are clearly demonstrated in both Figure 4.6 and Figure 4.7, where; stage 1 has a rapid drop in the energy dissipated lasting for a very small number of cycles compared to the overall number of cycles to failure; stage 2 is a shallow incline of increasing energy dissipated lasting for the majority of the fatigue life; and stage 3, beginning after approximately 80% of the fatigue life, has a more rapid increase in the energy dissipated leading to failure.
Figure 4.8: Normalised loss of tensile modulus against normalised cycle number for warp-direction specimens fatigue loaded with different peak stresses. The tangent modulus was normalised to the initial tangent modulus, while the cycle number was normalised by the number of cycles to failure.

It is interesting to note that the energy dissipation per cycle trend during the final stage of loading in Figure 4.6 and Figure 4.7 has the opposite trend to the secant stiffness reduction. Again, just like the secant modulus, the third stage of the energy dissipated per cycle curve can only be captured if failure, or damage leading to failure, occurs within the gauge length of the extensometer.

The energy dissipation per cycle curve in Figure 4.10 is for a specimen fatigue loaded along the warp-direction with a peak stress of 100 MPa, stopped after approximately 110,000 cycles. In this specimen, visible damage did not initiate for the first few thousand cycles, and upon initiation grew slowly. Unlike the 175 MPa peak stress tests there is an initial increase in energy dissipated per cycle for approximately 10,000 cycles before the energy dissipated per cycle began to reduce in magnitude. The increase in energy dissipation may possibly be related to the viscoelasticity of the matrix and heat
build-up through cycling due to the fatigue frequency used. Some damage, in the form of transverse matrix cracks, did develop within the first 10,000 cycles, during which time the energy dissipated per cycle can be seen to increase. It can be noted that the growth of these transverse matrix cracks was slow. Unlike at high stresses where damage developed toward saturation continually reduces the energy dissipated per cycle, the damage developed when fatigued at low peak stresses does not seem to impact the energy dissipated the same way. The damage observed pre-10,000 cycles will be responsible for the loss of tensile modulus seen in Figure 4.10; after 10,000 cycles, the modulus has decreased by approximately 3%.

Figure 4.9: Normalised loss of tensile modulus against normalised cycle number for weft-direction specimens fatigue loaded with different peak stresses. The tangent modulus was normalised to the initial tangent modulus, while the cycle number was normalised by the number of cycles to failure.

Figure 4.11 and Figure 4.12 show the energy dissipated per normalised cycle for each of the peak stress tests in the warp and weft-direction loading respectively. From these it can be seen that; (1)
for higher peak stresses, more energy is dissipated; (2) the general trend of each curve is similar; and, (3) the energy dissipated per cycle at the end of stage two (i.e. after approximately 80% of the fatigue life) is on average slightly lower for warp-direction specimens at all peak stresses with the exception of 350 MPa (see Table 4.3). The increased average energy dissipation in weft-direction specimens appears to correlate well with the lower number of cycles to failure observed when compared with warp-direction specimens.

Table 4.2: Average loss of tensile modulus before failure for warp and weft-direction specimens fatigue loaded at several peak stresses. These numbers are recorded as normalised tangent modulus, with the percentage loss of stiffness in brackets.

<table>
<thead>
<tr>
<th>Peak Stress (MPa)</th>
<th>Average loss of tangent modulus</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Warp</td>
</tr>
<tr>
<td>175</td>
<td>0.74 (26%)</td>
</tr>
<tr>
<td>200</td>
<td>0.76 (24%)</td>
</tr>
<tr>
<td>250</td>
<td>0.78 (26%)</td>
</tr>
<tr>
<td>300</td>
<td>0.80 (20%)</td>
</tr>
<tr>
<td>350</td>
<td>0.82 (18%)</td>
</tr>
</tbody>
</table>

Figure 4.10: Example of measurements made using the data collected during fatigue loading of a warp-direction specimen with a peak stress of 100 MPa. Included here is the reduction of secant and tangent stiffness normalised by the initial secant and tangent stiffness, as well as the amount of energy dissipated per cycle.
Figure 4.11: Comparison of energy dissipation per cycle number (normalised by the number of cycles to failure) for warp-direction specimens fatigue loaded at different peak stresses

Figure 4.12: Comparison of energy dissipation per cycle number (normalised by the number of cycles to failure) for weft-direction specimens fatigue loaded at different peak stresses

It was mentioned above that the reduction in stiffness between the warp and weft-direction loaded specimens was similar, with the exception of the lower stress levels where the warp had a greater
loss of stiffness and higher number of cycles to failure. It was suggested that this was related to the
development of damage such as delaminations, which are shown later to be more prevalent in the warp-
direction loaded specimens than weft-direction specimens (see chapter 5). With warp-direction
specimens generally lasting longer under cyclic loading than weft-direction specimens, this indicates
that delaminations may play a major role in fatigue life extension. This seems counter intuitive since
the purpose of the z-binder is to reduce/ eliminate interlaminar fracture (delamination). However, it
appears in this case that the growth of delaminations may be beneficial to the fatigue life of a specimen.

Table 4.3: Approximate average energy dissipated after 80% of fatigue life for warp and weft-direction
specimens fatigue loaded at several peak stresses.

<table>
<thead>
<tr>
<th>Peak Stress (MPa)</th>
<th>Approx. average energy dissipated after 80% of fatigue life (J/m$^3$)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Warp</td>
</tr>
<tr>
<td>175</td>
<td>7,000</td>
</tr>
<tr>
<td>200</td>
<td>8,000</td>
</tr>
<tr>
<td>250</td>
<td>18,000</td>
</tr>
<tr>
<td>300</td>
<td>28,000</td>
</tr>
<tr>
<td>350</td>
<td>50,000</td>
</tr>
</tbody>
</table>

4.4. Concluding remarks

From the work presented in this chapter, a number of key conclusions can be made. For
instance, under quasi-static tensile loading conditions, the quasi-static tensile mechanical properties of
the warp and weft direction are very similar. This is probably related to the practically equal fibre
volume content in each of the principal directions. Under these conditions the orthogonally bound z-
binder may not contribute much to the static loading performance of the warp direction, thus the
performance in each direction is equal.

In tension-tension fatigue, the warp direction performs better than the weft direction. It was
shown that the average loss of tensile modulus before failure at different peak stresses was greater in
warp-direction specimens. This indicates that the overall damage accumulation is probably greater in
warp-direction specimens prior to failure. One failure mechanism that is more prevalent in warp-
direction specimens is delamination damage (see Chapter 5) and may be responsible for this difference
in measured loss of tensile stiffness. However, the average energy dissipated per cycle for each peak
stress was generally higher in weft-direction specimens, which correlates well with the lower number
of cycles to failure for these specimens compared to the warp-direction. As it will be suggested in
Chapter 5, the development of delamination damage likely blunts stress concentrations that may
otherwise initiate fibre fracture, and thus enables warp-direction specimens to be fatigued longer.
Chapter 5

Development of damage in 3D-78 GFRP

5.1. Introduction

In this chapter, a 3D non-crimp orthogonal woven (3DNCOW) composite is characterised with regards to the damage developed during various loadings. The 3DNCOW discussed here was produced by the company 3TEX under their 3WEAVE trademark and is designated 3D-78. All specimens discussed in this chapter were loaded in either quasi-static tension or tension-tension fatigue, with the details of each loading case reported in Chapter 3. Both principle directions (warp and weft) are characterised here to better understand how damage influences the mechanical properties presented in the previous chapter.

5.2. 3TEX fibre architecture

Before specimens are tested under various forms of loading, it is often useful to inspect the fibre architecture of the fabric preform to get a better understanding of its structure. As it was shown in the previous chapter, the 3D orthogonal woven fabrics used in this project have three weft tow layers, two warp tow layers, and through-thickness orthogonally woven z-binders which run parallel to the warp direction (Figure 5.1a). Both images in Figure 5.1 show idealised schematics of a 3DNCOW, with each highlighting the representative unit cell of the material. The unit cell along both the warp and weft directions in 3D-78 material has been measured to be approximately 7.2 mm.

![Idealised unit cell schematics of the 3D non-crimp orthogonal woven “3D-78” composite: a) 3D unit cell, showing three weft tow layers (blue), two warp tow layers (red) and through-thickness z-binders (green); b) surface view schematic indicating the dimensions of the unit cell of the 3D orthogonal woven material, known as 3D-78, used in this work.](image-url)
For characterisation of the 3D woven architecture used in this project, there are three cross-sectional planes (Figure 5.1a) that are useful to examine. It should be noted in this work that the x-direction is represented by the presence of warp tows extending across the length of a cross-sectional image, whilst for the y-direction these would be the weft tows. The z-direction corresponds to through-the-thickness. Cross-sectional examples of each of these planes can be seen in Figure 5.2; there are two x-z cross-sections, one through warp tows (Figure 5.2a) and the other through a z-binder (Figure 5.2b), and one y-z cross-section that goes through the weft tows (Figure 5.2c).

Several significant features can be observed in Figure 5.2. In Figure 5.2a, all three weft tows and the two warp tows can be observed. While this is a non-crimp fabric, there is still a small amount of curvature present in the warp tows. Figure 5.2c shows a cross-section along the weft-direction, and in a similar manner to the warp-direction, the weft tows have some slight curvature. The curvature present in both the warp and weft tows can be explained by considering the relative position of cross-sections with respect to the location of the z-binders. As it can be seen in both Figure 5.2b and Figure 5.2c, the only component crossing the path of the z-binders is the weft tows; the absence of warp tows in the dry preform results in an empty pocket between the stacked weft tows. During weaving, a tension is applied to the z-binder tows causing the surface weft tows to deflect into these empty pockets. The location of the cross-section of Figure 5.2a is indicated by the dotted red line in Figure 5.2c. Close examination of Figure 5.2c shows that when the weft tow begins to curve below the z-binder, a resin pocket forms between the surface of the specimen and weft tow. As there is no z-binder at the opposite surface in this region, all fabric components below this surface weft are pushed toward the opposite surface. This effect can be noted at each location of a z-binder in Figure 5.2c. Thus, the warp tows are forced to follow the path provided by the weft tows and do not lie straight, rather they have a twisted pathway along the length of the fabric. This distortion of the warp tows, due to proximity to the z-binder can be seen clearly in the cross-section of Figure 5.2a. An x-z cross-section taken along the centre of the warps tows should appear to follow a straighter path since there would be less distortion along this plane.

Another feature of this fabric architecture is the sinusoidal path of the z-binders (see Figure 5.2b). For an orthogonal woven 3D composite the ideal z-binder path through the thickness is vertical, though in practice a slight angle is usually observed. However, in this fabric the angle of the z-binder when it traverses though the thickness is quite large. The reasons for this binder pathway are most likely three-fold: (1) the tension applied to the z-binder tows during weaving is likely to be quite high; (2) the distance between neighbouring stacks of weft tows, or the number of ends/cm, is large enough that when tension is applied to the z-binder tows it easily nestsles into the sinusoidal shape; and (3) the number of weft and warp tow layers is low i.e. the fabric is thin (increasing the number of layers would result in the binder tows to follow a more vertical path through the thickness).
A final feature of the fabric architecture which is worth noting is the difference in size, shape, and stacking of the weft tows. As indicated in Chapter 3, each weft tow is of the same size with regards to its tex. From such data, without looking at a cross-section, it could be assumed that the size and shape of the weft tows within the structure of the fabric would appear similar. However, as it can be noted from both Figure 5.2a and Figure 5.2b, only the size and shape of the weft tows at each surface appear similar, whilst the central weft tow is much wider and thinner. The semi-circular cross-section of the surface weft tows are likely the result of the interaction with the z-binder as it passes over them. Finally, the stacking of the weft tows does not follow an idealised layup. From Figure 5.2a and Figure 5.2b single columns or stacks of weft tows appear to lean, with each adjacent column leaning a different way. Again, this is most likely due to the interaction between the z-binder and the weft tows.

![Figure 5.2: 3TEX 3D-78 GFRP architecture showing: a) x-z plane through the warp tows; b) x-z plane through a z-binder; c) y-z plane through the weft tows. Note: Dark lines around the tows are the result of relief produced during polishing.](image)

5.3. Quasi-static tensile damage development

The transparency of the manufactured composites enables damage accumulation to be monitored during testing and photography of specimens in-situ allowed for damage development to be characterised. Figure 5.3 and Figure 5.4 show the development of damage during quasi-static tension testing in warp and weft-direction loaded specimens, respectively. Both Figure 5.3a and Figure 5.4a are the specimens in their unloaded and untested form, whereby the locations of the warp/weft tows, as well
as the z-binders have been indicated. It can be noted that each specimen has several voids as result of the wet lay-up manufacturing method.

5.3.1. Warp-direction

At around 0.69% strain (or 168MPa) there is no visible damage (see Figure 5.3b). However, when compared to the specimen in its unloaded state (Figure 5.3a), regions of the specimen become darker. The darkened regions of the specimen correspond to the location of the weft tows, whereas the clear regions indicate the resin-rich channels between adjacent weft tows. As shown in [70], fibres oriented in the transverse direction can become partially debonded from their surrounding matrix at relatively low strains. Thus, as light travels through these specimens it can be refracted at these interfaces, reducing transparency and making the weft tow regions appear visibly darker. Since the resin-rich channels between adjacent weft tows are free of transverse fibres, straining this area will have negligible effect on its colouration.

The initiation of significant damage during warp-direction loading appears to begin in two locations. As shown in Figure 5.3c, the first significant damage is a transverse matrix crack located within a weft tow. The transverse matrix cracks appear to initiate preferentially both from the edges of the specimen and along the centre of the tow, extending toward the centre of the specimen (see Figure 5.3d). As shown by Parvizi et al [71], a thicker transverse ply in an cross-ply laminate results in a lower crack initiation strain. Given that the surface weft tows have their thickest cross-section toward their centre, it is therefore not surprising that crack initiation occurs initially at the centre of the tow, though at higher strains multiple cracks can form across the width of the weft tows, as indicated in Figure 5.3e. From the in-situ photography, the initiation of the transverse weft tow cracks occurred at a strain of approximately 0.6%.

The second damage location, as shown in Figure 5.3c, is in the resin-rich region where the z-binder traverses between neighbouring weft tows. The damage in this region is a combination of two damage types: a resin crack and a debonding of the z-binder from the surrounding material. Many of these matrix cracks appear to form toward the edge of the weft tow – though it is not unusual to see these cracks develop midway between the neighbouring weft tows. The debond occurs at the interface between the z-binder and a weft tow - this will be shown in more detail below (see Section 5.4.1). Propagation of the crack front across the specimen’s width in this region occurs much more rapidly than the cracks within the weft tows. From Figure 5.3d-f, it can be noted that debonding of the z-binder occurs regularly across the entire length and width of the specimen, wherever a crack in the resin-rich region intersects the z-binder.

From the photographs taken during testing, a noticeable darkening begins to develop from many of the weft tow transverse matrix cracks for strains above about 1.3%. Figure 5.3e shows the specimen at a strain of approximately 1.83% where these regions have developed sufficiently clearly to be
identified. It is likely that these are delamination cracks initiating from the transverse matrix cracks at the interface between warp and weft tows. The shape and location of these darkened regions are like the delaminations observed during early-stage fatigue damage development, as will be seen in Section 5.4.1

Figure 5.3f shows the specimen close to fracture. Cracking and debonding has accumulated to a point where the specimen appears almost opaque under load and Figure 5.3g shows the specimen after failure where the final fracture can be clearly observed. Interestingly, failure of the specimen results in it becoming unloaded, which returns much of the specimen’s transparency.

![Figure 5.3: Photograph of a warp-direction 3D-78 specimen taken during quasi-static tensile loading; a) Unloaded; b) 168 MPa/0.69% strain; c) 210 MPa/0.9% strain; d) 248 MPa/1.17% strain; e) 357 MPa/1.83% strain; f) 452 MPa/2.35% strain; g) failed specimen = 517 MPa/2.74% strain]
5.3.2. Weft-direction

Figure 5.4 shows the development of damage in a weft-direction loaded specimen. In a similar manner to loading in the warp-direction, Figure 5.4b shows that darkened regions are produced when loading along the weft-direction. In this case the darkened regions are associated with the warp tows, and the non-darkened areas correspond to the locations of the z-binders. Since the z-binders essentially runs parallel to the warp tows, it could be assumed that fibre/matrix debonding should cause the whole specimen to become darker. However, the z-binders traverse between both surfaces of the laminate in a sinusoidal fashion. If debonding was to occur in the same way as it does for the warp tows, then the transmitted light is clearly not lost at the fibre/matrix interfaces in the same way, or is not observed in the same way because of the path of the z-binders. Alternatively, it is possible that fibre/matrix debonding in the z-binders is not occurring for some reason.

In a similar way to the warp-direction loading, macroscopic damage initiates during weft-direction loading in the form of warp tow transverse matrix cracks (see Figure 5.4c). Initiation of these transverse cracks tends to occur preferentially along the edges of the specimens, though with continued loading many cracks develop in addition at varying positions across the specimen’s width, which can be seen in Figure 5.4c-f.

As indicated in Figure 5.4d, transverse cracks also form in the z-binders. The propagation of these transverse cracks along the width of the specimen is quite rapid in comparison to the warp tow transverse cracks. This is evident in Figure 5.4d where many long z-binder transverse cracks can be seen, while most of the warp tow transverse cracks are still quite short. It was shown in Section 5.2 that the presence of a z-binder produces large resin pockets between adjacent warp tows. In a similar manner to transverse crack growth along the resin-rich channel in warp-direction loaded specimens, the z-binder transverse cracks here extend into the resin-rich pockets whereby crack growth is accelerated – cross-sectional images of this damage will be shown later in Section 1.4.2.

Comparing warp-direction and weft-direction loaded specimens near failure (see Figure 5.3f and Figure 5.4f respectively), the crack density appears higher in specimens loaded along the weft direction. The reason for this is probably related to a more regular profile of the warp tows (see Figure 5.2c). In warp-direction loading it was mentioned that the surface weft tows were thickest toward their centre, where cracks generally initiate first. As warp tows have a more regular cross-sectional thickness, the observation that cracks initiate preferentially at the thickest part of the weft tows (in warp-direction loading) does not apply, consequently, it is possible for a warp tow (in weft-direction loading) to sustain more cracking.

Unlike loading in the warp-direction, images of the damaged weft-direction loaded specimens (Figure 5.4c-g) show either very little or no delamination development from the transverse matrix cracks in the warp tows. As mentioned above, there appears to be a higher crack density in weft-
direction loading. This is possibly because the rectangular cross-section of the tows promotes more cracking per tow, and because of the absence of the development of delaminations.

An unusual type of damage does develop in weft-direction loading which is related to interfacial fracture, or debonding, between the z-binder and the corresponding weft tow. As indicated in Figure 5.4d, this type of damage appears almost triangular in plan-view images, with the vertex of the triangle pointed towards the centre of the weft tows; cross-sections detailing the location and morphology of this damage can be found in Section 5.4.2 on weft-direction damage under fatigue loading.

As loading reaches higher strains (Figure 5.4e and f) it appears that the weft tows become darker. It is possible that this is the result of transverse strains causing fibre/matrix debonding in the same way the longitudinal strains cause fibre/matrix debonding of the warp tows at low strains.

![Figure 5.4: Photograph of a weft-direction 3D-78 specimen taken during quasi-static tensile loading; a) Unloaded; b) 177 MPa/0.71% strain; c) 262 MPa/1.17% strain; d) 296 MPa/1.4% strain; e) 365 MPa/1.8% strain; f) 462 MPa/2.36% strain; g) failed specimen = 541 MPa/2.89% strain](image-url)
5.4. Tension-tension fatigue damage development

In order to investigate the morphology of damage developed in tension-tension fatigue loading, tests were carried out on specimens at different peak stress levels, all with an R-value of R = 0.1. At a low peak stress (100 MPa) the fatigue lifetime in both the warp and weft directions were in excess of a million cycles. Under this condition, initiation and propagation of damage was sufficiently slow that the development of damage could be observed easily. At higher peak stresses (e.g. 200 MPa), many early stage damage mechanisms developed very rapidly making it difficult to determine damage development, although those damage mechanisms critical to the failure of the specimen were easier to monitor.

5.4.1. Warp-direction

Figure 5.5 shows the early stage damage development in a specimen before loading and after 25, 2000, 4000, 16000, and 32000 cycles where the peak fatigue stress per cycle is 100 MPa. In the initial image of the unloaded/untested specimen (Figure 5.5a), the locations of the Z-binders, warp, and weft tows are indicated; there are also lighter coloured, resin-rich regions, traversing the width of the specimen, which separate each weft tow. In addition, many small voids are evident.

Under fatigue loading, a darkening of the weft tows could be seen at an early stage. Figure 5.5b shows this darkening after 25 cycles which, as in quasi-staic loading, is probably due to partial debonding of the 90° fibres from the matrix as discussed above.

Figure 5.5c shows that the development of the first macroscopically visible damage, a weft tow transverse matrix crack initiating from the edge of the specimen and propagating across the width of the specimen. The crack highlighted in Figure 5.5c has been labelled “A” and its progressive growth across the width is shown in Figure 5.5d-g. In Figure 5.5e, there are regions where multiple transverse matrix cracks appear to have formed close together within a single weft tow. An example of this type of cracking has been highlighted at “B”. As will be shown subsequently, this can be explained by considering the geometry of the 3D fabric. Since there are three separated weft tow layers through the thickness of this fabric, transverse matrix cracks can form in each weft tow without necessarily occurring in the same layer.

As well as weft tow transverse matrix cracks, some transverse cracks can be seen to form at the boundaries of weft tows or in the resin region between the boundaries of weft tows (boundary transverse cracks). As will be shown below, most of these transverse cracks are through-thickness, except where they intersect the warp tows or a Z-binder. One such crack has been highlighted and enlarged in Figure 5.5f. In the plan view of the specimen shown in Figure 5.5g, the location where the boundary transverse cracks intersect Z-binders appears as a darkened region. These darkened regions form regularly along
the length of the specimen, as can be seen in Figure 5.5g. The darkened regions are the consequence of interfacial cracking at the Z-binder (i.e. these are Z-binder debonds).

![Figure 5.5: Photographs of a specimen fatigue loaded along the warp-direction with a peak stress of 100 MPa for approximately 60,000 cycles; a) 0 cycles - unloaded (before testing); b) 25 cycles; c) 2000 cycles; d) 4000 cycles; e) 8000 cycles; f) 16000 cycles; g) 32000 cycles](image)

Associated with the weft tow transverse matrix cracks, tow-level interfacial fractures (micro-delaminations) develop between the weft tows and the warp tows (Figure 5.5f). These micro-delaminations are oval-shaped and extend both along the loading direction as well as transverse to the loading direction. Due to the use of fibre tows, the warp/weft tow interface is a relatively small rectangular section, bound by resin-rich regions. As a direct consequence, the micro-delaminations tend not extend past these boundaries; an example of micro-delaminations terminating along the edge of a warp tow has been highlighted in Figure 5.5g. Figure 5.6a-e follows the growth of a micro-delamination in a specimen fatigue loaded with a peak stress of 200 MPa. This micro-delamination can be seen growing along one edge of the warp tow, extending towards the edges of the weft tow. In contrast, the same micro-delamination tends to a point along the opposite edge of the warp tow; Figure 5.7a illustrates this growth schematically as if viewed from the surface of the specimen. In relation to the geometry of this 3D fabric, the part of the micro-delamination tending to a point also corresponds to the crown of the Z-binder. It is as if the Z-binder is causing a pinching effect that is restricting the
growth of the micro-delamination at the weft tow/warp tow interface immediately beneath the Z-crown. The architecture of the 3D fabric leads to a discernible repetition of the micro-delaminations (shown schematically in Figure 5.7b), with each delamination pointing towards the crown of a Z-binder.

Figure 5.6: Plan view photographs of a 3D-78 specimen fatigue loaded along the warp-direction with a peak stress of 200MPa; each indicating the development of damage at various stages over the specimen’s fatigue life; a) 200 cycles; b) 500 cycles; c) 1000 cycles; d) 8000 cycles; e) 140000 cycles

Plan view images at higher magnification, and images of polished cross-sections at appropriate locations, enabled the morphology of the 3D damage to be understood. Figure 5.8 consists of two images taken from another specimen fatigue-loaded along the warp-direction for 100,000 cycles at a peak stress of 100 MPa. The lower image shows the plan view of the specimen and the upper image is a cross-section of the same specimen at the same magnification, taken through the plane of a Z-binder. The plane of the cross-section of the upper image is indicated in the lower image by the line A-A’.
Figure 5.7: Diagrams indicating: a) delamination growth during fatigue loading specimens along the warp-direction, b) delamination growth pattern over a larger region of the material.

A number of observations can be made from Figure 5.8. First, transverse matrix cracks in the weft tows, which apparently formed close together when looking at the plan view images, can be seen to be at different locations through the thickness of the composite; such an example is seen at “C”. Second, it was mentioned above, and highlighted in Figure 5.5f and g, that there are some transverse cracks which form between weft tow boundaries and extend across the thickness of the specimen, except where they terminate at warp tows or at Z-binders; at these locations, the Z-binder debonds appear as a darkened region. In the plan view image of Figure 5.8, two of these darkened regions have been indicated, at “D” and “E”. It can be observed from the cross-section that the darkened regions correspond to interfacial fractures at the Z-binder/resin pocket interface or at the Z-binder/weft tow interface i.e. Z-binder debonding has occurred. The Z-binder debonding at “D” and “E” has fully developed, while there is the beginning of Z-binder debonding at two locations at “F”. It should be noted that in all four examples shown in Figure 5.8, the Z-binder debonding is associated with a matrix crack at a weft tow boundary or in the resin pocket between weft tow boundaries. The debonding of the Z-binders in these plan view images appears similar to the damage found in [47] for tensile loading, and in [58] for fatigue loading. Both papers [3, 4] imply that these debonds occur at the interface between the Z-binder crown and a surface weft tow, but that does not appear to be the case here (it should be noted that in [47] and [58] a thicker non-crimp 3D orthogonal woven fabric, with four weft and three warp layers, was tested). Finally, a void can be seen in Figure 5.8; perhaps surprisingly, the void does not appear to be associated with the fatigue damage.
Figure 5.8: Cross-section and plan view photograph of a 3D-78 specimen fatigue loaded along the warp-direction with a peak stress of 100 MPa for approximately 100,000 cycles. Both images have the same scale and show damage mechanisms such as Z-binder micro-debonds, transverse cracks between weft tow boundaries, and weft tow transverse matrix cracks.

The geometry of the micro-delaminations, which form between the weft tows and the warp tows, can be seen in Figure 5.9 with the cross-section indicated by the line B-B’ in the surface view photograph below. The cross-sectional image was taken approximately midway across the width of a warp tow and consequently, the Z-binder cannot be seen. The matrix cracks which appear sharply in the lower image, at “G” and “H”, can be seen to be in the upper surface weft tows (i.e. those nearer the camera) at G’ and H’. These weft tow matrix cracks are also associated with interfacial fractures between the surface weft tows and the adjacent warp tow; since these fractures lie in the plane of the interface between the weft and warp tows, it seems reasonable to refer to them as micro-delaminations. The weft tow matrix cracks at “J” and “K” are also associated with micro-delaminations at the weft tow/warp tow interface. Figure 5.9 also shows examples at “L” and “M” of transverse cracks between weft tow boundaries which extend across the thickness of the specimen, except where they meet the warp tows. There are no delaminations at the matrix/warp tow interface associated with these cracks, which suggests that the mismatch in Poisson’s ratio between the warp and weft tows leads to the micro-delaminations.
Figure 5.9: Cross-section and plan view photograph of a 3D-78 specimen fatigue loaded along the warp-direction with a peak stress of 100 MPa for approximately 100,000 cycles. Both images have the same scale and show damage mechanisms including micro-delaminations, transverse cracks between weft tow boundaries, and weft tow transverse matrix cracks.

Taking a cross-section close to the edge of a warp tow, although not within the Z-binder which consequently cannot be seen in this cross-section (Figure 5.10), it is observed that wide micro-delaminations form at the weft/warp interface furthest from the Z-crown, whilst narrower micro-delaminations form at the weft/warp interface closest to the Z-crown; this is in agreement with the schematic diagrams of Figure 5.7. In late stage fatigue, it has been observed that fibre fractures occur preferentially at the edge of a warp tow nearest a Z-binder crown [72]. At these locations, toward the edges of warp tows, the weft/warp micro-delaminations have reduced in width to zero, so that stress concentrations caused by weft tow transverse matrix cracks are not eliminated by the micro-delaminations here; consequently, it is not surprising to find extensive fibre fractures at these locations i.e. at the edges of warp tows, as shown in Figure 5.11.
5.4.2. Weft-direction

Figure 5.12 shows images of the early stage damage development of a specimen subjected to fatigue loading in the weft-direction, at the same number of cycles as for Figure 5.5. In Figure 5.12a (an unloaded/untested specimen) the locations of the warp/weft tows, and Z-binders have been indicated; additionally, it can be noted that this specimen contains a small number of voids. After 25 fatigue cycles (Figure 5.12b), a darkening of the warp tows can be seen which is, again, probably due to partial debonding of the 90° fibres from the surrounding matrix.

In a similar way to the specimens fatigued in the warp-direction described above, visible damage initiates as matrix cracks along both edges of the specimen, though this time growing in the warp tows (Figure 5.12c). A number of cracks initiate within each warp tow and slowly grow across the width of the specimen; examples have been highlighted at “N” in Figure 5.12c-g. After 32,000 cycles, very few of these warp tow matrix cracks had extended completely across the specimen width. In Figure 5.12f and g, additional cracks can be seen to have formed towards the centre of the specimen.
Like the warp-direction loaded specimens, matrix cracks for weft-direction loading can form in different planes (there are two warp tow layers through the thickness).

![Diagram showing the loading direction and a 3D-78 specimen fatigue loaded along the weft-direction with a peak stress of 100 MPa for approximately 60,000 cycles.]

Figure 5.12: Photographs of a 3D-78 specimen fatigue loaded along the weft-direction with a peak stress of 100 MPa for approximately 60,000 cycles. All images were taken under continued loaded conditions after a number of cycles, except “a)” which occurred before testing; a) 0 cycles – unloaded (before testing); b) 25 cycles; c) 2000 cycles; d) 4000 cycles; e) 8000 cycles; f) 16000 cycles; g) 32000 cycles.

In addition to transverse cracks forming in the warp tows, some transverse cracks can be seen to have formed through the Z-binder (Figure 5.12e). These cracks form anywhere across the width of the Z-binder, although many form at the centre of the Z-binder tows. Many of these cracks extend across the specimen width and through the thickness of the specimen as a single extended crack, as shown by the crack labelled “P” in Figure 5.12e. Often, multiple cracks form along the length of the Z-binder, growing until they meet (presumably because stress-shielding then inhibits crack growth); an example is shown at “Q” in Figure 5.12f. Due to the geometry of a Z-binder, these cracks also create a fracture through the resin pockets. Compared with warp tow matrix cracks, cracks in this region propagate rapidly.

The most striking damage which develops when fatigue loading in the weft-direction is the formation of unusual triangular-shaped damage; in Figure 5.12e-g, regions typical of this phenomenon are highlighted. These regions are associated with the transverse Z-binder cracks. In the plan view...
images of Figure 5.12f and g, the triangular-shaped damage occurs regularly with a noticeable, repeating pattern. From this pattern, it can be noted that the triangular-shaped damage appears in pairs, with each pair facing in opposite directions. An example of one of the pairs is highlighted in Figure 5.12f and g with the label “R”. Measurements from micrographs have shown that each pair extends across half the width of a weft tow as well as the width of a Z-binder. As the micrographs described below will show, the triangular-shaped damage pairs are the result of interfacial debonding between the Z-binders and adjacent weft tows.

Figure 5.13: Plan view photographs of a specimen fatigue loaded along the weft-direction with a peak stress of 200MPa; each indicating the development of damage at various stages over the specimen’s fatigue life. a) 200 cycles, b) 500 cycles, c) 1000 cycles, d) 8000 cycles; e) 45000 cycles

Unlike in the warp-direction, micro-delaminations do not initiate during the early stage of fatigue in weft-direction loaded specimens. Figure 5.13 shows a series of photographs taken during a test where the peak stress was 200MPa. Figure 5.13a-c shows limited, if any, micro-delamination development in the first 1000 cycles, which is in direct contrast to the warp-direction loading where significant micro-delaminations have developed by 1000 cycles. However, by 8000 cycles (Figure 5.13d), some micro-delaminantion growth has occurred from many of the warp tow transverse matrix
cracks; a micro-delamination highlighted in Figure 5.13d can be seen extending across the width of a weft tow. Over the fatigue life of a specimen, micro-delamination growth is slow; however, as the specimen nears failure, the area covered by micro-delaminations is quite extensive (Figure 5.13e).

The final easily distinguishable damage to form during fatigue loading in the weft-direction is longitudinal splitting; Figure 5.13e highlights an example. These splits originate from the centre of the surface weft tow and Z-binder crown; the splits grow in both directions parallel to the loading direction at a roughly equal rate away from the site of their initiation at the Z-binder. None of the splits were observed to grow sufficiently to link up before specimen failure.

Interestingly, in contrast to final failure for the warp-direction, which appears to occur through the development of fibre fractures at the edges of warp tows, for the weft-direction no distinct region has been identified from which final fatigue failure develops. Indeed, it is possible that the extensive micro-delaminations which occur throughout the weft-loaded specimens in the later stages of their fatigue lifetime may remove the stress concentrations associated with the warp tow matrix cracks.

![Figure 5.14: 3D-78 specimen fatigued with a peak stress of 100MPa for approximately 60,000 cycles along the weft-direction and sectioned along the weft plane (loading direction). The image displays various damage mechanisms including warp tow matrix cracks and Z-binder transverse cracks](image)

Before considering the triangular-shaped damage, the other types of damage will be discussed. Figure 5.14 shows a cross-section taken in the plane of a weft tow for a specimen fatigue loaded with a peak stress of 100 MPa for approximately 60,000 cycles. Transverse warp tow matrix cracks at “S” can be seen, both in different tows and in different locations through the thickness. In addition to these cracks, through-thickness transverse cracks in the Z-binder and associated resin-rich pockets can be seen at “T”, “U”, and “V”. Some of these cracks (e.g. “U”) form in the middle of the Z-binder and extend through the middle of the resin pocket between warp tow boundaries. Others (e.g. at “T” and “V”), form near the edges of the Z-binder and extend along the edges of the warp tow boundaries. No micro-delaminations of the type found in the warp-direction specimens were observed in this weft specimen, which matches what was observed above in Figure 5.12.
Figure 5.15: 3D-78 specimen fatigue loaded along the weft-direction at a peak stress of 100MPa for 60,000 cycles. Each image is a cross-section taken through the thickness of a Z-binder. A surface view image is attached to each with a red line indicating the position of corresponding cross-section. Blue dashed lines are used to match feature on the surface image with the features shown in the cross-section.

The morphology of the triangular-shaped damage pairs can be understood with reference to Figure 5.15 where multiple cross-sections are shown across the width of a Z-binder for a weft-direction, fatigue-loaded specimen; the images have been achieved by gradually polishing through the Z-binder. The six pairs of images in Figure 5.15 shows a plan view image (below) and a cross-section (above). In each plan view image, the red line shows the location of the complementary cross-section shown above; hence, in moving from Figure 5.15a-f, the red line sweeps from the bottom of the lower image to the
top, showing the location of the polished cross-section. The morphology of the damage is complex: fundamentally it consists of (i) interfacial fracture between the Z-binder and the surface weft tow, (ii) debonding of the Z-binder from the central weft tow, and (iii) interfacial fracture between the central weft tow and the adjacent resin pocket. Although the Z-binder traverses asymmetrically from one surface of the composite to the opposite surface at this location, the apparent symmetry of the paired nature of the damage results from viewing the damage in the plan view.

5.5. Crack density

In this section, the crack density of warp and weft-direction specimens fatigued with a peak stress of 200 MPa is calculated. The first part of this section is related to the methodology used to calculate the crack density of these 3DNCOW composites, while the second section compares the measured crack density of specimens loaded along the warp and weft direction.

5.5.1. Methodology

When comparing crack accumulation for warp-direction loading and weft-direction loading, either for quasi-static tension or tension-tension fatigue, it is apparent that the number of cracks is greater for weft-direction loading. This was verified by measuring the crack density, or cracked area per unit volume, for specimens loaded in each direction. In a simple layup, such as a [0/90]s cross-ply, the crack density for through-width, through thickness cracks in the 90 layer, (i.e. the cracked area per unit volume), is simply the number of cracks multiplied by the width and thickness of the 90° layer and then divided by the volume of the 90° layer. For sufficiently thick transverse plies [71], when a transverse matrix crack in a 90° ply forms under tensile loading, it propagates across both the width and thickness almost instantaneously. It should be noted that the authors found that reducing the thickness of the 90° ply not only increased the crack initiation strain but also had the effect of reducing the growth rate across the width of the specimen for increasing load/strain. Whilst the crack density measurement for a [0/90]s cross-ply is simple, it is not the case for a 3D woven composite. In a 3D woven composite the complexity of the fibre architecture makes it difficult to determine the precise extension of a crack. Unlike a cross-ply laminate, a 3D woven preform is made up of fibre tow layers rather than continuous sheets of fibres. Added to this, the addition of Z-binder tows creates resin pockets that can enable a crack to grow through the thickness – this is not possible for [0/90]s, or other fibre layups manufactured with UD prepreg, or for 2D non-crimp fabrics.

To estimate the crack density in a 3D composite, however, a knowledge of a crack’s location in relation to the various components within the fibre architecture can enable an “educated guess” to be made of the path of the crack through the composite. This means an approximate crack density measurement can be made for the specimen; the use of lower and upper bounds on this “educated guess” enables useful measurements to be made.
A more accurate way of measuring the crack density would be to look at the cross-section of a specimen and follow the path of each crack present across the width of the specimen and there are two methods that could be used to do this: optical microscopy and X-ray μCT tomography. In the case of optical microscopy, while the images are very clear, many cross-sections would be required for an accurate measurement of each crack to be made. On the other hand, whilst μCT can obtain information on cracks in a complete volume of a specimen, enabling the inspection of the corresponding cross-section, there are difficulties in detecting individual cracks. Specimens can be soaked in a dye-penetrant [62], which will often make it easier to see cracks in a μCT scan. However, this only works if there are connections between the cracks within the specimen and the specimen surface.

In this work, 2D micrographs have been used, together with a method for estimating crack paths in relation to the fibre architecture in order that estimates of the accumulation of cracking with fatigue cycles could be made.

The 3D woven specimens had a sufficient level of transparency that in-situ photographs of the specimen could be taken during testing to monitor the damage developed. This was especially useful when trying to determine the crack density of a specimen at various cycles during fatigue loading. However, as only one surface was photographed, it is not possible to say with certainty that all cracks were visible in the images; some cracks (most probably toward the far surface) appeared faintly in the images. Due to the complex structure of 3D woven composites, some assumptions were made to simplify the calculations used to determine the crack density of a specimen. The assumptions were:

1) The use of rectangular cross-sections to construct an idealised structure in each direction.

Figure 5.16 and Figure 5.17 are schematic diagrams that show representative cross-sections along the y-z and x-z planes (see Figure 5.1 for a reminder of the structure) when idealised into rectangular blocks. The most notable differences between these idealised cross-sectional schematics and the actual geometry is the shape of the surface weft tows and the Z-binder, which in reality are semi-circular and sinusoidal respectively. While the use of rectangular blocks does not accurately reproduce the 3D woven geometry, they do enable a simplification of the equations used to calculate the cracked area for cracks present in the 3D structure.

2) All cracks initiate within a tow, whether that be a warp, weft, or Z-binder, and span the entire thickness of that tow. While transverse cracks grow through resin-rich pockets and channels, they generally do not initiate in these locations, with the resin-rich channel between adjacent weft tows during warp-direction loading being the exception.

3) It is assumed that the propagation of transverse matrix cracks in weft and warp tows, during warp and weft-direction loading respectively, can occur in one of two ways, either (1) the crack grows entirely through the thickness (ignoring only fibres running along the loading direction) or (2) the cracks are constrained to the tow in which it initiated.
Since the photographs taken during testing only provide a plan view of the specimen, it is unclear exactly how each transverse matrix crack propagates through the thickness of the material. As illustrated in Figure 5.16 and Figure 5.17, there are many resin-rich pockets that can act as a pathway for cracks to propagate into other transverse tow layers located at other locations through the thickness. However, crack propagation through the thickness is only possible if the crack is situated in a suitable location with respect to the fibre architecture, i.e. that the crack extends across some portion of a resin-rich pocket. It is the resin pockets which allow cracks to extend into other layers of the composite.

Figure 5.16: Idealised cross-sections along a weft plane indicating transverse crack propagation through a warp-direction loaded specimen. The crack propagations shown here are considered the two extremes, namely an “under-estimate” and “over-estimate” of the crack areas.

Figure 5.17: Idealised cross-sections along a warp plane indicating transverse crack propagation through a weft-direction loaded specimen. The crack propagations shown here are considered the two extremes, namely an “under-estimate” and “over-estimate” of the crack areas.

To recap: to measure the crack density in these 3D woven specimens, the crack area needs to be calculated. This can only be achieved if the through-thickness crack length is known. Given that the
through-thickness crack length cannot be determined from a surface view image of a specimen, equations encompassing two extreme cases were formed.

The first case assumed that all the transverse matrix cracks have not propagated any further through the thickness than the tow in which they initiate. Depending on the location and direction of loading, equations (1), (4), or (5) from Table 5.1 can be used for this condition (note that the symbols used are defined in Figure 5.16 and Figure 5.17). However, many cracks can progress further through the thickness, so these equations produce an “under-estimate” of the cracked area. While these equations are used on cracks extending over resin-rich areas, they are also used for cracks that are constrained by the transverse tows above and below. Light grey rectangular blocks have been used in Figure 5.16 and Figure 5.17 to illustrate the idea of an under-estimated crack area.

In contrast, the second case “over-estimates” the cracked area by assuming that any crack that extends across a resin-rich region will propagate further through the thickness until another boundary has been reached. For this condition, the transverse crack area is essentially considered a block, which extends across a portion or the entire thickness of the specimen, minus sections of longitudinal fibre tows, i.e. the crack length is the same in each transverse tow, or resin pocket depending on the location the transverse crack. Again, in Figure 5.16 and Figure 5.17, yellow blocks have been used to illustrate cracked areas that extend through the thickness further than a single tow thickness, thus providing the “over-estimated” crack area.

For loading in the warp direction, equations (2) and (3) assume that transverse cracks will be propagating completely through thickness if the above condition is met as the warp tow boundaries occur at the same point. This is not quite the same for weft-direction loading, with the width of the central weft tows greater than the surface weft tows; this is the result of binding from the z-binder. As indicated in Figure 5.17, this difference in geometry means some cracks cannot completely propagate through the thickness. Depending on the position of the cracks in relation to the boundaries, either equation (6) or (7) can be used.

Using photographs of images with lots of damage, it is possible to determine the approximate location of the edge of the longitudinal fibre tows. These can then be used as boundary conditions so that the most appropriate equation can be used. As mentioned in section 5.4.2 (weft-direction development of damage during fatigue), the Z-binder debonds appear as two triangular formations when viewed from the surface. The flat edge of these debonds (running parallel to the loading direction) can be used as a reference for the edge of a weft tow, thus enabling the boundaries of the weft tows to be determined. The same principal can be used for warp-direction loaded specimens, however, in this case it is the flat edges of the micro-delamination cracks that enable the warp tow boundaries to be determined.
Table 5.1: Equations used to calculate the approximate crack area of various transverse cracks present in warp and weft-direction loaded specimens based on an idealised specimen cross-sectional geometry

<table>
<thead>
<tr>
<th>Loading direction</th>
<th>Crack Area (mm²)</th>
<th>Equation No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Warp</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Under-est.</td>
<td>$t_{wf}L$</td>
<td>(1)</td>
</tr>
<tr>
<td>Over-est.</td>
<td>$t_cL - 2t_{wp}[L - L_1] - t_z[L - L_2]$</td>
<td>(2)</td>
</tr>
<tr>
<td></td>
<td>$t_cL - 2t_{wp}L$</td>
<td>(3)</td>
</tr>
<tr>
<td>Weft</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Under-est.</td>
<td>$t_{wp}L$</td>
<td>(4)</td>
</tr>
<tr>
<td></td>
<td>$t_zL$</td>
<td>(5)</td>
</tr>
<tr>
<td>Over-est.</td>
<td>$t_cL - 2t_{wf1}[L - L_3] - t_{wf2}[L - L_4]$</td>
<td>(6)</td>
</tr>
<tr>
<td></td>
<td>$\frac{t_cL}{2} - t_{wf1}[L - L_3]$</td>
<td>(7)</td>
</tr>
</tbody>
</table>

5.5.2. Crack density measurements

Due to the time-consuming nature of making crack density measurements throughout the lifetime of a specimen, measurements were only produced up to the point where a plateau in the crack density occurred. These measurements were determined using test specimens fatigue loaded with a peak stress of 200 MPa along both the warp and weft-direction. Figure 5.18 shows crack density measurements for warp-direction loaded specimens as a function of cycle number. There is a rapid increase in crack density up to about cycle 500, after which the rate of cracking begins to decrease and approaches a plateau. When compared to the initial development and growth, the increase in crack density from cycle 4000 to cycle 8000 is small.

For weft-direction loaded specimens, Figure 5.19 shows a similar trend to warp-direction loaded specimens, but rising to a higher plateau. However, it took until cycle 1000 for the rate of increase in crack density with cycle number to begin reducing. Although the reduction in rate of crack density increase took a higher number of cycles in weft-direction fatigue loading compared with warp-direction fatigue loading, by around 4000 cycles the weft-direction crack density can be seen to be reaching a plateau. The small increase in crack density between cycles 4000 and 8000 indicates that crack growth is nearing saturation.

Figure 5.20 shows a comparison of the crack density measurements with cycling for both loading directions. Up to cycle 500 both loading directions have approximately the same crack densities – approximately 0.3 mm⁻¹. However, the plateau crack density in weft-direction loaded specimens is approximately twice that of the warp-direction. It was indicated in a section 5.4.2, that the higher crack density noted in weft-direction fatigue loaded specimens is likely a combination of the transverse warp
tow geometry and the absence of delamination growth during early-stage fatigue loading. The difference in warp-direction and weft-direction crack density measurements correspond well with damage observations.

In Figure 5.21, the normalised tangent modulus is plotted against the crack density for the warp-direction loaded specimens. Here it can be seen that the loss of stiffness of the specimens is reasonably linear with increasing crack density up to a crack density of approximately 0.3 mm\(^{-1}\). After this crack density, the rate of stiffness reduction increases for only a small increase in overall crack density. A close examination of the images used to measure the crack density in these specimens showed that micro-delaminations started to grow from around cycle 400/500 – these micro-delaminations were discussed earlier (see section 5.4.1). The growth of these micro-delaminations impeded further cracking in the weft tows because of load transfer around the crack and the micro-delaminations. However, because micro-delaminations are still a form of damage, the overall stiffness of the material is still affected, producing a reduction in stiffness for very little increase in matrix crack density.

The loss of stiffness as a function of crack density, as measured here, was compared to the crack density predicted from an F.E. voxel model of a representative volume element (RVE) of this 3D orthogonal architecture (see Figure 5.21) [72]. In this work, increasing numbers of cracks were inserted into the model and the corresponding reduction in stiffness was determined. In Figure 5.21 the reduction of stiffness with increasing crack density can be seen to correspond well between the F.E. model and
the measured crack density up to approximately 0.3mm\(^{-1}\). The model does not include the development of micro-delamination and predicts a linear decrease in stiffness with increasing crack density.

![Graph showing crack density vs. fatigue cycle number for weft-direction loaded 3D-78 specimens fatigue loaded at a peak stress of 200MPa.](image)

Figure 5.19: Crack density vs. fatigue cycle number for weft-direction loaded 3D-78 specimens fatigue loaded at a peak stress of 200MPa

By contrast, micro-delaminations in weft-direction loading do not initiate as early as in warp-direction loaded specimens. This is reflected in Figure 5.22 where there is an almost linear reduction in the specimen stiffness with an increasing crack density – although it must be said that the data has considerable scatter, both for each test coupon analysed and between coupons. Comparing the stiffness reduction against crack density plots for each loading direction (Figure 5.23) clarifies the linear reduction of stiffness with increasing crack density for the weft-direction loaded specimens compared to the warp-direction loaded specimens.
Figure 5.20: Crack density vs. fatigue cycle number comparison for both the warp-direction and weft-direction loaded 3D-78 specimens fatigue loaded at a peak stress of 200MPa.

Figure 5.21: Normalised tangent modulus vs. crack density for warp-direction loaded specimens fatigue loaded with a peak stress of 200MPa, with comparison made to crack density measurements made by Topal [72] in an idealised FE voxel model representation of this fabric architecture.
Figure 5.22: Normalised tangent modulus vs. crack density for weft-direction loaded 3D-78 specimens fatigue loaded with a peak stress of 200MPa

Figure 5.23: Normalised tangent modulus vs. crack density comparison of warp-direction and weft-direction loaded 3D-78 specimens fatigue loaded with a peak stress of 200MPa
5.6. Concluding remarks

It has been shown in this chapter that, while similar in many ways, the damage developed is generally specific to each loading direction.

When loading warp-direction specimens in either quasi-static tension or tension-tension fatigue it is found that damage generally forms in a similar way. In this loading direction, the main damage mechanisms include: weft tow transverse matrix cracks, resin-rich channel transverse matrix cracks, through-thickness debonding of the z-binder, delamination at the interface between warp and weft tows, and longitudinal splitting of the warp tows. As a result of the woven architecture, the delamination cracks develop a shield-like shape, with the pointed end of the shield-like shape corresponding to the position of the z-binder crown – most notable in specimens cyclically loaded. This restriction of the delamination cracks at these locations makes it easier for stress concentration to build at the edge of a warp tow, thus enabling the final failure to initiate at these locations.

Many of the same damage mechanisms were noted for specimens loaded along the weft-direction. However, positioning and extent of damage were different, with there being a clearly higher density of cracks in the weft-direction than the warp. This increase in cracks was coupled with a limited amount of delamination between interfaces. Most interfacial fractures in this loading case were due to debonding of the z-binder from the wefts tows. Unlike the warp-direction, there are no obvious regions that would enable the initiation of final failure.

Additionally, the initiation and development of delamination cracks in warp-direction loading has been found to reduce the development of transverse matrix cracks whilst maintaining a reduction in stiffness. Conversely, the lack of delamination formation in weft-direction loading has shown an almost doubling of the possible crack density for a similar loss of stiffness. It could be noted that the lack of transverse matrix cracks in warp-direction loading, as opposed to weft-direction loading, may be the result of tow cross-sections where the surface weft tows are more semi-circular and warp tows more rectangular; thicker sections will crack more readily and at lower strains than thinner sections [71].
Chapter 6
The mechanical characterisation of the 3DMG GFRP material

6.1. Introduction

In this chapter, an all-glass 3D non-crimp orthogonal woven (3DNCOW) composites manufactured by the University of Manchester is characterised with regard to its quasi-static and fatigue properties. For the purposes of this report the all glass 3D fabric produced by the University of Manchester will be referred to as 3DMG (3D Manchester glass). While fundamentally similar to the 3D-78 material discussed in Chapter 5, there are a few notable differences in the architecture that will be discussed in the next section.

All test specimens discussed in this chapter were loaded in quasi-static tension or tension-tension fatigue. Unlike the previous chapters most of the data presented here characterises the specimens loaded along the warp direction, with a brief characterisation of the weft direction.

6.2. 3DMG fibre architecture

As mentioned above, the 3DMG fabrics are fundamentally the same as 3D-78; each have three weft tow layers, two warp tow layers, and a z-binder that interlaces through-thickness orthogonally perpendicular to the warp direction. Before proceeding further, it should be noted that two 3DMG manufacturing set ups were tested in this work, with the difference being the tension applied to the z-binder during the weaving process. The initial fabric had a low binder tension and the final 3DMG fabric produced had an increased binder tension to try to produce a fabric closer to the 3TEX 3D-78 fabric; this will be discussed later in the chapter. To differentiate between the two 3DMG, a “T1” and “T2” suffix has been added to the fabric name, referring to the lower and higher z-binder tensions respectively. The tension applied to each fabric during the weaving process, and the unit cell size, is shown in Table 6.1.

For the characterisation of the architecture of the 3DMG materials, three cross-sections of composites with each z-binder tension were examined. Two cross-sections were inspected along the x-z plane, with x in the warp direction, and one cross-section along the y-z plane, with y in the weft direction. Each of these cross-sections for 3DMG-T1 and 3DMG-T2 can be seen in Figure 6.1 and Figure 6.2.
Table 6.1: Comparison of unit cells between the various all-glass 3DNCOW fabrics used during this project. Also included here is the amount of tension in gram-force (gf) applied to the z-binders during weaving.

<table>
<thead>
<tr>
<th>Fabric</th>
<th>Tension (gf)</th>
<th>Unit Cell (direction)</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Warp (mm)</td>
<td>Weft (mm)</td>
</tr>
<tr>
<td>3D-78</td>
<td>Unknown</td>
<td>7.12 ± 0.18</td>
<td>7.12 ± 0.09</td>
<td></td>
</tr>
<tr>
<td>3DMG-T1</td>
<td>80</td>
<td>6.49 ± 0.17</td>
<td>6.51 ± 0.14</td>
<td></td>
</tr>
<tr>
<td>3DMG-T2</td>
<td>120</td>
<td>6.26 ± 0.15</td>
<td>6.17 ± 0.11</td>
<td></td>
</tr>
</tbody>
</table>

Referring to Chapter 5, it was shown that the path of the z-binder in the 3D-78 fabric was sinusoidal; this is quite different from the idealised shape of a 3DNCOW fabric which is closer to a square wave-type shape. It was noted that the sinusoidal shape in the 3D-78 had a direct influence on the rest of the fibre architecture. In comparison, the 3DMG-T1 fabric architecture in Figure 6.1 is more orthogonal, with the z-binder path in Figure 6.1b closer to the idealised shape so that the weft tow layers remain more consistently stacked in the through-thickness direction.

It is also possible to speculate that the more consistent structure, i.e. the more orthogonal path of the z-binder, is due to the reduced unit cell (Table 6.1) in the 3DMG when compared with the 3D-78 material. Although the fibre tows used in 3DMG are not the same as 3D-78 (Chapter 3), they are similar.
in size. A smaller unit cell is the direct result of reducing the spacing between tows along the length and width of the specimen. Reducing this spacing will affect the architecture of the fabric in several ways: (1) it forces the through-thickness region of the z-binder to become more vertical due to restricted spacing between neighbouring weft tows; and (2) a more vertical z-binder pathway means a smaller resin-rich channel between neighbouring weft tows.

![Diagram of fabric architecture](image)

**Figure 6.2:** Micrographs of cross-sections through MG-T2 taken along three planes: a) x-z through the warp tows, b) x-z through a z-binder, and c) y-z through the weft tows

Because of the z-binder in the preform, there are spaces between warp tows. With the surface weft tows passing over this space, and a tension being applied to the z-binder, an out-of-plane compressive force acts against the surface weft tows creating an area of crimp within these weft tows. This can clearly be seen in Figure 6.1c and Figure 6.2c. In Chapter 5, the tension on the z-binder caused an additional impact on the warp tows as a direct result of the crimp in the surface weft tows. Interestingly, if the fabric had many more layers, the effect on tow layers located more centrally would be reduced, with the amount of crimp increasing toward the outer layers. Whilst the tension on the z-binder in the 3D-78 material is unknown, it appears likely that it is higher than either of the tensions applied to the 3DMG material. In Figure 6.1b/c and Figure 6.2b/c it is apparent that the warp and weft
tows are quite straight, unlike in the 3D-78 material; the reduced tension in the 3DMG material as well as the more orthogonal pathway of the z-binder, most likely contributes to this.

It was mentioned above that the 3DMG material was produced with two different tensions. It should be noted that other than the increased tension, to the best of the manufacturer’s knowledge, no other changes were made during the set up and weaving of the 3DMG-T2 preform. Close inspection, however, reveals two main differences between 3DMG-T1 and 3DMG-T2; 3DMG-T2 was consistently thicker upon consolidation with resin and had an overall smaller unit cell than 3DMG-T1. Comparison here is made between laminates manufactured using the wet-layup method, where it was found that 3DMG-T2 was approximately 10-15% thicker than 3DMG-T1. Additionally, there was a 4-6% reduction in the unit cell dimensions for 3DMG-T2 compared to 3DMG-T1. As the manufacturing process used was the same for both preforms, it is unclear why such differences occurred.

6.3. Mechanical properties of 3DMG manufactured using the wet-layup method

6.3.1. Quasi-static tensile mechanical properties

To determine some basic mechanical properties of the 3DMG fabrics used in this project, quasi-static tensile tests were conducted along both principal directions, i.e. warp and weft. Three to five tests were conducted on both 3DMG-T1 and –T2 specimens. With a limited supply of material from each production run woven by the University of Manchester, only a few laminates could be produced for tests to be conducted on.

Table 6.2: Quasi-static tensile properties of 3DMG loaded along the warp and weft directions, including 3TEX data for comparison (see Chapter 4) – it should be noted all data in this table comes from specimens manufactured using the wet-layup method and that the warp and weft specimens were taken from different panels which had slightly different thicknesses – the panel thicknesses are shown.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>E (GPa)</th>
<th>E (kN/mm)</th>
<th>(\sigma_{\text{ULT}}) (MPa)</th>
<th>(\sigma_{\text{ULT}}) (N/mm)</th>
<th>(\varepsilon_{\text{max}}) (%)</th>
<th>Vf (%)</th>
<th>t (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3TEX</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Warp</td>
<td>26.1 ± 0.7</td>
<td>-</td>
<td>519 ± 33</td>
<td>-</td>
<td>2.8 ± 0.2</td>
<td>47.4 ± 2.9</td>
<td>2.11 ± 0.09</td>
</tr>
<tr>
<td>Weft</td>
<td>25.3 ± 0.4</td>
<td>-</td>
<td>514 ± 28</td>
<td>-</td>
<td>2.8 ± 0.2</td>
<td>46.9 ± 0.9</td>
<td>2.14 ± 0.04</td>
</tr>
<tr>
<td>3DMG-T1</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Warp</td>
<td>30.3 ± 1.8</td>
<td>68.1 ± 3.8</td>
<td>537 ± 29</td>
<td>1206 ± 72</td>
<td>2.5 ± 0.2</td>
<td>51.2 ± 2.4</td>
<td>2.25 ± 0.01</td>
</tr>
<tr>
<td>Weft</td>
<td>24.3 ± 0.4</td>
<td>59.8 ± 1.2</td>
<td>500 ± 9</td>
<td>1228 ± 26</td>
<td>3.1 ± 0.1</td>
<td>49.1 ± 0.9</td>
<td>2.46 ± 0.03</td>
</tr>
<tr>
<td>3DMG-T2</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Warp</td>
<td>25.5 ± 0.8</td>
<td>64.0 ± 2.0</td>
<td>550 ± 17</td>
<td>1383 ± 46</td>
<td>3.0 ± 0.1</td>
<td>48.8 ± 1.5</td>
<td>2.48 ± 0.03</td>
</tr>
<tr>
<td>Weft</td>
<td>25.3 ± 1.0</td>
<td>61.6 ± 0.9</td>
<td>510 ± 15</td>
<td>1244 ± 44</td>
<td>2.8 ± 0.1</td>
<td>50.8 ± 0.5</td>
<td>2.44 ± 0.05</td>
</tr>
</tbody>
</table>
Table 6.2 shows the mechanical properties of 3DMG material loaded along the warp and weft directions, for both z-binder tensions i.e. T1 and T2 specimens. When loading along the warp-direction, the tensile modulus and ultimate strength of the 3DMG-T1 is higher, and the strain-to-failure lower, than the warp-direction mechanical properties of 3D-78. Conversely, the tensile modulus and ultimate strength of specimens loaded along the weft-direction are relatively similar for both 3DMG-T1 and 3D-78 specimens. Comparison of the 3DMG-T1 warp and weft-direction loaded specimens shows that the tensile modulus is approximately 20% greater along the warp direction than the weft, and vice versa for the strain-to-failure. It can be noted that the weft-direction specimens were generally thicker than the warp-direction specimens; specimens of each loading direction were cut from different panels, however, the method of manufacture was the same and therefore it is unclear why there is such a difference in specimen thickness. It could be assume that the difference in thickness is the result of excess resin; the contribution of resin to some of the in-plane properties can be small, so it may be possible to make a property comparison ignoring this element. However, ignoring the effect of thickness on these properties, i.e. using force per unit width, shows little change to the difference in tensile modulus between each loading direction. In contrast, the difference in strength-to-failure between the two loading directions becomes more similar. There is clearly an unbalance in mechanical properties between each loading direction for 3DMG-T1 when compared to those measured from 3D-78 specimens.

![Stress-strain curve](image)

**Figure 6.3:** Stress-strain curve of 3DMG-T1 specimens quasi-statically loaded in tension along the warp-direction – these specimens were manufactured using the wet-layup method

It can be seen from the property data in Table 6.2 that by increasing the tension on the z-binder, there is a reduction in tensile modulus and an increase in the strain-to-failure, and ultimate strength, for
specimens loaded along the warp direction. In contrast, little change occurred in the mechanical performance of specimens loaded along the weft-direction, with the exception of a slight reduction in strain-to-fail. It appears that increasing the tension on the z-binder during weaving caused the mechanical properties along each loading direction to become more balanced. With little noticeable difference in cross-sectional structure between 3DMG-T1 and –T2 specimens, it is not obvious how the increase in z-binder tension has affected the mechanical performance of specimens loaded along the warp-direction.

Figure 6.3 and Figure 6.4 show the stress-strain curves for 3DMG-T1 and T2 warp-directional loaded specimens respectively. Here it can be seen that during initial loading, up until the onset of damage, the stress-strain curves of each type/direction show good reproducibility. It was mentioned in Chapter 4 that due to the structure of these composites, i.e. an alternating layup, cracks that can reach the surface will cause localised asymmetric bending. Using a strain gauge, or in this case an extensometer, the asymmetric bending due to crack formation produces instantaneous increases or decreases in strain. After the initiation of damage, the stress-strain curves show an increased scatter for warp-direction loaded specimens of both T1 and T2 materials, even if the final strength and strain-to-failure values are similar.

Comparatively, stress-strain plots of weft-direction loaded specimens shows less scatter during the entire loading, with variation mostly in the final stress/strain-to-failure (Figure 6.5 and Figure 6.6). It should be noted that for the weft-direction loaded specimens, the transverse cracks are mostly in the warp tows which are not at the surface (see section 7.2.2). The only transverse cracks that do penetrate
to the surface are those that develop through the z-binders, though these typically saturate quite quickly in quasi-static loading. Consequently, while the development of matrix cracking does cause instantaneous changes in the strain, these changes are not as large as for the warp-direction loaded specimens, and so the stress-strain curves appear smoother.

Figure 6.5: Stress-strain curve of 3DMG-T1 specimens quasi-statically loaded in tension along the weft-direction – these specimens were manufactured using the wet-layup method

Figure 6.6: Stress-strain curve of 3DMG-T2 specimens quasi-statically loaded in tension along the weft-direction – these specimens were manufactured using the wet-layup method
6.3.2. Tension-tension fatigue properties

In Chapter 4 the tensile fatigue properties of the 3D-78 material were characterised. In a similar fashion, this section presents fatigue data on the 3DMG materials for both z-binder tensions. Unlike the 3D-78 material, 3DMG was available only in relatively small quantities and it was not possible to produce an S-N curve. For purposes of comparison between these materials, most of the testing was conducted at the same peak stress –200 MPa peak stress was used here; comparisons in terms of force per unit width are also shown.

As already been mentioned above, the 3DMG and 3D-78 architectures are quite different despite having an equal number of layers in each principal direction. The differences between the two fabric architectures also seem to influence the fatigue properties. Under the fatigue loading conditions of 200 MPa peak stress, 3D-78 loaded along the warp and weft directions had average fatigue lifetimes of approximately 100,000 cycles and 25,000, respectively (see Section 4.3). By comparison, the average fatigue life of the 3DMG-T1 was approximately 10,000 cycles in both warp and weft directions Figure 6.7 (left). While the 3DMG-T1 fatigue properties are much lower than those of 3D-78, unlike 3D-78 the 3DMG-T1 lifetimes at this peak stress were similar for the warp and weft directions.

![Graph](image-url)

**Figure 6.7:** Comparison of the number of cycles to failure for warp and weft direction specimens of 3DMG-T1 (left) and 3DMG-T2 (right) fatigued with a peak stress of 200MPa; these specimens were manufactured using the wet-layup method

It is possible that the differences in the fatigue lifetime between 3D-78 and the 3DMG-T1 material could be explained by considering the effects of thickness, given that the 3D-78 specimens were slightly thinner than the 3DMG-T1 material. Using this approach the peak force per unit thickness of the 3D-78 specimens is lower than those of the 3DMG-T1. Thus, some extension to fatigue lives for each loading direction would be expected for 3D-78 specimens compared with 3DMG-T1.

For the 3DMG-T1 specimens fatigue loaded along the warp and weft direction, cycles to failure were approximately the same. By increasing the z-binder tension (i.e. for the 3DMG-T2 material), a separation of the warp and weft direction tensile fatigue lives occurred (Figure 6.7b), such that the number of cycles to failure for the warp-direction loaded specimens (Figure 6.8a) increased, whereas
weft-direction loaded specimens remained relatively unchanged (Figure 6.8b). The effect of the z-binder tension on the tensile fatigue properties is therefore similar to its effect on the quasi-static properties; there were significant changes to the warp direction properties, and only minor changes for the weft direction.

![Figure 6.8: Comparison of the number of cycles to failure for warp (left) and weft (right) direction loaded 3DMG-T1 and T2 specimens fatigued with a peak stress of 200MPa; these specimens were manufactured using the wet-layup method]

For warp-direction loaded specimens, it was showed in section 6.3.1 that there is a difference in thickness between 3DMG-T1 and -T2 specimens. Accounting for the difference in thickness, the force per unit width for the 3DMG-T1 specimens is lower than 3DMG-T2 specimens (see Figure 6.9a). This means that despite having a lower force per unit width, 3DMG-T1 specimens still failed sooner than 3DMG-T2 specimens. Even using peak initial strain, it is difficult to account for the differences in numbers of cycles to failure between 3DMG-T1 and 3DMG-T2. It is clear that increasing the z-binder tension during weaving influences the performance of 3DMG materials when loaded along the warp direction. It will be shown in Chapter 7 that the increase in fatigue life is the result of a subtle change in the way delamination damage is developed when the z-binder tension is increased.

![Figure 6.9: Comparison of the number of cycles to failure for warp direction loaded 3DMG-T1 and T2 specimens fatigued with a peak stress of 200MPa plotted in terms of force per unit width (left) and peak initial strain (right); these specimens were manufactured using the wet-layup method]
Interestingly, a difference in warp-direction fatigue performance was seen between different production runs of 3DMG-T2, as shown in Figure 6.8a. For each production run of 3DMG-T2 the quasi-static properties remained similar, as did volume fraction and thickness. Accounting for thickness, and looking at the peak initial strain, it is unclear where these differences arise (see Figure 6.9a/b). To the best of the fabric manufacture, no changes were made to the weaving process for either production run.

Figure 6.10 compares the loss of stiffness, plotted as the normalised tangent modulus, as a function of cycle number for tests conducted with a peak stress of 200 MPa for both z-binder tensions of the Manchester material; included here is a comparison of production runs for 3DMG-T2. The same data has been plotted in Figure 6.11, but as a function of cycles normalised by the number of cycles to failure. There are no significant differences in the behaviour of the various materials. However, an important observation which emerges from Figure 6.11 is that there is a limit to the overall loss of stiffness for these specimens. Generally, the loss of stiffness is approximately 22-24% of its original value before failure occurs.

Another way of analysing the fatigue data is to compare the energy dissipated per cycle. In Figure 6.12, the energy dissipated per cycle up to cycle 10,000 is plotted for all the 3DMG specimens cycled in the warp direction with a peak stress of 200 MPa. It was shown earlier that the 3DMG-T1 specimens had smaller fatigue lifetimes than 3DMG-T2 specimens. From Figure 6.12 it can be observed
that the energy dissipated per cycle for the 3DMG-T1 specimens is, on average, higher (by about 1½ to 2 times) than the 3DMG-T2 specimens during stage two of fatigue loading. During this stage, the energy dissipation per cycle remains relatively constant, lasting for much of the life of the composite. A higher constant energy dissipation indicates that there is more damage per cycle for these specimens, and it is therefore perhaps not surprising that they have shorter fatigue lifetimes. The constant energy loss probably occurs from mechanisms such as, but not limited to, friction between crack faces, heat loss, and slow growth damage. The difference in energy dissipated is also clear in Figure 6.13 where the cycle number has been normalised by the specimen lifetime.

![Figure 6.11: Comparison of the loss of stiffness (normalised tangent modulus) over the entire fatigue lifetime (cycle number normalised by to number of cycles to failure) for 3DMG-T1 and –T2 specimens fatigued with a peak stress of 200 MPa along the warp-direction; these specimens were manufactured using the wet-layup method](image)

Most of the specimens shown in Figure 6.13 failed within the gauge length of the extensometer, so that both energy dissipation data (and stiffness reduction data, as discussed above) could be collected up to final failure. For most specimens, after an initial relatively rapid reduction, the energy dissipated has a very shallow, constant positive slope. Generally, within the last 10-20% of a specimen’s fatigue life there are some noticeable sharp increases in energy dissipated, which occur relatively rapidly up to failure. As discussed in Chapter 4, it was not possible to determine the precise cause of these sudden changes. Usually at this point in the fatigue life, the matrix-dominated damage mechanisms (matrix cracking, delamination, bundle debonding) have saturated and growth is either none existent or very
slow. It is possible that the increases in energy dissipation are due to the development of fibre fractures, or fibre bundle fractures.

Figure 6.12: Energy dissipation per cycle comparison over the first 10,000 cycles for 3DMG-T1 and -T2 specimens fatigued with a peak stress of 200 MPa along the warp-direction; these specimens were manufactured using the wet-layup method

Sufficient specimens were available from the second production run of 3DMG-T2 (i.e. 3DMG-T2 PR2) so that some fatigue tests could be conducted at higher peak stresses to produce a basic stress-cycle (S-N) plot (Figure 6.14). This plot emphasises the improved fatigue behaviour in the warp direction of this material compared to 3DMG-T2 PR1 and the 3DMGT1 material. When compared with the 3D-78 S-N curve for the warp direction (see Figure 4.3), it is apparent that the 3DMG-T2 PR2 material shows less scatter in the fatigue lifetimes, in general. This may be because of the greater uniformity and regularity of the fabric structure.
Figure 6.13: Comparison of the energy dissipated per cycle over the entire fatigue lifetime (cycle number normalised by number of cycles to failure) for 3DMG-T1 and -T2 specimens fatigued with a peak stress of 200 MPa along the warp-direction; these specimens were manufactured using the wet-layup method.

Figure 6.14: S-N curve comparison for 3DMG-T1 and -T2 warp-direction fatigue loaded specimens; these specimens were manufactured using the wet-layup method.
6.4. Mechanical properties of 3DMG manufactured using VARTM

6.4.1. Introduction

As discussed in Chapter 3, the vacuum assisted resin transfer moulding (VARTM) method was used for some specimens, enabling the elimination of voids within the laminates. It was also useful to see whether the properties of the 3DNCOW composites changed when manufactured using a more industrial method. Of the 3DMG material, two laminates were produced using VARTM from the second production run (i.e. PR2) of 3DMG-T2. Sufficient material was available to produce warp direction specimens which were tested under both quasi-static and cyclic loading.

6.4.2. Quasi-static tensile mechanical properties

Some basic mechanical properties of the 3DMG-T2 PR2 material manufactured using VARTM are shown in Table 6.3. It is interesting to note that while the manufacturing method was identical, the fibre volume fraction of each VARTM panel was quite different, as were the laminate thicknesses. Taking into account the effect of thickness, it can be seen that both panels have similar properties. Although the 3DMG-T2 specimens manufactured via the wet-layup method had many voids, the quasi-static tensile properties (see 6.3.1) show good agreement with the VARTM 3DMG-T2 properties.

Table 6.3: Quasi-static tensile properties of 3DMG-T2 specimens loaded along the warp-direction; these specimens are manufactured using VARTM

<table>
<thead>
<tr>
<th>Specimen</th>
<th>E (GPa)</th>
<th>E (kN/mm)</th>
<th>σ_{ULT} (MPa)</th>
<th>σ_{ULT} (N/mm)</th>
<th>ε_{max} (%)</th>
<th>Vf (%)</th>
<th>t (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>VARTM 1</td>
<td>24.1 ± 0.4</td>
<td>63.4 ± 1.3</td>
<td>538 ± 10</td>
<td>1411 ± 11</td>
<td>3.1 ± 0.1</td>
<td>46.9 ± 0.9</td>
<td>2.62 ± 0.06</td>
</tr>
<tr>
<td>VARTM 2</td>
<td>23.2 ± 0.3</td>
<td>64.8 ± 0.5</td>
<td>493 ± 16</td>
<td>1374 ± 16</td>
<td>3.1 ± 0.1</td>
<td>43.5 ± 1.1</td>
<td>2.79 ± 0.06</td>
</tr>
</tbody>
</table>

6.4.3. Tension-tension fatigue properties

As is shown in Table 6.3, two VARTM panels were produced using 3DMG-T2. Each panel was produced from the same production run and used the same manufacturing method. However, both the fibre volume fraction and the thickness of each panel are different, with no cross-over in scatter. Due to the differences in panel thickness, peak stresses for fatigue testing were determined by normalising the effect of thickness. Therefore, peak stresses of 190 MPa and 180 MPa were used for test specimens from VARTM1 and VARTM2 respectively; these are both approximately 500 N/mm. This is the same force per unit width value as the 3DMG-T2 wet-layup specimens fatigued with a peak stress of 200 MPa.
Figure 6.15 shows the fatigue lifetimes plotted in terms of force per unit width, peak stress and peak initial strain. In Figure 6.15a, specimens from both VARTM panels have the same force per unit width, and the results show specimens from VARTM2 have generally longer fatigue lives. When analysing the same data in terms of peak stress (Figure 6.15b), and initial peak strain (Figure 6.15c), this difference is maintained, with the lower peak stress, and the lower peak initial strain, giving longer fatigue lives. This would indicate, at least for VARTM specimens, that small changes in thickness have little effect on the overall fatigue lifetime. However, comparing VARTM fatigue specimens with wet-layup specimens which were also manufactured using 3DMG-T2 PR2, this inference does not work. The wet-layup specimens were thinner, yet when loaded with a peak fatigue stress of 200 MPa (peak force per unit width of 500 N/mm), the number of cycles to failure were on average greater than VARTM1 and less than VARTM2 specimens (see Figure 6.16). With the number of cycles to failure for the wet-layup specimens being greater than for VARTM1, it would appear that the manufacturing method influences the overall fatigue performance. This is probably related to the presence of voids and different surface topology in the wet-layup specimens, which are not present in the VARTM specimens.

![Figure 6.15](https://example.com/fig6_15.png)

**Figure 6.15: Various S-N plots for 3DMG-T2 warp-direction loaded specimens (VARTM manufacture): a) Force/unit width vs. log number of cycles to failure; b) Peak stress vs. log number of cycles to failure; c) Peak initial strain vs. log number of cycles to failure**

Examining the stiffness reduction of specimens fatigued from each of the VARTM panels (Figure 6.17), the rate of loss of stiffness is approximately the same for both panels and the loss of stiffness over the first 20,000 cycles appears independent of the eventual number of cycles to failure for each specimen. Figure 6.18 shows that the stiffness reduction normalised over the lifetime of VARTM1
specimens are similar, and not very different from the VARTM2 specimens. The total stiffness lost prior to failure is again between 22-24%, the same stiffness loss noted for specimens manufactured using the wet-layup method.

Figure 6.19 shows the energy dissipated over the first 20,000 fatigue cycles for each of the VARTM specimens. Here it can be seen that there is a clear distinction in energy dissipated for specimens tested from each VARTM panel. Specimens from VARTM1 (cycled at a peak stress of 190 MPa) dissipate more energy per cycle than VARTM2 specimens (cycled at a peak stress of 180 MPa); this is additional evidence that normalising by using force/unit width is not the best method of comparing specimens in fatigue. Since the number of cycles to failure for VARTM1 specimens were on average less than VARTM2 specimens, it would be expected that the energy dissipated per cycle would be higher for VARTM1 specimens, as found.

![Figure 6.16: Comparison of wet-layup and VARTM manufactured 3DMG-T2 PR2 specimens fatigue loaded with a peak force per unit width of 500 N/mm](image-url)
During the initial fatigue cycles, the energy dissipated per cycle for the wet-layup manufactured specimens starts high and decreases steadily over many cycles before reaching an almost steady-state value (Figure 6.12). The same trend can be seen in VARTM manufactured specimens (Figure 6.19), although the initial energy dissipation per cycle is much lower, and the steady decrease in energy dissipation per cycle occurs at a lower rate.

In Figure 6.20, the energy dissipation data is plotted against cycle number normalised by the lifetime of the specimens. The trend shown here is similar to that seen earlier for the wet lay-up specimens, where the energy dissipation increases at a constant rate for most of the fatigue life, and then within the last 10-20%, more rapid changes occur until final failure. Many of these specimens failed outside the limits of the extensometer, so that final failure was not recorded for all of the specimens. However, specimens that did fail within the extensometer limits do show similarities with the wet lay-up specimens in the lead-up to failure.
Figure 6.18: Comparison of the loss of stiffness (normalised tangent modulus) over the entire fatigue lifetime (cycle number normalised by to number of cycles to failure) for 3DMG-T2 specimens fatigued with a peak stress of 180 MPa and 190 MPa along the warp-direction; these specimens were manufactured using the wet-layup method.

Figure 6.19: Energy dissipation per cycle comparison over the first 10,000 cycles for 3DMG–T2 specimens fatigued with a peak stress of 180 MPa and 190 MPa along the warp-direction; these specimens were manufactured using VARTM.
In this chapter, an all-glass 3D orthogonal weave manufactured by the University of Manchester, called 3DMG, has been characterised in terms of its mechanical performance. This material was manufactured with two different z-binder tensions, with the suffix T1 and T2 added to differentiate between the lower and higher z-binder tensions respectively. Both materials were tested in quasi-static tension and tension-tension fatigue. In addition, two different methods of manufacturing the 3DMG-T2 specimens were used, with properties obtained from each compared to each other to see if there were any differences; a wet-layup method and VARTM were used to manufacture the specimens. It should be noted that only warp-direction testing was done on VARTM specimens. From the testing undertaken it was found:

- Under quasi-static tensile loading that there some differences in the mechanical properties between 3DMG-T1 and 3DMG-T2. With a lower z-binder tension (3DMG-T1) it was found that the tensile modulus and strength was greater, and tensile strain-to-failure lower, along the warp-direction than the weft-direction. By increasing the z-binder tension it was found that the warp tensile modulus decreased, becoming similar to the tensile modulus along the weft-direction. Additionally, the tensile strain-to-failure along the warp direction increased. The strength to failure of the warp and weft-direction did not change with an increase in the z-binder
tension. Weft-direction properties were also found to change little with the change in z-binder tension. This indicates that the z-binder only really influences the mechanical performance along the warp-direction.

- The VARTM panels manufactured varied in thickness, and were generally thicker than the wet-layup panels. Assuming any increase in thickness was the result of an increased resin content, properties were compared in terms of force per unit width. Using this method of comparison, it was noted that there was no difference in the quasi-static tensile mechanical properties between the wet-layup and VARTM 3DMG-T2 specimens.

- Tension-tension fatigue lifetimes of 3DMG-T1 warp and weft-directions specimens fatigue loaded with a peak stress of 200 MPa were the same. Increasing the z-binder tension (3DMG-T2) showed an increase in the fatigue lifetimes of the warp-direction, while the weft-direction remained the same as the 3DMG-T1 weft-direction specimen. It is interesting to note that for despite having the same peak force per unit width, peak initial strains, and fibre volume fraction, two panels of wet-layup 3DMG-T2 produced drastically different numbers of cycles to failure. A similar trend of increasing number of cycles to failure was noted between VARTM panels, however in this case the peak initial strain was slightly lower for specimens with the higher number of cycles to failure.

- Comparison of the loss of tensile stiffness over the lifetime of 3DMG-T1 and 3DMG-T2 showed little difference between both materials. For both materials, the total loss of stiffness by failure was approximately 22-24%. While the loss of stiffness was similar for both materials, it was observed that the energy dissipated per cycle for 3DMG-T1 specimens was 1 ½ to 2 times higher than the 3DMG-T2 specimens during stage 2; this is where the energy dissipation per cycle trend is a shallow incline for most of the fatigue life. A higher energy dissipation usually results in fewer cycles to failure, which was the case for 3DMG-T1 compared with 3DMG-T2.
Chapter 7

Damage development in 3DMG – wet-layup and VARTM manufactured specimens

7.1. Introduction

As seen in the 3D-78 specimens described in Chapter 5, the combination of epoxy resin and 3DMG fabric led to very transparent specimens. This allowed the damage development during various loadings to be characterised. It is the aim of this chapter to describe the damage development in the 3DMG material and make comparisons with the 3TEX (3D-78) material. During this work, the tension of the z-binders was increased in the 3DMG material and two different manufacturing methods were used (wet lay-up and VARTM); variations in damage development between the different specimen routes are described, where appropriate.

7.2. Quasi-static loading damage development

7.2.1. Warp-direction

Figure 7.1 shows the damage development in a 3DMG-T1 (low binder tension) specimen loaded quasi-statically in tension along the warp direction. 3DMG-T1 specimens were only manufactured using the wet-layup method. It is useful to note at the outset that all of the 3D-78 damage mechanisms described in Chapter 5 are found to develop in the 3DMG-T1 specimens. For instance, damage does not develop in 3D-78 specimens until a strain of about 0.6/0.7%, whereas it is clear in Figure 7.1b that at 0.7% strain, damage has already developed along the resin-rich channels between adjacent weft tows. In fact, damage appears to initiate in 3DMG-T1 specimens as early as 0.4% strain in form of very fine transverse matrix cracks. Although damage initiates earlier in 3DMG-T1 specimens compared with 3D-78 specimens, the amount of damage present in both materials appears similar by approximately 1.2% strain.

When comparing 3DMG-T1 and 3D-78 warp-direction loaded specimens, differences in the amount of damage, notably matrix cracking, are observed. Comparing Figure 7.1e and for 3DMG-T1 specimens and Figure 5.3f in Chapter 5 for 3D-78 specimens, it is clear 3DMG-T1 specimens have a higher crack density. In 3D-78 specimens, matrix cracks will generally develop within the centre of a weft tow and multiple cracks would only occasionally develop within the same weft tow. By contrast, it is quite common for there to be two cracks per weft tow in 3DMG-T1 specimens (Figure 7.1e and f).
Figure 7.1: Quasi-static damage development of 3DMG-T1 warp-direction specimen (wet-layup manufacture): a) unloaded; b) 193MPa/0.73% strain; c) 313MPa/1.21% strain; d) 408MPa/1.67% strain; e) 560MPa/2.5% strain; f) failed – 575MPa/2.6% strain

Figure 7.2: Comparison of weft tow cross-sectional geometry in; a) 3D-78; b) 3DMG-T1; and, c) 3DMG-T2; the scale is the same for each cross-section

The development of multiple cracks across the weft tow width in 3DMG-T1 specimens is probably due to the influence of the z-binder on the weft tow cross-sections. As described in Section 6.2, the path of the z-binder in 3DMG material is more orthogonal than the z-binder path in 3D-78. It is the path of the z-binder that influences the cross-sectional shape of the weft tows, in particular the surface weft tows. In 3D-78 specimens, the surface weft tows have a semi-circular cross-section and while the surface weft tow cross-sections are still semi-circular in 3DMG specimens, they square off slightly more than the 3D-78 specimens (see Figure 7.2). In addition, Figure 7.2 shows that the central weft tow in 3DMG-T1 is thicker by approximately 40% and more uniform in cross-section that the central weft tow in 3D-78 specimens. Consequently, based on the fact that first ply failure occurs at a
lower strain in a thicker ply [73], it is possible to understand why matrix cracks occur earlier in 3DMG-T1 specimens than in 3D-78 specimens.

Turning to the z-binder damage, for warp-direction loaded specimens of both 3D-78 and 3DMG material, the through-thickness portion of the z-binder tends to debond from the surrounding material. In the 3D-78 specimens, these debonds were contained to the limits of the z-binder. However, for the 3DMG-T1 specimens, damage extends from many debonds with what looks like a crack extending along a 45° plane away from the z-binder. This damage can be seen in Figure 7.1c, but is more obvious in Figure 7.1d through to Figure 7.1f. Initial crack growth appears angled along one orientation, but under continued loading, the cracks deviate and link up with similar cracks from neighbouring z-binder debonds probably through resin-rich regions through the thickness; viewed from the surface, these cracks follow a curved path (Figure 7.1e). This damage appears to be restricted to the boundary of the resin-rich channel between adjacent weft tows.

Figure 7.3: Quasi-static damage development of 3DMG-T2 warp-direction specimen (wet-layup manufacture): a) unloaded; b) 182MPa/0.78% strain; c) 308MPa/1.6% strain; d) 407MPa/2.24% strain; e) 515MPa/2.98% strain; f) failed – 523MPa/3.09% strain

Figure 7.3 shows the development of damage in a warp-direction 3DMG-T2 (high binder tension) specimen loaded in quasi-static tension; the damage development is very similar to 3DMG-T1
specimens. Each of the photographs in Figure 7.3 represent a similar stress to the images in Figure 7.3, but the strains are different because increasing the z-binder tension has reduced the warp-direction Young’s modulus from about 30 GPa to about 25 GPa (see Section 6.3).

One of the differences between the damage developed in 3DMG-T1 and T2 specimens is related to the growth of damage around the z-binder debond along the through-thickness portion of the z-binder. It was shown above, that for 3DMG-T1 specimens, there appear to be cracks that emanate from the z-binder debond at about 45°, and then link with neighbouring, similar cracks. While this damage does still occur in 3DMG-T2 specimens, the extent of the damage is reduced. Looking at Figure 7.3d, there are fewer regions with this type of crack extending from the z-binder.

It should be noted here that the 3DMG-T1 and T2 specimens that are discussed above were manufactured using the wet-layup method. Figure 7.4 shows a 3DMG-T2 specimen that was manufactured using VARTM where it can be seen that essentially the same damage mechanisms occurred as in the wet-layup specimens.

![Figure 7.4: Quasi-static damage development of 3DMG-T2 warp-direction specimen (VARTM manufacture): a) unloaded; b) 174MPa/ 0.73% strain; c) 315MPa/ 1.71% strain; d) 409MPa/ 2.29% strain; e) 527MPa/ 3.12% strain; f) failed – 544MPa/ 3.26% strain](image-url)
7.2.2. Weft-direction

Figure 7.5 shows the damage development in a 3DMG-T1 specimen (wet lay-up) loaded quasi-statically in tension along the weft direction. Damage initiates as transverse matrix/resin cracks within both warp tows and z-binders. The z-binders are located between neighbouring transverse (warp) tows, where there are also large resin-rich pockets that enable rapid propagation of the transverse cracks associated with the z-binders through the thickness and across the width of the specimen. In contrast, the warp tow transverse matrix cracks grow much more slowly (with increasing strain) than those along the z-binder/resin-rich pocket region. Comparing the initiation of damage in 3DMG-T1 warp- and weft-direction loaded specimens, it was observed that damage initiated at about 0.4% strain for both loading directions. Damage for weft-direction specimens initiated in the form for small transverse cracks along the free edges of the specimens. Figure 7.1b and Figure 7.5b show specimens loaded, in the warp and weft direction respectively, to a similar strain and it is clear that more damage present at this strain in the weft-direction loaded specimen.

Figure 7.5: Quasi-static damage development of 3DMG-T1 weft-direction specimen (wet-layup manufacture): a) unloaded; b) 178MPa/0.78% strain; c) 253MPa/1.23% strain; d) 361MPa/1.88% strain; e) 460MPa/2.47% strain; f) failed – 528MPa/2.92% strain. The yellow dash lines indicate the boundary of some z-binders.
For weft-direction loading it is more common to observe multiple cracks in the transverse warp tows than individual cracks; this is more evident when cracking has saturated and the specimen approaches final failure (Figure 7.5d). This behaviour is similar to the weft direction loaded 3D-78 specimens (Section 5.3.2.), where it was noted that this is probably due to the uniform rectangular cross section of the warp tows. It is interesting to note that in the 3DMG-T1 specimens, many of the warp tow transverse matrix cracks extend completely across the width of the specimen, whereas in 3D-78 specimens, tended to be many smaller cracks (see Figure 5.4).

![Warp tow Z-binder](image)

**Figure 7.5**: Quasi-static damage development of 3DMG-T2 weft-direction specimen (wet-layup manufacture): a) unloaded; b) 178MPa/0.73% strain; c) 313MPa/1.21% strain; d) 408MPa/1.67% strain; e) 560MPa/2.5% strain; f) failed – 575MPa/2.6% strain

Weft tow longitudinal splits develop near failure (Figure 7.5e). The longitudinal splits develop in the weft tows below the crown of a z-binder and extend a short distance either side of this z-binder region. In Figure 7.5e, yellow dashed lines have been used to show the boundaries of z-binders in order to make the longitudinal split growth easily observable.
Weft-direction loaded 3DMG-T2 specimens (Figure 7.6) show the same damage development as in the 3DMG-T1 specimens. This is probably because the increased z-binder tension has little impact on the weft direction properties in general (see Tables 2 in Chapter 6).

7.3. Tension-tension fatigue damage development

Due to the limited availability of material, fatigue tests were mostly conducted at one peak stress level, 200MPa; where necessary, the peak stress was adjusted to maintain similar force per unit widths to account for any major differences in thickness. During most of the tests, photographs of the specimens were taken to monitor the damage development over the fatigue life. Some specimens were then sectioned to compare in-situ, plan view damage observed through the camera with the through-thickness damage.

Since production runs of fabric were produced with different z-binder tensions, as well as the use of different manufacturing methods, it is useful to compare the damage developed in each to see if there are any noticeable changes. It is the aim of the following sections to provide a detailed account of the damage developed during fatigue loading of 3DMG material in all forms. Photographs of damage development in 3DMG-T1, 3DMG-T2 PR1, 3DMG-T2 PR2 and 3DMG-T2 PR2 (VARTM) specimens after various cycles are shown in Figure 7.7 -Figure 7.10 for the warp-direction and Figure 7.13 and Figure 7.14 for the weft-direction. Each of the photographs, except the last one in each figure, shows damage after the same number of fatigue cycles, with the last image showing the damage close to final failure.

7.3.1. Warp-direction

Figure 7.7a - Figure 7.10a show the warp-direction specimens prior to fatigue cycling. There are numerous voids present in the wet-layup specimens (Figure 7.7 - Figure 7.9), as discussed in Section 3.3; the voids are mostly located within the resin-rich pockets between neighbouring warp tows (the location of the z-binders). In contrast, the VARTM specimens are void-free (Figure 7.10). The development of damage will be discussed using the various fatigue cycles to indicate changes in their growth.

At cycle 25 (Figure 7.7 - Figure 7.10b), various forms of damage have already developed. Transverse cracks within the resin-rich channels, as well as debonding of the through-thickness portion of the z-binders, have clearly become well established. Weft tow transverse matrix cracks have developed to a much greater extent in all of the 3DMG-T2 specimens than in the 3DMG-T1 specimen. It was shown in Section 6.3.2 that initial (first cycle) fatigue strains in T2 specimens were on average higher than the first cycle fatigue strain in T1 specimens due to the lower Young's modulus of the T2 specimen. Hence, this higher strain probably leads to a more rapid development of the weft tow transverse matrix cracks. An interesting point to note for the early (and later) fatigue damage is that,
while there are many voids in each of the wet-layup manufactured specimens, they do not seem to be initiators of damage. Some damage does go through some voids, but they do not appear to be damage instigators. The effect of voids is discussed further in Section 7.4.

Figure 7.7: Tension-tension fatigue damage development of 3DMG-T1 warp direction specimen loaded with a peak stress of 200MPa (wet-layup manufacture): a) Unloaded, b) 25 cycles, c) 200 cycles, d) 500 cycles, e) 1000 cycles, f) 3000 cycles, g) 7800 cycles

At cycle 200 (Figure 7.7 - Figure 7.10c), damage in the form of transverse cracks and z-binder debonding has developed further. At the same time, micro-delamination cracks begin to form from several of the weft-tow cracks already present. It was seen in Chapter 5 that, for the warp-direction fatigue loading of 3D-78 material, micro-delamination cracks generally initiated from weft tow matrix
cracks that developed centrally along the weft tow width. While many micro-delamination cracks still developed in this way in the 3DMG specimens, there are also numerous micro-delamination cracks that developed from weft tow cracks which are located toward the edges of the weft tows. Examples of micro-delamination cracks developing from the edge of a weft tow have been highlighted in Figure 7.7, Figure 7.9 and Figure 7.10(d-h). It can generally be observed that all micro-delamination cracks tend to have one well-defined edge corresponding to the warp tow boundary. During cycling, micro-delamination cracks that develop from the edge of a weft tow extend over the entire width of the adjacent warp tow and toward the centre of the weft tow. The growth of these micro-delamination cracks forms an arc-type shape. Occasionally, two of these delamination cracks developing from both weft tow boundaries, an example of which can be seen in Figure 7.9f and g. In Figure 7.9f and g, pink dashed lines indicate the boundaries of a weft tow, while a red ellipse has been used to highlight the micro-delaminations extending from either boundary; an enlargement of this region has also been provided.

As a consequence of the transparency of the specimens, it is possible to observe some of the micro-delamination cracks through the thickness, including and on the remote side of the specimen, so that some delaminations appear to overlap. An example is highlighted in yellow in Figure 7.7f of what appears to be two overlapping micro-delamination cracks; however, these are at different locations through the thickness.

It is interesting to note that micro-delamination damage developing from cracks at the edge of a weft tow forms more extensively in 3DMG-T1 than in 3DMG-T2. While some micro-delamination damage still develops from these types of cracks in 3DMG-T2, most develops toward the centre of a weft tow. The difference in micro-delamination development is probably related to the tension placed on the z-binder during weaving. While the z-binder constrains movement in the z-axis, a looser tension will allow for easier separation of the warp/weft interfaces, notably in this case toward the edge of the weft tow boundary. By increasing the tension on the z-binder, the layer separation becomes more restricted, since the z-binder is now pinching down harder on the warp/weft interfaces. In 3DMG-T2, the pinching enables delaminations to develop more readily toward the centre of the weft tows.

It was shown in 3D-78 specimens that these central weft tow transverse cracks, due to the compressive force from the z-binder under tensile loading, would lead to specimen failure as the result of a stress concentration build-up. Delamination damage at the base of these cracks was found to reduce the size of this stress concentration, allowing it to only develop on one side of a warp tow. In 3DMG-T1, transverse cracks still develop toward the centre of the weft tows, while delamination damage develops from the edge. In contrast, micro-delamination damage in 3DMG-T2 develops more similarly to 3D-78. As a result, the stress concentration at the base of centrally located weft tow transverse cracks in 3DMG-T1 specimens will probably cause failure to occur more readily than in 3DMG-T2 specimens.
During the initial fatigue loading of 3DMG specimens, the resin-rich channel between adjacent weft tows becomes distinct from the weft tows where fibre/matrix debonding produces a darker image. It has already been pointed out that transverse cracks form along these channels, extending across the specimen width. Around cycle 1000 in Figure 7.7 - Figure 7.10 it can be seen that the visibility of many of these resin-rich channels starts to reduce because of micro-delamination growth across the channels. In Figure 7.7 - Figure 7.10 yellow dashed lines have been added to indicate the location of a resin-rich
channel. Additionally, a yellow ellipse has been used to highlight the micro-delamination cracks extending across the channel.

Figure 7.9: Tension-tension fatigue damage development of 3DMG-T2 PR2 warp direction specimen loaded with a peak stress of 200MPa (wet-layup manufacture): a) Unloaded, b) 25 cycles, c) 200 cycles, d) 500 cycles, e) 1000 cycles, f) 5000 cycles, g) 10,000 cycles, h) 121,500
For a better understanding of the geometry of the damage, Figure 7.11 and Figure 7.12 show many examples of the various damage mechanisms observed in the plan view images. In Figure 7.11, transverse cracks can be seen in each of the weft tows, as well as through the resin-rich channel between weft tow boundaries. Several transverse cracks line up relatively well through the thickness, indicating that they are probably a singular crack that has grown into each weft tow. Other transverse cracks appear independent of other transverse cracks around them suggesting that these have not propagated much further than a single tow. In addition, Figure 7.11 shows micro-delamination damage along a number of different interfaces. Some of the micro-delaminations here are the same as those shown previously.
for warp-direction fatigued 3D-78 specimens (see Chapter 5), seen here extending from a weft tow transverse matrix crack along the interface between the surface weft tow and adjacent warp tow. As mention above, the transparency of the resin-rich channel reduced with an increase in fatigue cycles, which was said to be the result of micro-delamination damage across this region. Examples of this damage can be seen in Figure 7.11, with these delaminations occurring along the interface between the top surface of the warp tow as it crosses through the resin-rich channel. This damage initiates from transverse cracks that develop through the resin-rich channel.

![Diagram of damage mechanisms](image)

**Figure 7.11: Cross-section along warp plane of warp direction specimen fatigue loaded with a peak stress of 190MPa (VARTM manufacture) – showing damage**

A cross-section through a z-binder of a warp-direction fatigue loaded VARTM specimen can be seen in Figure 7.12. Again, like Figure 7.11, Figure 7.12 shows many of the damage mechanisms observed in the plan-view photographs, including; through-thickness weft tow transverse matrix cracks, transverse cracks through resin-rich regions and channels, and z-binder debonds. It can be observed in this micrograph that some of the transverse cracks, especially those that go through the resin-rich pockets, do not always follow a straight path and can curve toward weft tow transverse matrix cracks.

Finally, one damage mechanism that has only been seen to develop in the fatigue of 3DMG-T2 PR2 specimens is warp tow longitudinal splits (see Figure 7.9h). Compared to the other plan view damage development images shown in this chapter, this specimen had the longest fatigue life at 121,843 cycles. Interestingly, warp tow longitudinal splits were seen to develop during warp-direction fatigue loading of 3D-78 specimens having a similarly large number of cycles to failure. It therefore seems likely that very long fatigue lives are required for this type of damage to develop.

In summary, all of the types of 3DMG composite material produced, showed similar amounts of damage over their fatigue lifetimes for warp-direction loading. There are no obvious differences that can explain the large variation in the number of cycles to failure.
Weft-direction

Due to availability of material, testing along the weft direction in fatigue was much more limited. Of the 3DMG material provided by the University of Manchester, fatigue testing of weft direction specimens was only conducted on 3DMG-T1 and 3DMG-T2 PR1 (both wet lay-up). Like warp-direction fatigue loading, photographs were taken during testing of the weft direction specimens, with cross-sectional micrographs produced in order to investigate the damage further. Figure 7.13 and Figure 7.14 show the damage development for 3DMG-T1 and 3DMG-T2 PR1 specimens respectively, fatigue loaded along the weft direction with a peak stress of 200MPa. As mentioned previously, the presence of voids is indicative of the wet-layup method of manufacture.

It has been shown in previous sections of this chapter that the mechanical properties, both quasi-static and fatigue, for weft direction loaded specimens with the different z-binder tensions are very similar and this is also true with regards to the development of fatigue damage. Comparison of Figure 7.13b (for 3DMG-T1) and Figure 7.14b (3DMG-T2 PR1) show that within the first 25 cycles, the damage developed is similar in both materials, with multiple transverse matrix cracks, both within the warp tows and the z-binders developing over the length of each specimen. At this stage in the fatigue life, the main difference between the two materials is a small increase in number of z-binder debonds in 3DMG-T1 (Figure 7.13b).

During the early stages of fatigue loading, the z-binder debonding in 3DMG-T2 specimens more closely resembles 3D-78 weft-direction loaded specimens. In contrast, the early stage debonding around the z-binder region in 3DMG-T1 specimens is limited to the edge of the longitudinal weft tows, extending at an angle slightly along the length of the weft tow away from the z-binder. With continued cycling, these debonds grow over the width of the z-binder region, as well as toward the centre of a weft tow. However, rather than growing straight toward the centre of a weft tow and remaining confined
within the boundary of the z-binder, as previously seen in 3D-78 specimens, opposite facing debonds from either side of a weft tow appear to curve toward each other (Figure 7.13d). While this debonding behaviour is most apparent in 3DMG-T1 specimens, there are some instances of this behaviour occurring in 3DMG-T2 specimens. The higher z-binder tension in the 3DMG-T2 specimens probably restricts this debonding from developing the same way as it did in the 3DMG-T1 specimens (Figure 7.14), restricting it more to the confines of the z-binder.

Figure 7.13: Tension-tension fatigue damage development of 3DMG-T1 weft direction specimen loaded with a peak stress of 200MPa (wet-layup manufacture): a) Unloaded, b) 25 cycles, c) 200 cycles, d) 500 cycles, e) 1000 cycles, f) 2000 cycles, g) 4000 cycles, h) 8000 cycles
Figure 7.14: Tension-tension fatigue damage development of 3DMG-T2 PR1 weft direction specimen loaded with a peak stress of 200MPa (wet-layup manufacture): a) Unloaded, b) 25 cycles, c) 200 cycles, d) 500 cycles, e) 1000 cycles, f) 2000 cycles, g) 4000 cycles, h) 10000 cycles

In both 3DMG-T1 and T2 specimens, micro-delamination cracks develop between the weft and warp tows. These micro-delamination cracks appear to initiate as early as 200 cycles in both specimens (Figure 7.13c and Figure 7.14c). To differentiate between the z-binder debonds and micro-delamination cracks, the location of some z-binders have been highlighted with yellow dashed lines in Figure 7.13 and Figure 7.14; the shape of the micro-delamination cracks is very similar to the z-binder debonds. Like the z-binder debonds, the micro-delamination cracks are widest along each weft tow edge and seem to become narrower toward the weft tow centre. The growth of some micro-delaminations have
been highlighted in Figure 7.13c-f and Figure 7.14c-h. It is interesting to note that while these delaminations initiate after a similar number of cycles in both materials, the 3DMG-T2 micro-delaminations appear to grow at a slightly slower rate. Examples of these micro-delamination cracks have been highlighted in cross-sections of fatigue loaded weft specimens in Figure 7.15 and Figure 7.16. It can be seen here that these micro-delaminations do not develop along any warp tow/weft tow interface preferentially. This is especially notable in the 3DMG-T2 cross section in Figure 7.16 where micro-delaminations have developed all warp tow/weft tow interfaces from most of the transverse matrix cracks. It should be noted that the difference in amount of damage present between Figure 7.15 and Figure 7.16 is because the cross-section in Figure 7.15 was taken from a specimen stopped after 2000 cycles, whereas the specimen used for Figure 7.16 had failed; also, the cross-section taken in Figure 7.16 was not near the ultimate failure site of the specimen, where it was assumed ultimate failure did not influence the damage accumulated.

Figure 7.15: Cross-section of a 3DMG-T1 specimen fatigue loaded along the weft-direction with a peak stress of 200 MPa for 2000 cycles before the test was stopped and the specimen sectioned

Figure 7.16: Cross-section of a 3DMG-T2 PR1 specimen fatigue loaded along the weft-direction with a peak stress of 200 MPa until failure. This cross-section was taken some distance away from the failure site.
7.4. Effect of Voids

In almost all the photographs taken of 3DMG specimens manufactured using the wet layup method, there is a significant number of voids present. Unlike many composite layups, 3D woven composites have a large number of resin-rich pockets where air can be trapped, thus creating the voids. In the 3D woven composites manufactured in this work, voids generally did not develop between fibres or within fibre tows, only in the resin-rich pockets.

Figure 7.17 and Figure 7.18 show cross-sections of 3DMG specimens fatigue loaded along the warp direction. It can be seen in these images that there are multiple through-thickness transverse cracks that propagate not only through the transverse weft tows, but also through the resin-rich pockets between them. Some of the transverse cracks can be seen to intersect with voids. It is interesting to note that these transverse cracks do not seem to originate from the voids, but, if passing close, deviate toward the void. Transverse cracks usually initiate as a consequence of the strain magnification between fibres due to the mismatch in the fibre and matrix moduli. For unidirectional glass/epoxy composites with a fibre volume fraction of 0.6, loaded perpendicular to the fibres, the strain magnification factor between the fibres is approximately 6, assuming a regular fibre array [74]. However, when the crack propagates into the resin-rich pocket, there is a strain magnification of 3 near the void that will attract the crack front. In Figure 7.18 there are two voids very close to one another. The effect of strain magnification around the voids is well demonstrated here as there is a crack that runs between both voids. This crack can be seen deviating toward the larger void since it has an overall closer proximity.

Figure 7.17: Cross-section of a warp-direction 3DMG-T2 PRI specimen fatigue loaded with a peak stress of 200 MPa – this cross-section shows damage in and around voids within the resin-rich regions in these specimens
**Figure 7.18:** Cross-section of a warp-direction 3DMG-T2 PRI specimen fatigue loaded with a peak stress of 200 MPa – this cross-section shows damage in and around voids within the resin-rich regions in these specimens.

### 7.5. Concluding remarks

In this chapter, it has been shown that the general failure mechanisms discussed in chapter 5 for the 3D-78 material have been observed in each of the materials (3DMG-T1 and -T2) described in this chapter. In addition, no significant difference in the damage developed was noted for specimens that were manufactured using the wet-layup or VARTM methods. The main difference between specimens manufactured using these methods was the void content.

For warp-direction loaded specimens loaded in quasi-static tension the damage developed the most common damage mechanisms was transverse cracks in both the transverse weft tows and through the resin-rich channel between neighbouring weft tows. Unlike the 3D-78 material, it was quite common to see multiple transverse cracks develop within all three weft tows through the thickness. Multiple weft tow transverse cracks can develop because the central weft tow is thicker and more rectangular than the surface weft tows, which are semi-circular in cross-section. Other damage mechanisms included z-binder debonding and limited amounts of micro-delamination.

Weft-direction quasi-static loading results in the development of many transverse cracks within warp tows. Both warp tows have a rectangular cross-section, and so cracks can develop independently in both tows. Transverse cracks also develop through z-binders. It is not clear that any z-binder debonding, or micro-delamination cracks growth during quasi-static loading along the weft direction. Near to failure longitudinal weft tow splits form under the z-binder and extend a small distance either side of the z-binder.

In a similar manner to quasi-static loading, the damage development during tensile fatigue loading along the warp-direction consisted of transverse cracks in the weft tows and along the resin-rich channel, debonding of the through-thickness portion of the z-binder from surrounding material, and micro-delamination damage. Micro-delamination growth from transverse cracks located centrally within a weft tow would occur along the surface weft/ adjacent warp tow interface and form a shield-
like shape, just like the 3D-78 material described in Chapter 5. However, in these materials micro-
delamination growth was found in many cases to develop from transverse cracks near the boundary of
a weft tow. These delaminations would have a similar shape as the central micro-delaminations, but
would only grow from one side of the transverse crack, i.e. along the warp/weft tow interface. It is
interesting to note that only one of these types of delamination would develop along these interface,
either from the edge growing inward, or from the centre of a weft tow. In 3DMG-T1 specimens,
delamination growth occurred preferentially from the edge of the weft tow, where delamination growth
was more central in 3DMG-T2 specimens. This difference in delamination growth is probably due to
the tension placed on the z-binder, and has a direct impact on the fatigue life of specimens. Centrally
grown delaminations seem to improve the fatigue life. Unlike 3D-78, delamination damage would
develop from the transverse cracks in the resin-rich channel between weft tow stacks. These
delaminations would generally develop between warp tow interface nearest the specimen surface and
the resin-rich region, growth during cycling reaching warp/weft tow interfaces.

Again, the damage development during weft-direction fatigue loading was found to be similar
in many ways to quasi-static loading, with many transverse matrix cracks developing through warp
tows and z-binders. Additional damage during fatigue loading included the debonding of the z-binder
crown from the surface weft tow, and micro-delamination damage. The shape of the z-binder debonds
in this work were seen to be triangular, with a wide base along the edge of the weft tow, reaching a
point toward the centre of the surface weft tow. The micro-delamination damage seen to develop in
these specimens had a similar geometry to the z-binder debonds, generally developing from a central
warp tow crack. It can be noted that multiple cracks would develop per warp tow, but delaminations
only seemed to develop from one of these.
Chapter 8
Mechanical characterisation and damage
development of the hybrid 3D weave – 3DMHyb

8.1. Introduction

Whilst much of this work has been focused on all-glass based 3D orthogonal woven composites, the effect of fibre hybridisation on the tensile quasi-static and fatigue properties has also explored. Like the fabric reported in Chapter 7, the hybrid 3DNCOW described in this chapter was manufactured by the University of Manchester; it will be referred to as 3DMHyb (3D Manchester Hybrid) to differentiate it from the various glass-fibre fabrics that have been used throughout this work. The aim of this chapter is to characterise the effect of fibre hybridisation on quasi-static and fatigue loading under tensile conditions, and compare the findings with the all-glass equivalent presented previously in Chapter 6 and 7.

8.2. 3DMHyb fibre architecture

Like all the 3D woven fabrics characterised thus far, the hybrid fabric (3DMHyb) has three weft tow layers, two warp tow layers, and an orthogonally interlacing z-binder that runs parallel to the warp direction. The glass fibre tows used for the warp and weft tow layers were the same as those used in the 3DMG fabric. Fibre hybridisation was achieved by replacing the glass fibre z-binder tows with carbon fibre z-binder tows. For comparative purposes, the z-binder tension during weaving was fixed to the higher z-binder tension used for the 3DMG material, i.e. 120 gf.

Figure 8.1 shows the fibre architecture of 3DMHyb taken along three cross-sectional planes. Two x-z planes were used to show the architecture of the warp tows (Figure 8.1a) and z-binder (Figure 8.1b), while only one y-z plane has been used to indicate the weft tow architecture (Figure 8.1c). The structure of the 3DMHyb material is comparable to the 3DMG structure (see Chapter 6) whereby there is a consistent regularity to the structure. Both warp tow layers are very straight along the fibre axis and have reasonably uniform rectangular cross-sections. For all three weft tow layers there is only a small amount of crimp present in regions where the z-binder crosses over the surface weft tows. Two voids can be seen in Figure 8.1b, positioned within the resin-rich pockets between weft tow layers; this is typical for wet-layup manufacture and similar to the 3DNCOW composites shown in previous chapters.
Using a resin burn-off method proposed by [75], the volume fraction of each component within the hybrid fabric was determined for both the wet-layup laminate and two laminates manufactured using VARTM. Table 8.1 shows the total fibre volume content, as well as the glass and carbon fibre volume contents. There is clearly a high level of repeatability between each of the panels. It can be seen in Table 8.1 that the carbon z-binders occupy approximately three to four percent of the total volume.

![Figure 8.1: 3DMHyb fibre architectures indicating the position of each component within the structure, as well as the thickness of each fibre tow; a) x-z plane through warps tows; b) x-z plane through z-binders; c) y-z plane through weft tows.](image)

It is interesting to noted that when the 3DMHyb fabrics were manufactured into composite panels, using both wet-layup and VARTM manufacturing techniques, they were generally thicker than the all-glass equivalent; hybrid composite panel thicknesses can be seen in Table 8.2. It is currently unclear what causes this difference since both weaving and composite manufacture techniques were unchanged from those used for the 3DMG material. The effect of thickness on various properties will be discussed further in subsequent sections of this chapter.
Table 8.1: Fibre volume fraction of the various 3DMHyb panels manufactured.

<table>
<thead>
<tr>
<th>Manufacture Technique</th>
<th>Total Vf (%)</th>
<th>Glass Content Vf (%)</th>
<th>Carbon Content Vf (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wet-layup</td>
<td>46.4 ± 1.2</td>
<td>42.7 ± 0.8</td>
<td>3.7 ± 0.7</td>
</tr>
<tr>
<td>VARTM 1</td>
<td>46.7 ± 0.7</td>
<td>43.6 ± 0.8</td>
<td>3.0 ± 0.6</td>
</tr>
<tr>
<td>VARTM 2</td>
<td>45.1 ± 1.1</td>
<td>41.3 ± 0.7</td>
<td>3.8 ± 0.7</td>
</tr>
</tbody>
</table>

Whilst the thickness of 3DMHyb has become slightly increased in comparison to 3DMG, the size of the unit cell in each direction remains relatively unchanged. Unit cell measurements of multiple cross-sections have shown the unit cell of the 3DMHyb material to be approximately 6.33 ± 0.09 mm along the warp-direction, and 6.18 ± 0.04 mm along the weft-direction, which is very similar to the unit cell size of the 3DMG-T2 material (see Section 6.2.)

8.3. Mechanical properties of 3DMHyb

Assessment of the mechanical performance of the 3DMHyb material was conducted using quasi-static tensile and tension-tension fatigue tests; data obtained was compared with 3DMG-T2 results reported in Chapter 6. Although testing was conducted along both principal directions, there was a greater focus placed on warp-direction loading. Tests were conducted on laminates manufactured using both the wet-layup and VARTM techniques.

8.3.1. Quasi-static tensile mechanical properties

In Table 8.2 the mechanical properties of both 3DMG-T2 and 3DMHyb wet-layup manufactured specimens can be seen for comparison. For 3DMHyb, it can be seen that the tensile modulus along the warp-direction is higher than along the weft-direction. This is in direct contrast to the 3DMG-T2 material, where the modulus was found to be similar in both directions. It is possible that the carbon fibre z-binder influences the stiffness of the material more along the warp-direction than the weft-direction. The carbon z-binders may also provide additional strength along the warp-direction as the ultimate strength of the warp-direction is slightly greater than the weft-direction.

It is interesting to note that the tensile modulus and the ultimate strength of the 3DMG-T2 material along the warp and weft-direction is greater than along the warp and weft-direction in 3DMHyb. By normalising these results to a fibre volume fraction of 50 percent (Table 8.2), it can be seen that the 3DMG-T2 and 3DMHyb weft-direction strength and stiffness are basically the same. For the warp-direction, the tensile modulus and strength-to-failure of 3DMG-T2 remains higher than 3DMHyb when measured using this metric, though the overall difference, when scatter is taken into account, is minimised. Additionally, the 3DMHyb material, when consolidated, was approximately 10% thicker than the 3DMG-T2 specimens. It can be assumed that the difference in thickness is the result of an increased resin content in the hybrid specimens; this is verified by the lower fibre volume fraction in 3DMHyb panels compared with 3DMG-T2 (see Table 8.3). Taking the thickness into
account, and using force per unit width, similar trends seen with fibre volume fraction normalisation are seen here for both the warp and weft loading directions when both materials are compared to each other. As shown in Table 8.3, the warp and weft-direction strain-to-failure for both 3DMG-T2 and 3DMHyb are essentially the same. This indicates that for the wet-layup manufactured specimens, the addition of a carbon fibre z-binder make little difference to the overall mechanical properties.

Table 8.2: Tensile modulus and strength measurements of 3DMG-T2 and 3DMHyb material manufactured using the wet-layup and VARTM techniques. Both the tensile modulus and ultimate strength are provided in standard form, as well as normalised with respect to 50% fibre volume fraction (50%Vf) and in terms of force per unit width (nt = normalised against thickness).

<table>
<thead>
<tr>
<th>Specimen (WL)</th>
<th>E  (GPa)</th>
<th>Eₜ₅₀VF  (GPa)</th>
<th>Eₙₜ  (kN/mm)</th>
<th>σᵿₜ  (MPa)</th>
<th>σᵿₜ₅₀VF  (MPa)</th>
<th>σᵿₜₙₜ  (N/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3DMG-T2</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Warp</td>
<td>25.5 ± 0.8</td>
<td>26.1 ± 0.8</td>
<td>64.0 ± 2.0</td>
<td>550 ± 17</td>
<td>564 ± 17</td>
<td>1383 ± 46</td>
</tr>
<tr>
<td>Weft</td>
<td>25.3 ± 1.0</td>
<td>24.9 ± 1.0</td>
<td>61.6 ± 0.9</td>
<td>510 ± 15</td>
<td>502 ± 15</td>
<td>1244 ± 44</td>
</tr>
<tr>
<td>3DMHyb</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Warp</td>
<td>24.3 ± 0.4</td>
<td>25.3 ± 0.2</td>
<td>65.4 ± 0.5</td>
<td>482 ± 7</td>
<td>519 ± 8</td>
<td>1341 ± 21</td>
</tr>
<tr>
<td>Weft</td>
<td>22.6 ± 0.7</td>
<td>24.5 ± 0.8</td>
<td>61.1 ± 1.9</td>
<td>469 ± 27</td>
<td>509 ± 29</td>
<td>1271 ± 69</td>
</tr>
<tr>
<td>Specimen (VARTM)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3DMG-T2</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Warp1</td>
<td>24.1 ± 0.4</td>
<td>25.7 ± 0.4</td>
<td>63.4 ± 1.3</td>
<td>538 ± 10</td>
<td>574 ± 11</td>
<td>1411 ± 11</td>
</tr>
<tr>
<td>Warp2</td>
<td>23.2 ± 0.3</td>
<td>26.7 ± 0.3</td>
<td>64.8 ± 0.5</td>
<td>493 ± 16</td>
<td>567 ± 19</td>
<td>1374 ± 16</td>
</tr>
<tr>
<td>3DMHyb</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Warp1</td>
<td>24.7 ± 1.3</td>
<td>26.5 ± 1.4</td>
<td>66.5 ± 0.2</td>
<td>493 ± 29</td>
<td>528 ± 31</td>
<td>1310 ± 52</td>
</tr>
<tr>
<td>Warp2</td>
<td>22.9 ± 0.8</td>
<td>25.4 ± 0.9</td>
<td>66.4 ± 2.1</td>
<td>474 ± 10</td>
<td>526 ± 11</td>
<td>1381 ± 33</td>
</tr>
</tbody>
</table>

Only warp-direction quasi-static tension testing was conducted on VARTM manufactured 3DMHyb specimens. It can be seen in Table 8.2 that there is a difference in tensile modulus and strength-to-failure for each 3DMHyb VARTM manufactured panel. When these values are normalised to a 50 percent fibre volume fraction, or to force per unit width, then the noted differences become negligible; the same trend is also seen for the 3DMG-T2 VARTM specimens. Comparing 3DMHyb to 3DMG-T2 when normalised to 50 percent fibre volume fraction, the tensile modulus for both materials is very similar. However, while the strength-to-failure is not overly too dissimilar for both materials under standard comparison, when normalised to a 50 percent fibre volume fraction, the strength-to-failure of the 3DMG-T2 specimens is on average higher than 3DMHyb. Again, comparing 3DMG-T2 and 3DMHyb by ignoring thickness, similar trends to those observed above can be seen. Additionally, as seen in Table 8.3, the strain-to-failure measured in the 3DMHyb test specimens is lower than in the 3DMG-T2 test specimens.

It appears clear from these observations that the all-glass fibre material 3DMG loaded along the warp-direction has slightly superior static strengths-to-failure than 3DMHyb, whereas the tensile modulus does not change that much. This indicates that changing the z-binder material from glass to
carbon does not have much impact on the low stress/low strain static performance of this 3DNCOW composite structure. However, when loaded toward failure, the carbon-fibre z-binder proves to be detrimental to the static strength. While carbon fibres have higher static strength axially than glass fibres, the vast majority of each carbon fibre z-binder is not in-plane with the warp loading direction, therefore cannot provide any extra support to the in-plane properties. Since carbon fibre z-binders appear detrimental to the static strength, it could be assumed that damage development of 3DMHyb may be different to 3DMG, such that final failure is induced at lower loads in the hybrid material. As shown later in this chapter, under static loading the hybrid test specimens do not appear to show any clear difference in damage developed when compared to the all-glass counterpart that could account for this noted change in strength-to-failure.

Table 8.3: Strain-to-failure, average specimen thickness, and fibre volume fraction for both 3DMG-T2 and 3DMHyb manufactured using both the wet-layup and VARTM techniques.

<table>
<thead>
<tr>
<th>Specimen (WL)</th>
<th>e_{\text{max}} (%)</th>
<th>t (mm)</th>
<th>VF (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3DMG-T2</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Warp</td>
<td>3.0 ± 0.1</td>
<td>2.48 ± 0.03</td>
<td>48.8 ± 1.5</td>
</tr>
<tr>
<td>Weft</td>
<td>2.8 ± 0.1</td>
<td>2.44 ± 0.05</td>
<td>50.8 ± 0.5</td>
</tr>
<tr>
<td>3DMHyb</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Warp</td>
<td>2.9 ± 0.1</td>
<td>2.75 ± 0.05</td>
<td>46.4 ± 1.2</td>
</tr>
<tr>
<td>Weft</td>
<td>2.9 ± 0.1</td>
<td>2.64 ± 0.07</td>
<td>46.1 ± 0.7</td>
</tr>
<tr>
<td>Specimen (VARTM)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3DMG-T2</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Warp1</td>
<td>3.1 ± 0.1</td>
<td>2.62 ± 0.02</td>
<td>46.9 ± 0.9</td>
</tr>
<tr>
<td>Warp2</td>
<td>3.1 ± 0.1</td>
<td>2.79 ± 0.06</td>
<td>43.5 ± 1.1</td>
</tr>
<tr>
<td>3DMHyb</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Warp1</td>
<td>2.7 ± 0.1</td>
<td>2.66 ± 0.08</td>
<td>46.7 ± 0.7</td>
</tr>
<tr>
<td>Warp2</td>
<td>2.8 ± 0.0</td>
<td>2.89 ± 0.03</td>
<td>45.1 ± 1.1</td>
</tr>
</tbody>
</table>

Figure 8.2: and Figure 8.3: show the stress-strain plots for the wet-layup and VARTM manufactured 3DMHyb specimens respectively. The scatter for wet-layup manufactured test specimens during loading is low up to 1.3% strain, after which a small divergence of the curves is seen up to failure. For VARTM specimens, scatter between curves is greater than the wet-layup specimens, with divergence of the curves occurring around 1% strain; scatter at failure is much larger for the VARTM specimens than for the wet-layup specimens. Some of the variation between specimens after 1% strain is related to the surface development of transverse cracks toward each surface causing local asymmetric bending and immediate jumps in strain that alter the progression of each curve; the effect of asymmetric bending was described in Chapter 4. In Figure 8.4:, for the hybrid wet-layup specimens testing in the weft-direction, this effect is seen mostly during the very early stages of loading, as a result of cracks developing along the z-binder/resin-rich pockets between neighbouring warp tows. As before, the smoother curves are related to the transverse matrix cracks within the warp tows that are not situated at the surface for weft-direction specimens.
Figure 8.2: Stress-strain curve comparison of 3DMHyb wet-layup manufactured specimens loaded in tension along the warp-direction.

Figure 8.3: Stress-strain curve comparison of 3DMHyb VARTM manufactured specimens loaded in tension along the warp-direction.
Figure 8.4: Stress-strain curve comparison of 3DMHyb wet-layup manufactured specimens loaded in tension along the weft-direction

8.3.2. Tension-tension fatigue properties

In this section, the tensile fatigue properties of the 3DMHyb material will be characterised, with comparisons made to 3DMG-T2. Due in part to the limited supply of hybrid material, and the differences in thickness of manufactured laminates, most of the fatigue tests were conducted with a peak force per unit width of approximately 500 N/mm along the warp-direction. A brief attempt was made to more fully characterise 3DMHyb material by conducting some fatigue tests with higher loadings, thus producing a partial S-N curve. However, this was only done for specimens loaded along the warp-direction.

8.3.2.1. Fatigue lifetimes of wet-layup manufactured 3DMHyb specimens

With a peak force per unit width of approximately 500N/mm, specimens manufactured using the wet-layup method were tested along both the warp and weft direction and the results are shown in Figure 8.5a. From this plot, it is clear that the fatigue life of the warp-direction loaded specimens is more than ten times that of the weft-direction loaded specimens. Since the thickness of warp and weft-direction specimens were different, to achieve a force per unit width of 500N/mm a peak stress of 180 MPa and 190 MPa were used respectively (Figure 8.5b). Additionally, the initial peak strain is slightly lower for the warp-direction loaded specimens than the weft-direction loaded specimens (Figure 8.5c). It is unlikely that a slight increase in peak initial strain in the warp specimens will yield a dramatic decrease in number of cycles to failure such that they match those measured along the weft-direction.
Therefore, it can be concluded that the fatigue life of warp-direction loaded specimens is greater than the weft direction. The difference in performance seen here between warp-direction and weft-direction loaded specimens for 3DMHyb is consistent with that seen for both the 3D-78 and 3DMG materials, where the warp-direction fatigue lifetimes were higher than for the weft-direction.

As mentioned above, some tests were done at different peak loads in order to produce a partial S-N curve characterisation of the warp-direction. Three different peak loads were chosen such that the force per unit width values would correspond with those used in testing the 3DMG material. Figure 8.6 is an S-N curve comparison of 3DMG-T2 and 3DMHyb materials in terms of peak stress. Included in this graph is the individual ultimate strengths of each material, along with log-linear trend lines for each material. Both the 3DMHyb and 3DMG-T2 trend line fit with the data well, with the 3DMHyb trend line having a slightly lower slope than the 3DMG-T2. However, when these data points are plotted in terms of force per unit width and initial peak strain (see Figure 8.7 and Figure 8.8) the difference between 3DMG and 3DMHyb appear to be almost negligible. However, considering the results around 500 N/mm in Figure 8.7, and 0.8% in Figure 8.8, it can be seen that most of the 3DMHyb specimens have longer fatigue lives than all but one 3DMG-T2 specimen. Although the data is rather limited, it could be inferred that the hybrid specimens perform better in fatigue than the all-glass specimens at these low stress values. This is supported by the 3DMHyb data points for specimens fatigued with a peak force per unit width of approximately 550 N/mm, which show cycles to failure similar to the 3DMG-T2 specimens fatigued with at 500 N/mm.

![Graphs](Figure 8.5: Plots of various peak loading parameters against number of cycles to failure for 3DMHyb wet-layup manufactured specimens; a) Force per unit width; b) peak stress; c) peak initial strain)
Figure 8.6: S-N curve comparison of 3DMG-T2 and 3DMHyb specimens; included here is the ultimate strengths of both materials and a log-linear trend line.

Figure 8.7: Peak force per unit width plotted against the number of cycles to failure for 3DMG-T2 and 3DMHyb specimens; included here is the ultimate strengths of both materials and a log-linear trend line.
8.3.2.2. Fatigue lifetimes of VARTM manufactured 3DMHyb specimens

Fatigue testing using the VARTM panels yielded some interesting results. A number of specimens from two different 3DMG-T2 and 3DMHyb panels were tested. For these VARTM specimens, tests were conducted only with a peak force per unit width of 500 N/mm. Despite using the same force per unit width, the fatigue lives of specimens from the second panel of each material type were longer than that of the first (Figure 8.9a). As a result of the different thicknesses of each panel, the peak stresses varied slightly. When the number of cycles to failure are plotted in terms of peak stress (see Figure 8.9b), the data seems to form a shallow trend; for a small decrease in peak stress there is a large increase in number of cycles to failure. The same trend can be seen in Figure 8.9c for the data plotted against initial peak strain. It can be inferred from these results that if the peak stresses and initial strains had been the same for all specimens, the number of cycles to failure may have been similar.

However, some of the 3DMG-T2 and 3DMHyb specimens were not only tested with the same peak force per unit width, but also with the same peak stress (190 MPa). Despite the slightly lower initial peak strains in the 3DMHyb-PR1 specimens compared with 3DMG-T2 PR2 VARTM1 specimens, the number of cycles to failure were clearly higher for the hybrid specimens by just over a factor of two. A similar trend can be seen for the 175 MPa 3DMHyb-PR2 specimens when compared with the 180 MPa 3DMG-T2 specimens. In this case, the peak stresses are similar and the peak initial strains for each material falls within the scatter, and still the hybrid specimens perform better by a factor of two or more. Many of the 3DMHyb specimens tested at 175 MPa peak stress were run outs, with the tests being...
stopped at approximately 1.7 million cycles. While damage still initiates readily during the early stage fatigue of these specimens, it is possible that these peak stresses are near the fatigue limit of the material.

![Figure 8.9: Plots of various peak loading parameters against number of cycles to failure. Each of these compare the performance of 3DMG-T2 and 3DMHyb specimens manufactured using VARTM; a) Force per unit width; b) peak stress; c) peak initial strain](image)

8.3.2.3. Energy dissipation per cycle

It has been shown thus far that for the wet-layup manufactured specimens, the fatigue performance is similar for both 3DMG-T2 and 3DMHyb specimens and the energy dissipated per cycle confirms this similarity. In Figure 8.10, the energy dissipated per cycle is plotted against a normalised cycle number in order to compare 3DMG-T2 and 3DMHyb wet-layup manufactured specimens fatigue loaded with peak stresses of 200 MPa and 180 MPa respectively. The comparison of these two peak stresses were chosen as they are equivalent to a force per unit width of 500 N/mm. It can be seen here that despite a difference in number of cycles to failure, the energy dissipated over the fatigue lives of these specimens is very similar. Not only is the magnitude of energy dissipated similar for all specimens shown in Figure 8.10, but the shallow incline of energy dissipated per normalised cycle is also very similar. This shows that the addition of a carbon-z-binder makes little difference to the mechanisms by which energy is dissipated throughout the fatigue life, except within the final stage. Figure 8.11 shows that the same trend can be seen when plotting the energy dissipated against actual cycle number, rather than normalised cycle number. Here, the graph shows up to cycle 20,000, where it can be seen that the amount of energy dissipated per cycle is the same for 3DMG-T2 specimens and 3DMHyb specimens.
This means that any differences between the hybrid specimens and all-glass specimens must be related to accumulation of damage toward the end of their fatigue life.

![Figure 8.10: Comparison of the energy dissipated per cycle plotted against normalised cycle number for 3DMG-T2 and 3DMHyb specimens fatigue loaded with peaks stresses of 200 MPa and 180 MPa respectively](image)

It can be noted that during the final forty percent of the fatigue life, the 3DMG-T2 specimens start to show an increase in energy dissipated, whereas the 3DMHyb specimens do not. This is related to the location of failure and the position of the extensometer during testing. The strain measured by the extensometer is sensitive to the location of the extensometer in relation to damage in the specimen. When damage, such as that leading to final failure of the specimen, occurs within the gauge length of the extensometer, sudden increases in strain are measured; this increases hysteresis area and measured energy dissipation. Failure of the wet-layup hybrid specimens mostly occurred outside the extensometer gauge length and near the grips.

As mentioned previously, all of the VARTM manufactured specimens were fatigue tested with a peak force per unit width of approximately 500 N/mm; this resulted in different peak stresses. Looking at the energy dissipated per normalised cycle for these specimens (see Figure 8.12), many of the curves can be seen to overlap, showing little effect of a carbon fibre z-binder. However, differences can be noted between specimens loaded at different peak stresses. The specimens with the highest energy dissipated per normalised cycle are the 3DMG-T2 190 MPa and 3DMHyb-PR1 190 MPa specimens; the average energy dissipated appears slightly higher for the 3DMG-T2 specimens. From this graph, it
appears that the 3DMHyb-PR2 specimens have the lowest average energy dissipated per normalised cycle. This matches well with the number of cycles to failure of these specimens presented in Figure 8.9. From the specimens shown in Figure 8.9, 3DMHyb-PR2 175MPa specimens had the highest number of cycles to failure, while the 3DMG-T2 190 MPa had the lowest. Higher energy dissipated per cycle generally results in a smaller number of cycles to failure during tensile fatigue. In Figure 8.13 the energy dissipated is plotted against actual cycle number, focused on the first 20,000 cycles. It is clear that the specimens with the shortest and longest fatigue lives (see section 8.3.2.2) also show on average the highest and lowest energy dissipated per cycle respectively.

![Figure 8.11: Comparison of the energy dissipated per cycle for 3DMG-T2 and 3DMHyb specimens fatigue loaded with peaks stresses of 200 MPa and 180 MPa respectively – each curve is only showing the first 20,000 cycles](image)

Many of the VARTM specimens plotted in Figure 8.12 failed within the gauge length of the extensometer, and therefore show large increases in energy dissipated per cycle as the specimens near the end of their fatigue life. It would useful to be able to use this data to determine what damage is causing such increases in energy dissipated, and how near to failure the specimen is. However, Figure 8.12 is a good example of how variable specimen failure can be. While this data may be useful to assess the amount of energy dissipated during loading, it is not easy to determine the energy markers associated with various mechanisms and therefore determine the proximity to failure.
Figure 8.12: Comparison of energy dissipation per cycle plotted against normalised cycle for 3DMG-T2 and 3DMHyb specimens manufactured using VARTM and fatigued at various peak stresses, each equivalent to 500 N/mm.

Figure 8.13: Comparison of energy dissipation per cycle for 3DMG-T2 and 3DMHyb specimens manufactured using VARTM and fatigued at various peak stresses, each equivalent to 500 N/mm – shown here is the first 20,000 cycles.
8.3.2.4. Loss of stiffness during fatigue loading of wet-layup and VARTM manufactured 3DMHyb specimens

Another way to compare the hybrid 3D orthogonal weave to the all-glass version is to compare the loss of material stiffness per cycle. Figure 8.14 shows a normalised stiffness reduction as a function of cycle number up to cycle 20,000 for the wet-layup manufactured specimens. Most of the damage input into the specimens occurs over a relatively short number of cycles in comparison to the overall number of cycles to failure; therefore, it is useful to compare the performance of specimens over this range. The loss of material stiffness for these materials clearly occurs at the same rate during the early stages of fatigue. Normalising the cycle number by the number of cycles to failure, Figure 8.15 indicates that the initial loss of stiffness (stage 1) is slightly more gradual 3DMG-T2 specimens than for the 3DMHyb specimens. In contrast, stage 2, which has a much shallower loss of stiffness, is similar for both materials. Since hybrid specimens have been found to have a slight increase in fatigue life compared to the all-glass specimens, it is possible to infer that further development of various damage mechanisms, which lead to the ultimate failure of the specimen, are somewhat suppressed by the carbon z-binder.

![Comparison of loss of tensile modulus (normalised) per cycle for 3DMG-T2 and 3DMHyb specimens manufactured using the wet-layup method and fatigue loaded with peak stresses of 200 MPa and 180 MPa respectively; each peak stress is equivalent to 500 N/mm – shown here is the first 20,000 cycles](image)

For the VARTM manufactured specimens the effect of cyclic loading on the reduction in stiffness for 3DMG-T2 and 3DMHyb is slightly different to the wet-layup manufactured specimens. In
the following figures, the loss of stiffness curves for all the VARTM tests have been split such that 3DMG-T2 and 3DMHyb 190 MPa tests (Figure 8.16 and Figure 8.18) and 3DMG-T2 180 MPa and 3DMHyb 175 MPa tests (Figure 8.17 and Figure 8.19) can be compared separately; these groupings have been chosen due to the similarities in the number of cycles to failure between the various stresses. Looking at the early stage (first 20,000 cycles) of fatigue loading for the 3DMG-T2 and 3DMHyb VARTM manufactured specimens in Figure 8.16 and Figure 8.17, the initial drop in stiffness of the all-glass specimens is generally greater over fewer cycles than the hybrid specimens. Despite the more rapid drop in stiffness for the 3DMG-T2 specimens, Figure 8.16 shows that the total stiffness loss becomes very similar for both materials after approximately 5,000 to 10,000 cycles. The same is not true in Figure 8.17, where the stiffness loss remains, on average, lower for the 3DMG-T2 specimens as opposed to 3DMHyb. When these curves are viewed with regards to the proportion of fatigue life (normalised cycles), the rate of loss of stiffness for both materials remain similar for the entire fatigue life (see Figure 8.18 and Figure 8.19), with failure occurring after a similar loss of material stiffness.

![Figure 8.15: Comparison of loss of tensile modulus (normalised) plotted against normalised cycle number for 3DMG-T2 and 3DMHyb specimens manufactured using the wet-layup method and fatigue loaded with peak stresses of 200 MPa and 180 MPa respectively; each peak stress is equivalent to 500 N/mm](image-url)
Figure 8.16: Comparison of loss of tensile modulus (normalised) per cycle for 3DMG-T2 and 3DMHyb specimens manufactured using VARTM and fatigue loaded with peak stresses of 190 MPa; the peak stress is equivalent to 500 N/mm – shown here is the first 20,000 cycles.

Figure 8.17: Comparison of loss of tensile modulus (normalised) per cycle for 3DMG-T2 and 3DMHyb specimens manufactured using VARTM and fatigue loaded with peak stresses of 180 MPa and 175 MPa respectively; each peak stress is equivalent to 500 N/mm. Only the first 20,000 cycles are shown here.
Figure 8.18: Comparison of loss of tensile modulus (normalised) plotted against normalised cycle number for 3DMG-T2 and 3DMHyb specimens manufactured VARTM and fatigue loaded with a peak stress of 190 MPa; this peak stress is equivalent to 500 N/mm

Figure 8.19: Comparison of loss of tensile modulus (normalised) plotted against normalised cycle number for 3DMG-T2 and 3DMHyb specimens manufactured VARTM and fatigue loaded with peak stresses of 180 MPa and 175 MPa respectively; each peak stress is equivalent to 500 N/mm
To summarise, there are no large differences in the stiffness reduction curves of 3DMG-T2 and 3DMHyb materials, for both wet-layup and VARTM manufactured specimens, so this method of analysis does not provide an obvious reason for the differences noted in the fatigue life. However, as shown earlier, there are clear differences in the energy dissipated per cycle for each of these materials. The energy dissipated per cycle for the hybrid specimens was generally lower than in the all-glass specimens. During fatigue loading, it is possible that the carbon fibre z-binders suppress, more than the glass fibres z-binders, further development of mechanisms leading to the ultimate failure of the specimens. The use of energy dissipation curves as a method for comparison of composites with similar structures could potentially assist in determining what mechanisms drive failure.

8.4 Damage development in 3DMHyb

It has been shown in previous chapters that the transparency of the glass-fibre specimens makes them ideal for monitoring damage development during various forms of loading. For the most part, the same is true for the hybrid specimens presented in this chapter. However, due to the dark colouration of the carbon fibre z-binders, it is more difficult to see damage that develops in regions surrounding them, such in various resin-rich regions. With some minor manipulation of the images (i.e. adjusting the contrast and brightness) it is possible to discern some damage in these regions.

8.4.1 Damage development during quasi-static tensile loading

8.4.1.1 Warp-direction

In Figure 8.20 the damage development of a wet-layup manufactured 3DMHyb specimen loaded in quasi-static tension along the warp direction can be seen. Each image represents a different stress/strain for the specimen up to fracture of the specimen. Figure 8.20a shows the specimen in its unloaded state, where the locations of the warp, weft, and z-binder tows have been highlighted. Since the z-binder is black it is much easier to see where the z-binder crosses the near surface (toward the camera lens) and the back surface, as well as where it traverses through the thickness of the material. It is interesting to note that the z-binder is quite narrow where it goes through the thickness of the material, but becomes wider as it reaches the centre of the surface weft tow. This is probably related to tension on the binder and the pressure placed on the fabric, by either weights or vacuum, during the curing. If there is enough free movement in the fabric when plates and weights, or vacuum pressure, are placed on the fabric, then fibres in the z-binder will spread. It is believed that where the z-binder crosses the surface there will be some portion of the z-binder acting along the loading direction and it will provide small support to the longitudinal tows in this loading direction. However, when the z-binder tow spreads to the shape seen in Figure 8.20a, many fibres within the tow will not be orientated parallel to the loading axis and may not provide significant added reinforcement. The same spreading was shown in micrographs of the 3DMG material, but was not so clearly seen in the surface view photographs taken during loading.
Figure 8.20b shows the specimen loaded to 0.62% strain or 134 MPa. Damage initiated in the form of a transverse crack along the resin-rich channel between neighbouring weft tows. In these specimens, transverse cracks initiated more readily at lower overall strain than transverse cracks that develop in the weft tows. An example of this can be seen in Figure 8.20c (1.03% strain/205MPa), where many of the resin-rich channels pictured have transverse cracks, while transverse matrix cracks in the weft tows are still relatively limited.

Figure 8.20: Damage development of a 3DMHyb specimen loaded along the warp-direction in quasi-static tension; a) unloaded; b) 134 MPa, 0.62% strain; c) 205 MPa, 1.03% strain; d) 287 MPa, 1.47% strain; e) 356 MPa, 1.87% strain; f) 420 MPa, 2.28% strain; g) 482 MPa, 2.71% strain; h) 489 MPa, 2.78% strain

In previous chapters it was suggested that the number, and position, of weft tow transverse matrix cracks that develop are related to the thickness and cross-sectional geometry of the surface weft
tows compared to the central weft tow. In 3DMG specimens it was observed that multiple transverse crack developed through a stack of weft tows, probably initiating within the central weft tow since this was generally thicker and had a more uniform rectangular cross-section than the surface weft tows. In the hybrid specimens, many of the same geometric features have been noted (see section 8.2.), where the central weft tow is thicker than the surface weft tow. However, it can be observed in Figure 8.20c-h that there were many regions where only a single weft tow transverse matrix crack developed, and only a few regions with two transverse cracks. Comparing 3DMG-T2 quasi-static tensile specimens with 3DMHyb specimens, the number of transverse matrix cracks appears greater in the 3DMG-T2 specimens. In the hybrid specimens there is a greater number of single weft tow matrix cracks, whereas in the 3DMG-T2 specimen there was a higher proportion of double cracks weft tow.

In Figure 8.20c, the initiation of debonding of the through-thickness portion of some z-binders can be seen. These debonds develop from the transverse cracks in the resin-rich channels that cross paths with the z-binder. It is not easy to see the z-binder debond in Figure 8.20c, so an example has been highlighted and enlarged. In addition to debonds, it can be seen in Figure 8.20d that micro-delaminations develop in two locations. The first is located along an interface between a weft and warp tow layer, most likely a surface weft tow and corresponding warp tow. An example of this type of micro-delamination has been circled in red and enlarged in Figure 8.20d-f. The second can be seen to have developed from a transverse crack along the resin channel; the resin-rich channel and micro-delamination along it has been highlighted using a dashed yellow line and ellipses. Here, the micro-delamination occurs along the interface between a weft tow and the surrounding matrix.

8.4.1.2. Weft-direction

Figure 8.21 shows the damage development of a weft-direction 3DMHyb specimen loaded in quasi-static tension. In Figure 8.21a, the unloaded state of the specimen can be seen, with the locations of warp, weft, and z-binder tows highlighted. Due to the colour of the z-binder relative to the resin/glass fibre combination, and the focus of these photographs, it is possible to determine in which surface each z-binder crown resides.

It can be seen in Figure 8.21b that by approximately 130 MPa or 0.58% strain, damage has already begun to develop in the form of transverse matrix cracks. In weft-direction loaded specimens, transverse cracks develop within warp tows and z-binders; an example of each has been highlighted in Figure 8.21b. Since there are large resin-rich regions surrounding the z-binder, the transverse cracks that develop within the z-binder also penetrate through these resin-rich regions. Due to the z-binders being made of carbon fibres, it is difficult to see the progression of z-binder transverse cracks along the width of the specimen; the portion a z-binder closest to the near surface obscures much of the transverse cracks in this region.
Growth of the warp tow transverse matrix cracks generally favours initiation from the edge of the specimen. This can clearly be seen in Figure 8.21c where multiple warp tow transverse matrix cracks have developed. There are a number of warp tow transverse cracks that span the full width of the specimen, and many which are either still growing from, or just beginning to develop along, the specimen edge. In these specimens, it appears to be quite common for two cracks to develop within the warp tows; an example can be seen in Figure 8.21d. While some of these warp tow transverse matrix cracks extend across the entire width of the specimen, other develop grow until they cross paths, at which point growth is terminated because of stress shielding around each crack. The development of
multiple transverse cracks within a warp tow is probably related to the consistent rectangular geometry of the warp tows, similar to 3DMG specimens. Unlike the case of warp-direction quasi-static loading of the 3DMHyb material, it is not clear that any micro-delaminations develop along warp/weft tow interfaces.

As loading is continued, diffuse damage appears to develop in the z-binder region surrounding the z-binder/resin-rich region transverse cracks, as highlighted in Figure 8.21e. It is believed that this is due to the debonding of the z-binder from the surface weft tows, as observed for the all-glass 3D weaves. Again, due to the colouration of the z-binder relative to the glass/resin combination, this can only be observed for every other z-binder crown region across the width.

8.4.2 Damage development during tension-tension fatigue loading

8.4.2.1 Warp-direction

To assess the damage development of the 3DMHyb material under tensile fatigue loading, photographs of the specimen were taken at various cycles over the fatigue life of different specimens. In Figure 8.22, the damage development for a specimen fatigue loaded with a peak stress of 180 MPa along the warp-direction can be seen; each image represents the state of the specimen when unloaded, after 25, 200, 500, 1000, 5000, 10000, and finally 270,000 cycles. It can be noted here that this specimen was manufactured using the wet-layup method, hence the presence of voids highlighted in Figure 8.22a.

Within 25 cycles a large amount of damage has already developed in the hybrid specimen. In Figure 8.22b transverse cracks in some weft tows and resin-rich channels between adjacent weft tows have formed. At this stage the transverse cracking is more developed along the resin-rich channels than within the weft tows, with most channels containing a crack. In the resin channels, the transverse cracks cover the entire width of the specimen, either as a single long crack or as multiple cracks (usually only two) that meet somewhere over the width. In addition to the transverse cracks along the resin-rich channel, z-binder debonding of the through-thickness portion of the z-binder has begun to develop at the intersection with many of these cracks. It is quite hard to see the z-binder debonds because of the colour of the z-binder, however an example has been highlighted in Figure 8.22b and the image contrast adjusted such that it becomes visible.

By cycle 200 (Figure 8.22c) the number of weft tow transverse cracks has grown, and all the resin-rich channels have transverse cracks running along them. Just like the quasi-statically loaded 3DMHyb specimen, many of the weft tow transverse matrix cracks appear to develop near the centre of the weft tows. Due to the slow growth of these cracks, initiation can occur anywhere over the specimen width, which result in more than one crack initiating along the same weft tow. During continued loading of this specimen, it can be seen that many weft tow transverse cracks have developed, with growth stopping shortly after crossing the path of another weft tow transverse cracks; examples have been highlighted in Figure 8.22f and g.
As well as the further development of weft tow transverse cracks, Figure 8.22c also shows the initial development of micro-delaminations from these cracks along a warp/weft tow interface. In this image the highlighted micro-delamination is small and can be seen curving off one side of the transverse crack. In Figure 8.22d the same highlighted micro-delamination has grown; this micro-delamination has still only developed off one side of the transverse crack, and is widest along the Warp tow edge, reaching a point along the other edge. Where the micro-delamination reaches a point also corresponds with a binder crown that is clearly restricting its growth, much like previous specimens; the binder at this location is much darker and more in focus, indicating that it is closer to the top of the surface. From continued growth of this micro-delamination (see Figure 8.22e-h) it can be seen that its shape is very similar to the micro-delaminations seen in both 3DMG and 3D-78 specimens previously.

Figure 8.22: Damage development during tension-tension warp-direction fatigue loading with a peak stress of 180 MPa of a 3DMHyb specimen; this stress is equivalent to 500 N/mm. This specimen was manufactured using the wet-layup method. a) unloaded; b) 25 cycles; c) 200 cycles; d) 500 cycles; e) 1,000 cycles; f) 5,000 cycles; g) 10,000 cycles; h) 270,000 cycles
Looking closely at this image, the z-binder toward the top of this micro-delamination is much darker and more in focus indicating that it is closer to the top the specimen. The warp tow edge where micro-delamination reaches a point, the nearest z-binder is clearly stopping it from growing in the same way as it has been seen in previous chapters; this is very clear from the continued growth of this micro-delamination, as seen in Figure 8.22e-h.

![Image](image-url)

**Figure 8.23**: Damage development during tension-tension warp-direction fatigue loading with a peak stress of 190 MPa of a 3DMMHyb specimen; this stress is equivalent to 500 N/mm. This specimen was manufactured using the VARTM. a) unloaded; b) 25 cycles; c) 200 cycles; d) 500 cycles; e) 1,000 cycles; f) 5,000 cycles; g) 10,000 cycles; h) 90,000 cycles

Micro-delaminations can also be seen to have developed from the resin-rich channel transverse cracks. These micro-delaminations form along the interface between the warp tows and resin-rich pockets. An example of this type of damage and its growth has been highlighted and enlarged with a
yellow ellipse in Figure 8.22c-h; the dashed yellow line in each image is there to indicate the position of the resin-rich channel.

Since two manufacturing methods were used to prepare warp-direction specimens, i.e. wet-layup and VARTM, it is useful to see if there are any differences in damage developed. Figure 8.23 shows the damage developed in a VARTM 3DMHyb specimen fatigue loaded with a peak stress of 190MPa. Each photograph in Figure 8.23 represents the same number of cycles as used in Figure 8.22, except for Figure 8.23h; this is due to differences in the number of cycles to failure for these two specimens. A comparison shows that there is no significant difference between the damage developed in the wet-layup and VARTM manufactured specimens, which is to be expected.

Figure 8.24 - Figure 8.26 show polished cross-sections of warp-direction fatigue damaged specimens at various positions through the width of a specimen. Figure 8.24 is a cross-section through a z-binder, showing the numerous resin-rich pockets between weft tow layers. Here, many transverse cracks can be seen to have formed within the weft tows, propagating through much of the thickness of the specimen by means of the resin-rich pockets between weft tow layers. In the surface weft tows it can be seen that there is either one main transverse crack toward its centre, or two transverse cracks spaced a distance apart; this corresponds well with the observations seen in the plan view images shown earlier.

In addition to the weft tow transverse matrix cracks, there are also transverse cracks through the resin-rich channels between adjacent weft tow columns (see Figure 8.24 and Figure 8.25). In the same manner as the all-glass orthogonal weaves, when this type of crack crosses the z-binder it debonds from the surrounding material along one side of the tow. In the all-glass specimens, the z-binder generally only debonds from the central weft tow and a portion of the resin-rich pocket. However, it can be seen here that some of the debonding extends further in the hybrid such that z-binder begins to debond from the surface weft tow.

Figure 8.24: Micrograph cross-section through a z-binder of a 3DMHyb specimen fatigue loaded along the warp-direction with a peak stress of 175 MPa – this shows damage developed during cycling
Figure 8.25 shows a cross-section located along the edge of some warp tows, as indicated by the remaining section of z-binder, and Figure 8.26 is somewhere toward the middle of both warp tows. In both micrographs, micro-delaminations can be seen along the warp/weft tow interfaces as well as between warp tows and the resin-rich channel between adjacent weft tows; these micro-delaminations are the same as those highlighted in Figure 8.22. It appears to be very common in these specimens for the intersection of the resin-rich channel transverse cracks with the warp tows to lead to fracture along the interface. This type of delamination more readily occurs along the warp tow interface nearest the specimen surface. In addition, the warp tow/resin-rich region delaminations can extend to inner warp tow interfaces during continued cycling of the specimen.

![Micrograph cross-section along the edge of some warp tows of a 3DMHyb specimen fatigue loaded along the warp-direction with a peak stress of 175 MPa – this shows damage developed during cycling](image1)

![Micrograph cross-section within some warp tows of a 3DMHyb specimen fatigue loaded along the warp-direction with a peak stress of 175 MPa – this shows damage developed during cycling](image2)

**8.4.2.2. Weft-direction**

Figure 8.27 shows the damage development in a wet-layup manufactured weft-direction specimen that was fatigue loaded with a peak stress of 190 MPa. Each image indicates the level of
damage present when the specimen is unloaded, cycled for 25, 200, 500, 1000, 5000, and 10000 cycles. Figure 8.27a shows the specimen prior to fatigue loading. It can be seen in this image that some of the carbon fibres from one of the z-binders do not lie neatly within the binder. This would have occurred during weaving, becoming fixed in place when infused with resin. There is no evidence that this influences the performance of the specimen since the number of carbon fibres that have come away from the bulk z-binder is small. Additionally, there are a number of voids contained in this specimen, many of which are present in the typical location within the resin-rich pockets around the z-binder; a few voids are also located along the resin-rich pockets between weft tows.

Figure 8.27: Damage development during tension-tension weft-direction fatigue loading with a peak stress of 190 MPa of a 3DMHyb specimen; this stress is equivalent to 500 N/mm. This specimen was manufactured using the wet-layup method. a) unloaded; b) 25 cycles; c) 200 cycles; d) 500 cycles; e) 1,000 cycles; f) 5,000 cycles; g) 10,000 cycles;

Figure 8.27b shows that after 25 cycles warp tow transverse matrix cracks have developed and a number of these cracks extend most of the way across the width of the specimen. In addition, the
longer cracks develop centrally within the warp tows. There are also a few shorter cracks that are developing from either edge of the specimen. At this stage the development of damage appears quite similar to the 3DMG-T2 material. Interestingly it can be noted that some of the transverse cracks in this specimen coincide with voids in the resin-rich pockets between weft tows. This indicates that these voids are stress raisers, though fracture may have initiated in a warp tow and then progressed toward the void.

As well as warp tow transverse matrix cracks, transverse cracks through the z-binder have also developed; an example has been highlighted and enlarged in Figure 8.27b. It is interesting to note that in these images, the transverse cracks in this region are more visible in the sections where the z-binder crosses the surface furthest away from the camera. This gives the appearance that the transverse cracks only extend short lengths over the width. However, this damage is very similar to that in the 3DMG specimens and therefore these transverse cracks will probably extend across most of the specimen width.

In each of the images in Figure 8.27 the z-binder appears out of focus when it passes through the thickness and reaches the surface furthest away from the camera. The surface nearest the camera in this region remains in focus, and it can be seen that continued cycling (Figure 8.27c-g) leads to a distinct darkening of the image – highlighted in a yellow ellipse. It does not appear from these images that this damage come directly from the z-binder transverse cracks present here since this damage appears to grow toward the z-binder crack. It is possible that this is due to some form of damage at the nearest warp/weft interface, such as a micro-delamination.

Between cycle 1000 and 5000, at the z-binder nearest the top surface (closet to the camera and most in focus), it appears that micro-delaminations have developed extending either side of the z-binder (see Figure 8.27f). There are flat edges on either side of these micro-delaminations that indicate that these have probably developed between the surface weft tow and adjacent warp tow; green lines have been used to point out the edge of the surface weft tow, and a blue arrow to indicate the edge of the central weft tow. Micro-delaminations of this type were not seen previously in either the 3D-78 or 3DMG weft-direction loaded specimens. Therefore, it is likely that this damage is related to some influence from the carbon fibre z-binder, though it is unclear what mechanisms are operating since the tension applied to the z-binder was the same as in the hybrid as in the all-glass specimens.

Continued development of warp tow transverse matrix cracks results in damage development that is similar to the 3DMG-T2 specimens shown previously. After 10,000 cycles (Figure 8.27g) there appears to be more than one transverse crack per column of warp tows. This is clearly seen in Figure 8.28, which shows a cross-section of a weft-direction fatigue loaded specimen. This cross-section is located toward the edge of the central weft tow, hence the missing surface weft tows in the image; the width of the surface weft tows is less than the width of the central weft tow. Here, at least two transverse
cracks can be seen per warp tow, with each crack extending through the resin-rich regions to the surface of specimen surface. In this cross-section, z-binder transverse cracks can also be seen, with many penetrating through the resin to the surface. From the z-binder transverse cracks, debonding along the interface between the z-binder and the central warp tow can be seen.

![Diagram](image)

**Figure 8.28:** Micrograph cross-section along the edge of the central weft tow of a 3DMHyb specimen fatigue loaded along the warp-direction with a peak stress of 190 MPa – this shows damage developed during cycling

8.5. Crack density comparison of 3DMG and 3DMHyb under warp-direction fatigue loading

Crack density measurements are a useful way of comparing the performance of similar materials under various loading conditions. In this section a comparison will be made between 3DMG and 3DMHyb specimens fatigue loaded along the warp direction. The method used to determine the crack density was described in Chapter 5. Since the crack density measurements were made using observations from images taken during testing, there is some additional level of uncertainty in the measurements made for the hybrid specimens because the colour of the z-binders obscures some of the cracks, making it difficult to determine their exact progression. Consequently, an overestimation of the hybrid specimen crack densities may occur.

In Figure 8.29 the crack density for two 3DMG and two 3DMHyb specimens fatigued with the same peak force per unit width are plotted against fatigue cycle number. Generally, the curves for each material follow a similar trend and can be split into two components. The first part of each curve corresponds to the development of most of the cracks within the specimen and is represented by a relatively large increase in crack density over a small number of cycles. The second part is a much shallower increase in crack density with cycle number, and corresponds to a slower development of damage in the specimen. It is clear from this plot that the crack density is generally higher for the 3DMG
specimens than 3DMHyb specimens. Despite the difference in crack density of each material, it seems that crack saturation is reached after a similar number of cycles. Since the fatigue life of a 3DMHyb specimen is generally longer than 3DMG specimens, the carbon z-binder appears to impede the development of matrix cracks. A lower crack density in the hybrid specimens could result in fewer regions of high stress less of a stress build up around the warp tows, thus reducing the probability of fibre fracture and extending the fatigue life.

![Figure 8.29: Comparison of the crack density per cycle for two 3DMG and two 3DMHyb specimens manufactured using the wet-layup method.](image)

As shown previously, during fatigue loading permanent damage results in a loss of stiffness in the material. In Figure 8.30 a plot of normalised stiffness against crack density is shown. Interestingly, for the same crack density, the loss of stiffness is greater in the 3DMHyb specimens than the 3DMG specimens. A loss in material stiffness is usually accompanied by the development of damage. Therefore, if the crack density is the same for both materials, then another damage mechanism must have developed, or be developing to a greater extent, in 3DMHyb specimens than 3DMG. Table 8.4 and Table 8.5 show a comparison of a 3DMG and 3DMHyb specimen with the same (or similar) crack density, but differing amounts stiffness loss. In the corresponding plan-view images it can be seen that the hybrid specimen has begun to develop micro-delaminations, whereas micro-delamination growth is more limited in the all-glass specimen. Consequently, the additional stiffness reduction in the 3DMHyb specimen is probably due to micro-delamination development.
Figure 8.30: Loss of stiffness against crack density comparison of two 3DMG and two 3DMHyb specimens manufactured using the wet-layup method.

Table 8.4: Comparison of a 3DMG and 3DMHyb specimen with the same crack density

<table>
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<th>3DMG</th>
<th>3DMHyb</th>
</tr>
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<tbody>
<tr>
<td>Cycle</td>
<td>200</td>
</tr>
<tr>
<td>Normalised modulus</td>
<td>0.91</td>
</tr>
<tr>
<td>Crack density (mm(^{-1}))</td>
<td>0.39</td>
</tr>
</tbody>
</table>
Table 8.5: Comparison of a 3DMG and 3DMHyb specimen with the same crack density

<table>
<thead>
<tr>
<th></th>
<th>3DMG</th>
<th></th>
<th>3DMHyb</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cycle</td>
<td>400</td>
<td>Cycle</td>
<td>2000</td>
</tr>
<tr>
<td>Normalised modulus</td>
<td>0.87</td>
<td>Normalised modulus</td>
<td>0.85</td>
</tr>
<tr>
<td>Crack density (mm$^{-1}$)</td>
<td>0.47</td>
<td>Crack density (mm$^{-1}$)</td>
<td>0.48</td>
</tr>
</tbody>
</table>

8.6. Modelling

In this section, a finite element (FE) model is described to examine the behaviour of an all-glass and hybrid 3D woven composite with the same structure as used throughout this report. The model will focus on loading along the warp-direction. Fatigue failure along this loading direction is thought to result from a lack of delamination growth, and therefore a stress concentration build up, toward the edge of a warp tow as a consequence of matrix cracking. Consequently, the stresses at the interface between a surface weft tow and the corresponding warp tow will be examined in a condition where only a single transverse crack is present centrally along the weft tow.

For 3D composites, many studies FE focus on producing voxel models of unit cell structures [72, 76, 77] with a more idealised and uniform structure than those observed experimentally. Other studies use information from CT scans in order to model a more realistic 3D composite RVE using more precise geometries and architecture [78, 79, 80].
Figure 8.31: FE voxel model of 3DNCOW with a singular crack that traverses through the thickness from the centre of the weft tows. Red represents the warp tow, blue represents the weft tows, and green is used for the z-binders.

The FE model used in this work was developed by Topal [72] and was adapted to the needs of this project. The initial model was created in Texgen [81]. Using this software, a microstructural representative volume element (RVE) of the 3D-78 material was produced. The RVE covered a single unit cell of this material along both the warp and weft directions, and had dimensions of 8 mm measured along the warp-direction, 8.35 mm along the weft-direction and a height of 2.2 mm; the RVE is slightly larger than an actual unit cell because of the way in which Texgen generates a 3D structure. In the case of this model, each yarn cross-section was represented by an ellipse; an example of the RVE and the cross-sections can be seen in Figure 8.31. In Figure 8.31 the warp tows are shown in red, the weft tows in blue, and z-binders are green. During structure manufacture in Texgen, the material orientations were assigned to each tow to account for the orthotropy.

Once the model had been created it was imported into Abaqus standard as a voxel model; a voxel is the volumetric equivalent of a pixel. The use of a voxel model allows the 3D geometry to be generated using prismatic hexagonal finite elements, though this does result in non-smooth, step-like, surfaces to the structure. Using smaller elements will produce a smoother surface for each component; however, a compromise has to be made between the component size, the amount of discretisation errors, and overall computational time. The mesh used in this model has 1.5 million elements and can be solved within a few hours.

Loading of the specimen involved stretching the model by 1 mm along the warp-direction (x-axis). Constraints in the way of boundary conditions were applied to various parts of the model to prevent movement during loading about various axes. One end face perpendicular to the loading direction was fixed so loading could be applied from the opposite face. To account for Poisson’s
contraction, a support was placed along an edge to allow contraction in the y- and z-axis from opposing surfaces.

Table 8.6: Material properties used for each component using in the voxel FE model.

<table>
<thead>
<tr>
<th>Matrix material (epoxy resin)</th>
<th>Glass fibre/ epoxy resin impregnated tows</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Ex = 46 GPa</td>
</tr>
<tr>
<td></td>
<td>Ey = 13 GPa</td>
</tr>
<tr>
<td></td>
<td>Ez = 13 GPa</td>
</tr>
<tr>
<td></td>
<td>E = 3.8 GPa</td>
</tr>
<tr>
<td></td>
<td>ν = 0.38</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Carbon fibre/ epoxy resin impregnated tows</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ex = 146 GPa</td>
</tr>
<tr>
<td>Ey = 9.3 GPa</td>
</tr>
<tr>
<td>Ez = 9.3 GPa</td>
</tr>
</tbody>
</table>

For each of the warp-direction loaded specimens used in this project, it is relatively common to see a crack develop centrally along through the weft tows. During fatigue, these cracks will develop a micro-delamination between the surface weft tow and corresponding warp tow. This micro-delamination generally forms a shield-like shape that can blunt the stress concentration of the matrix crack (see Chapter 5). To this end, a crack was placed down the centre of the weft tow column located centrally in the FE model RVE of the 3DNCOW. This crack spanned the entire width and thickness of the RVE, cutting through all three weft tows and the resin-rich regions, leaving the warp tows intact. The creation of a crack in the model required the deletion of elements from the model.

The model was solved in two different ways: (1) with all the fibre tows consisting of GFRP properties, i.e. all-glass; and (2) the properties of the z-binder were replaced with those of a UD CFRP, i.e. hybrid. The matrix used was an epoxy resin. The materials were treated as linear elastic throughout the model and properties of the materials used can be seen in Table 8.6. The fibre tows properties are orthotropic, while the resin properties are isotropic. UD GFRP properties were taken from [82], while approximate values were calculated using the rule of mixture (ROM) for a UD CFRP using carbon fibre data from the fibre manufacturer [83], assuming a fibre volume fraction of 0.6; the properties of the resin were gathered experimentally. While ν_{23} for CFRP is not usually the same as ν_{12} or ν_{13}, in the absence of other values all three values were kept the same.
After running the model, the stresses and displacements were analysed at the surface weft tow/warp tow interface along the surface of the warp tow. Figure 8.32 show a comparison of the all-glass and hybrid 3D weaves in terms of the von Mises stresses, through-thickness stresses, and the through-thickness displacement, respectively. The z-binder path along the top of each image (Edge A) crosses through to the opposite surface, while the z-binder at the bottom of each image (Edge B) passes over the near surface.

In Figure 8.32, von Mises stresses have been plotted for the all-glass and hybrid 3DNCOW RVE, with the same stress limits in place for ease of comparison between the two. The von Mises stresses are useful as they provide a representation of the combination of stresses for the three principal directions. In this case the magnitude of these stresses are not particularly important, but does provide an insight into the stress distribution. As mentioned earlier, the z-binder crosses through to the opposite surface along edge A, and crosses over the near surface along edge B. The shape of the stress distribution in both Figure 8.32a and b is wider along edge A, and becomes narrower along edge B. While the loading of this model is quasi-static in nature, the distribution of stresses form a pattern similar to the formation of micro-delaminations during fatigue loading (see Figure 5.6 and 5.7). It can also be seen that the magnitude of the stresses are higher in the hybrid model.

When looking at the through-thickness, or peel, stresses in Figure 8.33, the stresses are generally higher along edge A than edge B. This indicates that failure (i.e. delamination) along this interface is more likely to initiate at edge A, where the crown of the z-binder is not present. What is even more striking is that the stress distribution along edge B is not only generally lower than edge A, but also tends to a point about the centre of the tow. This corresponds well with observations made experimentally with regards to micro-delaminations, supporting the idea that the z-binder crown causes a pinching effect limiting micro-delamination growth along this edge.

Comparison of the all-glass specimen with the hybrid model (see Figure 8.33a and b) suggest that the through-thickness stresses along the warp/weft interface are higher in the hybrid than in the all-glass. This suggests that, at least initially, failure of the warp/weft interface is more likely to occur in the hybrid material. In the experimental work it was seen that during the very early stage of fatigue, the hybrid specimens had a lower crack density and greater amount of delamination. The results from the FE model support these observations.
Figure 8.32: Von-mises stresses along the surface of a warp tow from a voxel (FE) model of a 3D orthogonal woven composite with a single crack through each weft tow layer; this is treated like the interface between the warp and surface weft tow. Both images have the same limits in the legend where the maximum and minimums stresses are 1400 MPa and 400 MPa respectively.
Figure 8.33: Through-thickness (peel) strength along the surface of a warp tow from a voxel (FE) model of a 3D orthogonal woven composite with a single crack through each weft tow layer; this is treated like the interface between the warp and surface weft tow. Both images have the same limits in the legend where the maximum and minimums stresses are 200 MPa and 20 MPa respectively.
8.7. Concluding remarks

In this chapter, a hybrid 3D orthogonal weave (3DMHyb) has been characterised in terms of quasi-static tensile and tension fatigue properties, and the damage development during each of these loading cases. The structure of the 3DMHyb material consists of two glass fibre warp tow lays, three glass fibre warp tow layers, and a carbon fibre z-binder. A comparison between the 3DMHyb material and the all-glass 3DMG-T2 material discussed in Chapters 6 and 7 was also made. It was found that:

- From quasi-static tensile loading of 3DMHyb specimens along the warp and weft-direction, the tensile strength and strain to failure were basically the same. However, the tensile modulus along the warp direction was higher than the weft direction indicating that the carbon z-binder may influence the stiffness during low strain loading. When the 3DMHyb material was compared to the 3DMG-T2 material, no difference in the mechanical properties in either the warp or weft direction was found. Additionally, no significant difference in properties could be found when comparing wet-layup manufactured and VARTM manufactured specimens.

- The fatigue life times of 3DMHyb along the warp-direction when fatigued with a peak force per unit width of 500 N/mm were superior by a factor of 10 or more. This appears to be a general trait of 3D orthogonal weaves with this number of layers as the trend of warp fatigue being better than weft fatigue has been seen for the other materials tested in this work.

- When fatigue loaded with a peak force per unit width of 500 N/mm along the warp direction, the 3DMHyb performed better than 3DMG-T2 by slightly more than a factor of two. This was generally noted for both wet-layup and VARTM manufactured specimens. The loss of stiffness over the fatigue life was very similar for both hybrid and all-glass specimens. The similarity in loss of stiffness was most notable during the early stage of loading. However, the energy dissipated per cycle was found to be lower for the hybrid specimens. It can be inferred from this that while some damage may develop at a similar rate in both material types, the development of this damage throughout the remainder of the fatigue life is much slower in the hybrids. This can be validated by the fact that the crack density and delamination growth during early stage loading was lower and higher respectively in the hybrid specimens than the all-glass. This indicates that the z-binder may suppress the development of some damage, thus extending the fatigue life.

- Damage development during quasi-static tensile and tension fatigue loading was similar for both wet-layup and VARTM manufactured specimens. In addition, the damage developed in warp and weft-direction loaded 3DMHyb specimens was generally the same as the damage seen in 3DMG-T2 warp and weft-direction specimens. For quasi-static and fatigue loading, damage mechanisms that were seen to occur within these specimens included: transverse cracks within transverse tows and resin-rich pockets, debonding of the z-binder from surrounding material, and micro-delamination damage. Micro-delamination growth during quasi-static
loading was usually small and very limited. In contrast, the micro-delamination damage during fatigue loading for warp direction specimens occurred along warp/weft tow interfaces as well as along warp/resin-rich channel interfaces. The warp/weft tow interface micro-delaminations would for the shield like shape seen in both 3DMG and 3D-78 specimens previously. The warp tow/resin-rich channel delamination on the other hand was only seen to develop along the warp tow interface nearest the specimen surface.

- Using a voxel FE model of the 3D orthogonal structure, loading it along the warp direction, and placing a crack down the centre of a stack of weft tows, it was possible to observe the stress distribution between the surface weft/ warp tow interface. Here, it was found that the peel stresses are higher along the edge of a warp tow where no z-binder is present, while these stresses were lower when the z-binder crosses over the adjacent surface weft tow. In addition, the stresses along this interface were higher for the hybrid specimen than the all-glass specimen. Both of these observations correspond well with those seen experimentally. Delamination damage appears to develop more preferentially along one edge of a warp tow, effectively being pinched by the z-binder along the opposite warp tow edge. Additionally, it appears to be easier for delaminations to develop in these hybrid specimens than the all-glass, at least during early stage loading.
Chapter 9
Conclusions and future work

9.1. Introduction

In this work, mechanical behaviour and damage development of a 3D non-crimp orthogonal woven composite has been investigated. The work was mostly focussed on the characterisation of this material when subjected to tensile fatigue loading, though some work on quasi-static tensile loading has also been undertaken. Three different 3D orthogonal woven composites were used, each with a similar structure consisting of three weft tow layers, two warp tow layers, and a z-binder interlacing along the warp direction. The first weave tested was manufactured by 3TEX and is known as 3D-78. The second weave is called 3DMG and was woven by a team at the University of Manchester; the 3DMG material was woven with two different z-binder tensions, designated 3DMG-T1 and 3DMG-T2 in order to distinguish between them. The final weave tested was produced by the University of Manchester and was a hybridised version of the 3DMG, and as such was called 3DMHyb. All tows in both the 3D-78 and 3DMG materials consisted of E-glass fibres, whereas in the 3DMHyb material the glass fibre z-binder tows were replaced with carbon fibre tows; the same epoxy resin system was used as a matrix for all three fabrics.

Each of these materials were quasi-statically loaded in tension to determine the basic material properties and observe the damage developed until failure. Analysis of the tensile fatigue performance for each material was achieved by comparing the number of cycles to failure at various peak stresses, the energy dissipation per cycle, and the loss of stiffness over the fatigue lifetime of specimens. Transparency of specimens enabled observation of damage to be made easily during quasi-static and fatigue loading. Fatigue loading of 3D-78 specimens was used to characterise the fatigue performance and observe the damage development in a commercial 3D orthogonal weave, which could then be used for comparison with the 3DMG and 3DMHyb materials. Since 3DMG and 3DMHyb were both manufactured by the same supplier, using the same setup, direct comparisons could be made between the hybrid material and the all-glass composites.

For much of this work, composite panels were manufactured using a wet-layup method. This method works well in a research environment, but not for industrial manufacture of composite panels. In addition to this, it was found to be difficult to remove voids from a 3D orthogonal woven structure using this method. Therefore, some 3DMG and 3DMHyb panels were manufactured using the VARTM (vacuum assisted resin transfer moulding) method, which is commonly used in industry; a comparison
was made with wet-layup specimens of the same material to determine if the manufacturing method affected the performance of the material.

9.2. Conclusions

9.2.1. 3D-78 – Mechanical characterisation and damage development

For the 3D-78 material the quasi-static tensile properties for both the warp and weft direction were found to be very similar, and may be related to the approximately equal fibre volume contents in each of these directions. In addition, these results indicate that the z-binder does not contribute much to the overall static loading performance along the warp direction. By contrast, the tensile fatigue performance of the warp-direction was better than the weft direction for all peak stresses tested. It was noted that the average loss of tensile stiffness up to failure was consistently higher in the warp-direction specimens, indicating that more damage accumulates in specimens loaded along this direction than the weft-direction. From damage observations, it was seen that delamination damage was more prevalent in warp-direction specimens, and would appear to be responsible for the difference in overall loss of stiffness by failure.

The damage that develops during both quasi-static and fatigue loading along the warp-direction is generally found to be quite similar and includes: transverse matrix crack within weft tows and resin-rich regions, through-thickness debonding of the z-binder, micro-delamination at the interface between warp and weft tows, and longitudinal splitting of the warp tows. Growth of the micro-delamination damage is greater during fatigue, forming a shield-like shape along the warp/surface weft tow interfaces as a result of the woven architecture. The pointed end of these micro-delaminations corresponds with the position of the z-binder, and is a consequence of the z-binder crown causing a pinching effect between the surface weft tow and the edge of the adjacent warp tow. Final failure is found to initiate preferentially along the edge of this warp tow since the lack of delamination damage allows stress concentrations to build at the edge of the warp tow.

Many of the damage mechanisms seen in warp-direction specimens were also found to develop in weft-direction loading. For weft-direction loading, both quasi-static and fatigue, the crack density was higher than for warp-direction loading, coupled with a limited amount of delamination between interfaces. Additionally, z-binder debonding was found to occur between the z-binder crown and the surface weft tow, as opposed to the debonding of the through-thickness region of the z-binder observed in warp-direction loading. Growth of z-binder debonding during weft-direction loading was similar to warp-direction micro-delamination growth in that it mostly occurred during fatigue loading. These z-binder debonds developed as triangular shaped damage from either side of a z-binder crown. Unlike the warp-direction loading, for weft-direction loading, there are no obvious stress concentrations that enable the initiation of final failure.
9.2.2. 3DMG – Mechanical characterisation and damage development

For quasi-static loading the 3DMG-T1 material along the warp-direction, it was found that both the tensile modulus and strength were greater, and the strain-to-failure was lower, than the weft-direction. Increasing the tension on the z-binder (3DMG-T2) reduced the tensile modulus and increased the strain-to-failure along the warp-direction to values similar to that of the weft-direction; the strength-to-failure remained the same for both materials. Little change to the weft-direction properties occurred with increased z-binder tension, indicating that the z-binder mostly influences the mechanical performance along the warp-direction.

Tension-tension fatigue testing of both the warp and weft-direction of 3DMG-T1 at a peak stress of 200 MPa showed virtually no difference in fatigue lifetimes. However, increasing the z-binder tension (3DMG-T2) significantly improved fatigue lifetimes along the warp-direction, while the weft-direction remained relatively unchanged. The loss of stiffness over the fatigue lifetime of both 3DMG-T1 and T2 specimens were virtually the same, with a total loss of stiffness around 22-24% by failure. However, the energy dissipated per cycle was 1½ to 2 times higher in the 3DMG-T1 specimens during stage 2 in fatigue loading. During stage 2 the energy dissipation is a shallow incline; the experimental results showed that the higher the energy dissipated per cycle in this region, the smaller the number of cycles to failure.

Damage development during quasi-static loading of the warp-direction of the 3DMG specimens consisted of transverse cracks in weft tows and resin-rich regions, z-binder debonding, and limited micro-delamination damage. It was common to see either two transverse cracks equally spaced within weft tows, or one central crack within a weft tow. The multiple transverse cracks observed in quasi-static loading probably developed from the central weft tow as a result of its thick, rectangular cross-section, whereas the single transverse crack will develop in the surface weft tow due to its semi-circular cross-section with only a small thick region where cracks can initiate more easily. The same damage develops during tensile fatigue loading, however there is greater micro-delamination growth. Micro-delamination development in the 3DMG material is similar to the 3D-78 material, but can be different in a number of ways. For instance, in 3DMG micro-delamination growth was found to either develop from a transverse crack toward the centre of a weft tow, or would grow from a crack near the boundary of a weft tow and grow toward centre of the weft tow. Unlike 3D-78, additional micro-delamination damage was found to develop from transverse cracks along the resin-rich channel between columns of weft tows. These micro-delaminations would develop along the warp tow/ matrix interface nearest the surface of the specimen.

While much of the damage developed during warp-direction tensile fatigue of 3DMG-T1 and T2 are similar, there are differences, most notably in damage positioning, that could explain the improved fatigue performance with increasing z-binder tension. The differences in damage here are
seen in relation to the micro-delamination damage development along the warp/weft tow interface. In 3DMG-T1, micro-delamination damage is seen to develop preferentially from weft tow cracks toward the weft tow boundary, which is the direct result of the looser z-binder tension applied during weaving. In contrast, 3DMG-T2 micro-delamination damage generally develops from weft tow transverse cracks located toward the centre of a weft tow. The higher z-binder tension in 3DMG-T2 causes the z-binder to push down harder on the surface weft tows, making it hard for delamination damage to develop from the edge of the weft tow. Like the 3D-78 material, transverse cracks still develop toward the centre of the surface weft tows in both 3DMG-T1 and -T2, producing a stress concentration along the surface of the warp tows that is accentuated by the compressive through-thickness force of the z-binder under tension. However, with micro-delamination development preferred from transverse cracks toward the edge of the weft tows in 3DMG-T1, this stress concentration is not reduced like that seen in both 3D-78 and 3DMG-T2, thus 3DMG-T1 specimens end up with a lower fatigue resistance.

Damage development during quasi-static loading along the weft-direction for 3DMG mostly consisted of many long, transverse cracks within warp tows and through the z-binders, that extended across most/all of the specimen width. There are no clear indications that z-binder debonding, or micro-delaminations develop during this type of loading. However, near failure, longitudinal splitting cracks formed through the weft tows just under the z-binder crown, and extended a small distance either side. For fatigue loading, the same type of transverse cracks was seen to develop, but there was additional damage in the form of z-binder debonding and micro-delaminations. Z-binder debonding occurred between the z-binder crown and surface weft tow. On the other hand, micro-delamination growth occurred along warp/weft interfaces. The shape of both the z-binder debonding, and micro-delaminations damage were similar in that they were wider along one edge of the weft tow and narrowed toward the weft tow centre.

It can be noted that both 3DMG-T1 and -T2 specimens showed very similar damage progression up to failure for loading along both the warp and weft-direction. In addition, little difference was noted in the damage development for both wet-layup and VARTM specimens.

9.2.3. 3DMHyb – mechanical characterisation and damage development

Comparison of the warp and weft-direction properties during quasi-static loading of the 3DMHyb material showed no difference in the strength and strain-to-failure, but the low-strain tensile modulus was found to be slightly higher in the 3DHyb composites. This suggests that the carbon fibre z-binder may only influence loading at low strain levels. 3DMHyb had approximately the same warp and weft properties to 3DMG-T2, though there was a slightly lower strength to failure along the warp direction in 3DMHyb; this was true for both wet-layup and VARTM specimens. It was noted that when consolidated the 3DMHyb composites were thicker than 3DMG composite laminates. Since both were woven and manufactured into panels using the same methods, it assumed that the difference in thickness
may be related to an increased resin content in the hybrid specimens. Accounting for the difference in thickness, it was found to be useful to tests the 3DMG-T2 and 3DMHyb specimens at the same force per unit widths.

Like the 3D-78 and 3DMG-T2 materials, the fatigue properties of 3DMHyb were superior along the warp-direction; at a peak force per unit width of approximately 500 N/mm the warp fatigue lifetimes were a factor of 10 greater than the weft-direction lifetimes. Comparison of 3DMG-T2 and 3DMHyb specimens fatigue loaded with a peak force per unit width of 500 N/mm along the warp-direction, manufactured either by wet-layup and VARTM, showed that the hybrid specimens had improved fatigue lifetimes (more than a factor of two). The loss of stiffness was very similar for both hybrid and all-glass specimens, however the energy dissipated per cycle was lower for the hybrid specimens. The crack density was also lower and, at least initially, the delamination growth was greater in the hybrid specimens than in the all-glass specimens. The increased performance of the hybrids compared to the all-glass indicates that the z-binder may supress some damage development during fatigue loading, thus slightly extending the fatigue life.

Compared to the 3DMG-T2 warp and weft-direction specimens loaded in quasi-static tension and in tension-tension fatigue, the damage developed in 3DMHyb specimens was very similar. Damage that was observed during testing of both loading directions included: transverse cracks within transverse tows and resin-rich regions, z-binder debonding, and micro-delamination growth. In quasi-static loading for both the warp and weft-direction, as well as weft-direction fatigue, the growth of delamination damage seems limited. In contrast, micro-delamination growth is significantly larger for warp-direction loading, developing similarly to the 3DMG-T2 specimens. Regardless of the manufacturing method, the observed damage mechanisms were seen to occur in all specimens.

Finally, a voxel model developed by Topal et al [72] of the 3D orthogonal all-glass woven structure tested was used to predict numerically the stresses along the surface weft/warp tow interface during tensile loading. A single transverse matrix crack was placed in the model at the centre of a stack of weft tows. It was found that under load, the peel stresses along the warp/weft tow interface were higher and extended over a slightly larger area along one edge of a warp tow; along the other warp tow edge, the peel stresses narrowed to a point. The lower peel stresses occur along the edge of the warp tow nearest a z-binder crown, and correspond well with experimental observations of the developing shape of micro-delaminations. A comparison of an all-glass and hybrid model found that there were higher peel stresses in the hybrid model suggesting that, at least initially, delamination damage initiates more readily in the hybrid specimens. Again, this corresponds well with experimental observations.

9.2.4. Comparison of 3D-78, 3DMG, and 3DMHyb

Structurally all three materials are very similar, with each containing the same number of warp and weft tow layers, and type of orthogonal path followed by the z-binder. When compared via
comparison of the internal structure, 3DMG and 3DMHyb are the most similar; the warp and weft tow layers remain in-line through the thickness, and the z-binder has an orthogonal path. In contrast, the 3D-78 material is the most different to the others with the z-binder following a more sinusoidal pathway and the weft tow layers not sitting in-line through the thickness. The difference in internal structure between 3D-78 and 3DMG/3DMhyb is probably the result of z-binder tension applied during weaving and the spacing between warp and weft tows along the length and width of the fabric. 3D-78 has a larger unit cell than 3DMG and 3DMhyb, and potentially has a higher z-binder tension, thus resulting in the structural appearance observed.

With the exception of the 3DMG-T1 warp-direction tensile modulus, the static tensile mechanical properties of all three materials has been shown to be reasonably similar for both loading directions. Generally, the weft-direction had the lower properties for all three materials, but often these were within scatter when compared to the warp-direction. With regards to tensile fatigue, 3DMG-T1 was noted to have a similar behaviour in both the warp and weft-direction, which was in direct contrast to that seen previously with 3D-78 where the warp-direction had a greater fatigue life at all strain levels. Increasing the z-binder tension in 3DMG to produce 3DMG-T2 showed improvement in the warp-direction fatigue properties, and made little difference to the weft-direction properties. Increasing the z-binder tension caused 3DMG-T2 fatigue properties to become similar to 3D-78 indicating that the effect of tension could improve the properties along the warp-direction. The increase relative to 3DMG-T1, as described above is believed to be related to the formation of micro-delamination damage under various z-binder tensions. Like 3DMG-T2 and 3D-78, 3DMHyb performed better along the warp direction. Compared with 3DMG-T2 it was shown that the crack density in 3DMHyb was slightly lower, and the delamination growth may be slightly higher. It is believed that the improved performance of the 3DMHyb material is the result of the carbon fibre z-binder suppressing some damage development, i.e. transverse cracks, thus enabling the fatigue life to become slightly extended.

For both quasi-static and fatigue loading, the damage mechanisms developed were similar for all three materials. These mechanisms of damage include, transverse cracks within transverse directed tows and resin-rich regions, debonding of z-binders and micro-delamination growth. Each of these mechanisms tended to develop in similar locations depending on the loading direction. The damage mechanism that altered the most between the three material types was the micro-delamination damage. The alteration of this damage mechanism was mainly positional and appears to be related to the amount of tension applied to the z-binder during weaving. In 3DMG-T1 (the material with the lowest z-binder tension), the micro-delamination damage was seen to develop preferentially from weft tow matrix cracks located near the edge of a surface weft tow. By increasing the z-binder tension (3DMG-T2 and 3DMHyb) it was found that micro-delamination damage shifted to form mostly from weft tow matrix cracks toward the centre of a surface weft tow, while still maintaining some delamination growth similar to 3DMG-T1. Finally, in 3D-78, which appears to have the highest z-binder tension, micro-
delamination development seemed to only develop around weft tow matrix cracks toward the centre of a surface weft tow.

9.3. Future work

This work has shown that a slight change in structure and material composition of a 3D non-crimp orthogonal woven composite consisting of three weft tow layers, two warp tow layers, and a z-binder interlacing along the warp direction, does not significantly change the overall damage development during quasi-static tensile, or tension-tension fatigue loading. In addition, the use of a carbon fibre z-binder in a glass fibre 3D weave mostly influenced tensile fatigue properties at low stress/low initial peak strain levels. Insufficient material was available for a comprehensive assessment and a more extensive S-N curve comparison between the hybrid and non-hybrid material should be made. It would be valuable to examine peak stresses lower than those used here, i.e. towards the fatigue limit, where fatigue lifetimes reach and/or exceed 5 million cycles to failure. This would be useful as it would confirm whether the carbon fibre z-binder has a more beneficial effect the lower the fatigue loading. In addition, the benefits of using a small amount of carbon fibre (i.e. replacing the z-binder) should be explored for other areas, in particular the impact and post-impact compression strengths, and the interlaminar toughness. Finally, the rather limited hybridisation achieved in this project can be extended by replacing glass tows in the weft or warp layers with carbon fibre tows, or by replacing whole layers of glass tows. This type of hybridisation is yet to be explored; there are a number of possible ways in which the 3D orthogonal woven structure could be altered to produce advantageous properties.
References


Appendix A

Below is the Visual Basic for Applications (VBA) macro code that can be used in excel to determine the energy dissipation, tangent stiffness, and secant stiffness.

Initial portion of code

Sub bla()

Application.ScreenUpdating = False

Dim Counter As Long
Dim Counter_1 As Long
Dim Cycle As Long
Dim Cycle_a As Long
Dim Strain_1 As Single
Dim Strain_2 As Single
Dim Load_1 As Single
Dim Load_2 As Single
Dim Msg_1 As String
Dim Msg_2 As String
Dim Specimen_Width As Single
Dim Specimen_Thickness As Single
Dim Specimen_Area As Single

Msg_1 = "Input Specimen Width (mm)"
Msg_2 = "Input Specimen Depth (mm)"

Specimen_Width = InputBox(Msg_1, "Input Value")
Specimen_Thickness = InputBox(Msg_2, "Input Value")
Specimen_Area = Specimen_Width * Specimen_Thickness * (10 ^ -6)

Range("A1:J91694").Select
Selection.Copy
Sheets.Add After:=Sheets(Sheets.Count)
ActiveSheet.Paste
Range("J1").Select
Range(Selection, Selection.End(xlDown)).Select
Application.CutCopyMode = False
Selection.ClearContents
Range("I1").Select

Sheets("Test1.steps.tracking").Select
Range("A1:J91694").Select
Selection.Copy
Sheets.Add After:=Sheets(Sheets.Count)
ActiveSheet.Paste
Range("J1").Select
Range(Selection, Selection.End(xlDown)).Select
Application.CutCopyMode = False
Selection.ClearContents
Range("I1").Select
ActiveSheet.Paste

'--Average Strain-----------------------------------------------
ActiveWorkbook.Worksheets(1).Activate

'------Chart------
Charts.Add

With ActiveChart
  .ChartType = xlXYScatterSmoothNoMarkers
  .Name = "Energy-Stiffness: Average"
  Do Until .SeriesCollection.Count = 0
    .SeriesCollection(1).Delete
  Loop
End With

'----------------
ActiveWorkbook.Worksheets(1).Activate

Dim Strain_F As Single
Dim Strain_B As Single
Dim Strain_Av As Single
Counter = 2
Cycle = Cells(2, 3)

Do Until Cycle = 0
  Strain_F = Cells(Counter, 9)
  Strain_B = Cells(Counter, 10)
  Strain_Av = (Strain_F + Strain_B) / 2
  Cells(Counter, 9) = Strain_Av
  Counter = Counter + 1
  Cycle = Cells(Counter, 3)
Loop

Range("J1").Select
Range(Selection, Selection.End(xlDown)).Select
Application.CutCopyMode = False
Selection.ClearContents

Cells(1, 9) = "Av. Strain (\(\mu s\n\))"
Range("A1").Select

Call Worksheet_Format

Cells(2, 10) = Specimen_Width
Cells(2, 11) = Specimen_Thickness

Call Energy(Specimen_Area)
Call Tangent_Stiffness(Specimen_Area)
Call Secant_Modulus_Tracking_File(Specimen_Area)

'--Strain: Front-------------------------------------------------------------

ActiveWorkbook.Worksheets(2).Activate

'-------Chart------

Charts.Add

With ActiveChart

  .ChartType = xlXYScatterSmoothNoMarkers
  .Name = "Energy-Stiffness: Front"

  Do Until .SeriesCollection.Count = 0
    .SeriesCollection(1).Delete
  Loop

End With

'----------

ActiveWorkbook.Worksheets(2).Activate

Range("A1").Select

Cells(1, 9) = "Strain Front (µsn)"

Call Worksheet_Format

Cells(2, 10) = Specimen_Width
Cells(2, 11) = Specimen_Thickness

Call Energy(Specimen_Area)
Call Tangent_Stiffness(Specimen_Area)
Call Secant_Modulus_Tracking_File(Specimen_Area)

'--Strain: Back-------------------------------------------------------------

ActiveWorkbook.Worksheets(3).Activate

'-------Chart------
Charts.Add

With ActiveChart

.ChartType = xIYSscatterSmoothNoMarkers
.Name = "Energy-Stiffness: Front"

Do Until .SeriesCollection.Count = 0

.SeriesCollection(1).Delete

Loop

End With

'----------------

ActiveWorkbook.Worksheets(3).Activate
.Range("A1").Select

Cells(1, 9) = "Strain Back (µsn)"

Call Worksheet_Format

Cells(2, 10) = Specimen_Width
Cells(2, 11) = Specimen_Thickness

Call Energy(Specimen_Area)
Call Tangent_Stiffness(Specimen_Area)
Call Secant_Modulus_Tracking_File(Specimen_Area)

Application.ScreenUpdating = True

End Sub

---------------------------------------------------------------------------------------------------

Sub Worksheet_Format()

Cells(1, 1) = "Total Time (s)"
Cells(1, 2) = "Cycle Elapsed Time (s)"
Cells(1, 3) = "Total Cycles"
Cells(1, 4) = "Elapsed Cycles"
Cells(1, 5) = "Step"
Cells(1, 6) = "Total Cycle Count"
Cells(1, 7) = "Position (mm)"
Cells(1, 8) = "Load (N)"
Cells(1, 10) = "Specimen Width (mm)"
Cells(1, 11) = "Specimen Thickness (mm)"
Cells(1, 12) = "Cycle Number"
Cells(1, 13) = "Energy Lost (J/m^3)"
Cells(1, 14) = "Tangent Stiffness (Pa)"
Cells(1, 15) = "Normalised Tangent Stiffness Reduction"
Cells(1, 16) = "Secant Stiffness (Pa)"
Cells(1, 17) = "Normalised Secant Stiffness Reduction"
Columns("A:A").Select
Selection.ColumnWidth = 11

Columns("B:B").Select
Selection.ColumnWidth = 13

Columns("C:C").Select
Selection.ColumnWidth = 7

Columns("D:D").Select
Selection.ColumnWidth = 8

Columns("E:E").Select
Selection.ColumnWidth = 5

Columns("F:F").Select
Selection.ColumnWidth = 11

Columns("G:G").Select
Selection.ColumnWidth = 11

Columns("H:H").Select
Selection.ColumnWidth = 11

Columns("I:I").Select
Selection.ColumnWidth = 11

Columns("J:J").Select
Selection.ColumnWidth = 12

Columns("K:K").Select
Selection.ColumnWidth = 15

Columns("L:L").Select
Selection.ColumnWidth = 9

Columns("M:M").Select
Selection.ColumnWidth = 12

Columns("N:N").Select
Selection.ColumnWidth = 13

Columns("O:O").Select
Selection.ColumnWidth = 19

Columns("P:P").Select
Selection.ColumnWidth = 13

Columns("Q:Q").Select
Selection.ColumnWidth = 19

With Range("A1:S1")
  .WrapText = True
  .VerticalAlignment = xlCenter
.HorizontalAlignment = xlCenter

With .Font
  .Bold = True
End With

End With

Range("A1:I1").Select

With Selection.Interior
  .Pattern = xlSolid
  .PatternColorIndex = xlAutomatic
  .ThemeColor = xlThemeColorAccent1
  .TintAndShade = 0.599963377788629
  .PatternTintAndShade = 0
End With

Range("J1:K1").Select

With Selection.Interior
  .Pattern = xlSolid
  .PatternColorIndex = xlAutomatic
  .ThemeColor = xlThemeColorAccent3
  .TintAndShade = 0.599963377788629
  .PatternTintAndShade = 0
End With

Range("L1:Q1").Select

With Selection.Interior
  .Pattern = xlSolid
  .PatternColorIndex = xlAutomatic
  .ThemeColor = xlThemeColorAccent2
  .TintAndShade = 0.599963377788629
  .PatternTintAndShade = 0
End With

Range("A1:Q1").Select

Selection.Borders(xlDiagonalDown).LineStyle = xlNone
Selection.Borders(xlDiagonalUp).LineStyle = xlNone
Selection.Borders(xlEdgeLeft).LineStyle = xlNone
Selection.Borders(xlEdgeTop).LineStyle = xlNone

With Selection.Borders(xlEdgeBottom)
  .LineStyle = xlContinuous
  .ColorIndex = xlAutomatic
  .TintAndShade = 0
  .Weight = xlMedium
End With

Selection.Borders(xlEdgeRight).LineStyle = xlNone
Selection.Borders(xlInsideVertical).LineStyle = xlNone
Selection.Borders(xlInsideHorizontal).LineStyle = xlNone

Range("J1:K2").Select

Selection.Borders(xlDiagonalDown).LineStyle = xlNone
Selection.Borders(xlDiagonalUp).LineStyle = xlNone

With Selection.Borders(xlEdgeLeft)
    .LineStyle = xlContinuous
    .ColorIndex = xlAutomatic
    .TintAndShade = 0
    .Weight = xlMedium
End With

Selection.Borders(xlEdgeTop).LineStyle = xlNone

With Selection.Borders(xlEdgeBottom)
    .LineStyle = xlContinuous
    .ColorIndex = xlAutomatic
    .TintAndShade = 0
    .Weight = xlMedium
End With

With Selection.Borders(xlEdgeRight)
    .LineStyle = xlContinuous
    .ColorIndex = xlAutomatic
    .TintAndShade = 0
    .Weight = xlMedium
End With

With Selection.Borders(xlInsideVertical)
    .LineStyle = xlContinuous
    .ColorIndex = xlAutomatic
    .TintAndShade = 0
    .Weight = xlMedium
End With

Range("A1").Select

End Sub
Energy Dissipation Code

Sub Energy(Specimen_Area)
,
' Energy Macro
' Macro recorded 09/07/2013 by Tobias Capell
' Edited by Matthew Poole: Jan 2013
,
' Keyboard Shortcut: Ctrl+Shift+E
,
Dim Counter_2 As Integer

Dim Area_Lower As Single
Dim Area_Upper As Single

Dim Stress_1 As Single
Dim Stress_2 As Single

Dim Energy As Single

Dim Width As Single
Dim Height As Single
Dim Area As Single

Dim Energy_Dissipated() As Single
Dim Energy_Cycles() As Single

ReDim Energy_Dissipated(i)
ReDim Energy_Cycles(i)

i = 0

Counter = 2
Counter_1 = 2
Counter_2 = 2

Cycle = Cells(2, 6)

Do Until Cycle = 0

Area_Upper = 0
Area_Lower = 0

Do Until Cycle_a > Cycle

Cycle_a = Cells(Counter, 6)

If Cycle_a > Cycle Then

Strain_1 = Cells(Counter, 9) * (10 ^ -6)
Strain_2 = Cells(Counter_1, 9) * (10 ^ -6)

Load_1 = Cells(Counter, 8)

End If

End Do

Dim Energy_Dissipated() As Single
Dim Energy_Cycles() As Single

ReDim Energy_Dissipated(i)
ReDim Energy_Cycles(i)

i = 0

Counter = 2
Counter_1 = 2
Counter_2 = 2

Cycle = Cells(2, 6)

Do Until Cycle = 0

Area_Upper = 0
Area_Lower = 0

Do Until Cycle_a > Cycle

Cycle_a = Cells(Counter, 6)

If Cycle_a > Cycle Then

Strain_1 = Cells(Counter, 9) * (10 ^ -6)
Strain_2 = Cells(Counter_1, 9) * (10 ^ -6)

Load_1 = Cells(Counter, 8)

End If

End Do

Dim Energy_Dissipated() As Single
Dim Energy_Cycles() As Single

ReDim Energy_Dissipated(i)
ReDim Energy_Cycles(i)

i = 0

Counter = 2
Counter_1 = 2
Counter_2 = 2

Cycle = Cells(2, 6)

Do Until Cycle = 0

Area_Upper = 0
Area_Lower = 0

Do Until Cycle_a > Cycle

Cycle_a = Cells(Counter, 6)

If Cycle_a > Cycle Then

Strain_1 = Cells(Counter, 9) * (10 ^ -6)
Strain_2 = Cells(Counter_1, 9) * (10 ^ -6)

Load_1 = Cells(Counter, 8)

End If

End Do

Dim Energy_Dissipated() As Single
Dim Energy_Cycles() As Single

ReDim Energy_Dissipated(i)
ReDim Energy_Cycles(i)

i = 0

Counter = 2
Counter_1 = 2
Counter_2 = 2

Cycle = Cells(2, 6)

Do Until Cycle = 0

Area_Upper = 0
Area_Lower = 0

Do Until Cycle_a > Cycle

Cycle_a = Cells(Counter, 6)

If Cycle_a > Cycle Then

Strain_1 = Cells(Counter, 9) * (10 ^ -6)
Strain_2 = Cells(Counter_1, 9) * (10 ^ -6)

Load_1 = Cells(Counter, 8)
Load_2 = Cells((Counter + 1), 8)

Stress_1 = Load_1 / Specimen_Area
Stress_2 = Load_2 / Specimen_Area

Width = Strain_2 - Strain_1
Height = (Stress_1 + Stress_2) / 2
Area = Width * Height

Area_Upper = Area_Upper + Area
Counter = Counter + 1
Exit Do
End If

Strain_1 = Cells(Counter, 9) * (10 ^ -6)
Strain_2 = Cells((Counter + 1), 9) * (10 ^ -6)

Load_1 = Cells(Counter, 8)
Load_2 = Cells((Counter + 1), 8)

Stress_1 = Load_1 / Specimen_Area
Stress_2 = Load_2 / Specimen_Area

If Strain_2 < Strain_1 Then
Width = Strain_1 - Strain_2
Height = (Stress_1 + Stress_2) / 2
Area = Width * Height
Area_Lower = Area_Lower + Area
Else
Width = Strain_2 - Strain_1
Height = (Stress_1 + Stress_2) / 2
Area = Width * Height
Area_Upper = Area_Upper + Area
End If

Counter = Counter + 1
Loop

Energy = Area_Upper - Area_Lower
Energy_Dissipated(i) = Energy
Energy_Cycles(i) = Cycle

Cells(Counter_2, 12) = Cycle
Cells(Counter_2, 13) = Energy

Counter_1 = Counter
Counter_2 = Counter_2 + 1

Cycle = Cells(Counter, 6)

If Cycle > 0 Then
    i = i + 1
End If

ReDim Preserve Energy_Dissipated(i)
ReDim Preserve Energy_Cycles(i)

Loop

Call Energy_Chart(Energy_Dissipated, Energy_Cycles)

End Sub

-------------------------------------------------------------------------------------
Sub Tangent_Stiffness(Specimen_Area)

' Macro written 16/12/2013 by Matthew Poole
' Edited Jan-May 2014
'

Dim Counter_2 As Long
Dim Counter_3 As Long
Dim Load_3 As Single
Dim Load_4 As Single
Dim Load_5 As Single
Dim Load_6 As Single
Dim Load_7 As Single

Dim Strain_3 As Single
Dim Strain_4 As Single
Dim Strain_UL As Single
Dim Strain_LL As Single

Dim Initial_Stiffness As Variant
Dim Stiffness As Variant
Dim Norm_Stiffness_Reduction As Variant

Dim a As Single
Dim i As Single
Dim j As Single

Dim L() As Variant
Dim S() As Variant

Dim myArray_1() As Variant
Dim myArray_2() As Variant
Dim myArray_3() As Single
Dim myArray_4() As Single
Dim myArray_5() As Single

ReDim L(a)
ReDim S(a)

ReDim myArray_1(i)
ReDim myArray_2(i)
ReDim myArray_3(j)
ReDim myArray_4(j)
ReDim myArray_5(j)

Counter = 2
Counter_1 = 2
Counter_2 = 2
Counter_3 = 2
a = 0
i = 0
j = 0
Cycle = Cells(2, 6)

Do Until Strain_1 > 3000
Load_1 = Cells(Counter_3, 8)
Strain_1 = Cells(Counter_3, 9)
Strain_2 = Cells(Counter_3 + 1, 9)
If Strain_1 < 1000 Then
    ElseIf Strain_1 < 3000 Then
        L(a) = Load_1
        S(a) = Strain_1
        a = a + 1
    If Strain_2 < 3000 Then
        ReDim Preserve L(a)
        ReDim Preserve S(a)
    End If
    Else
        Exit Do
    End If
End If
Counter_3 = Counter_3 + 1
Loop

Slope_Stiffness = WorksheetFunction.Slope(L, S)
Initial_Stiffness = Slope_Stiffness * 10 ^ 6 / Specimen_Area
Cells(Counter_2, 14) = Initial_Stiffness
myArray_3(j) = Initial_Stiffness
myArray_4(j) = Cycle
Norm_Stiffness_Reduction = Initial_Stiffness / Initial_Stiffness
myArray_5(j) = Norm_Stiffness_Reduction

'Cells(Counter_2, 12) = Cycle
Cells(Counter_2, 15) = Norm_Stiffness_Reduction

j = j + 1

Counter_2 = Counter_2 + 1

ReDim Preserve myArray_3(j)
ReDim Preserve myArray_4(j)
ReDim Preserve myArray_5(j)

Do Until Cycle_a > Cycle
Cycle_a = Cells(Counter, 6)
Counter = Counter + 1
Loop

Do Until Cycle = 0

Do Until Cycle = 0

Do

Load_1 = Cells(Counter, 8)
Load_2 = Cells(Counter + 1, 8)
Load_3 = Cells(Counter + 2, 8)
Load_4 = Cells(Counter + 3, 8)
Load_5 = Cells(Counter + 4, 8)
Load_6 = Cells(Counter + 5, 8)
Load_7 = Cells(Counter + 6, 8)

If Load_2 < Load_1 And Load_3 < Load_1 And Load_4 < Load_1 And Load_5 < Load_1 And Load_6 < Load_1 And Load_7 < Load_1 Then
  Exit Do
End If

Counter = Counter + 1
Loop

Counter = Counter + 1

Do

Load_1 = Cells(Counter, 8)
Load_2 = Cells(Counter + 1, 8)
Load_3 = Cells(Counter + 2, 8)
Load_4 = Cells(Counter + 3, 8)
Load_5 = Cells(Counter + 4, 8)
Load_6 = Cells(Counter + 5, 8)
Load_7 = Cells(Counter + 6, 8)

Strain_1 = Cells(Counter, 9)
Strain_2 = Cells(Counter + 1, 9)
Strain_3 = Cells(Counter, 9)

If Load_2 > Load_1 And Load_3 > Load_1 And Load_4 > Load_1 And Load_5 > Load_1 And Load_6 > Load_1 And Load_7 > Load_1 Then

Cycle = Cells(Counter, 6)
Exit Do
End If

Counter = Counter + 1
Loop

Exit Do
Loop

Strain_4 = WorksheetFunction.MRound(Strain_3, 100)
Strain_UL = Strain_4 + 1300
Strain_LL = Strain_4 + 300

Counter = Counter + 1
Do Until Cycle = 0

Do Until Cycle_a > Cycle
Cycle_a = Cells(Counter, 6)
If Cycle_a = 0 Then
Exit Sub
End If

Strain_1 = Cells(Counter, 9)
Strain_2 = Cells(Counter + 1, 9)
Load_1 = Cells(Counter, 8)
Load_2 = Cells(Counter + 1, 8)
If Strain_1 < Strain_LL Then
    ElseIf Strain_1 < Strain_UL Then
        Exit Do
    End If

    Counter = Counter + 1

    Loop

    If Strain_1 > Strain_UL Then
        Exit Do
    End If

    myArray_1(i) = Load_1
    myArray_2(i) = Strain_1

    'Cells(Counter_1, 10) = myArray_1(i)
    'Cells(Counter_1, 11) = myArray_2(i)

    i = i + 1

    Counter = Counter + 1
    Counter_1 = Counter_1 + 1

    If Strain_2 < Strain_UL Then
        ReDim Preserve myArray_1(i)
        ReDim Preserve myArray_2(i)
    End If

    Loop

    Stiffness = WorksheetFunction.Slope(myArray_1, myArray_2)

    Cells(Counter_2, 14) = Stiffness * 10^6 / Specimen_Area

    myArray_3(j) = Stiffness * 10^6 / Specimen_Area
    myArray_4(j) = Cycle

    Norm_Stiffness_Reduction = myArray_3(j) / myArray_3(0)
    myArray_5(j) = Norm_Stiffness_Reduction

    'Cells(Counter_2, 12) = Cycle
    Cells(Counter_2, 15) = Norm_Stiffness_Reduction
Counter_1 = 2
Counter_2 = Counter_2 + 1
Cycle = Cells(Counter, 6)

If Cycle > 0 Then
    i = 0
    j = j + 1

End If

ReDim myArray_1(i)
ReDim myArray_2(i)
ReDim Preserve myArray_3(j)
ReDim Preserve myArray_4(j)
ReDim Preserve myArray_5(j)

Exit Do

Loop

Loop

Call Stiffness_Chart(myArray_4, myArray_5)

End Sub

================================================================================================
Secant Modulus Code

Sub Secant_Modulus_Tracking_File(Specimen_Area)
,
,
, 
Dim Counter_3 As Long 

Dim Load_3 As Single 
Dim Load_4 As Single 
Dim Load_5 As Single 
Dim Load_6 As Single 
Dim Load_7 As Single 

Dim Stress_1 As Single 

Dim Slope_Stiffness As Single 
Dim Initial_Stiffness As Single 
Dim Stiffness As Single 
Dim Norm_Stiffness_Reduction As Single 

Dim a As Single 
Dim i As Single 
Dim j As Single 

Dim L() As Variant 
Dim S() As Variant 

Dim Stress_Array As Variant 
Dim Strain_Array As Variant 

Dim Stiffness_Array As Variant 
Dim Cycle_Array As Variant 

ReDim L(a) 
ReDim S(a) 

ReDim Stress_Array(i) 
ReDim Strain_Array(i) 

ReDim Stiffness_Array(j) 
ReDim Cycle_Array(j) 

a = 0 
i = 0 
j = 0 

Counter = 2 
Counter_1 = 2 
Counter_3 = 2 

Cycle = Cells(2, 6)
Do Until Strain_1 > 3000

Load_1 = Cells(Counter_3, 8)
Strain_1 = Cells(Counter_3, 9)
Strain_2 = Cells(Counter_3 + 1, 9)

If Strain_1 < 1000 Then
    ElseIf Strain_1 < 3000 Then
        L(a) = Load_1
        S(a) = Strain_1
        a = a + 1
    If Strain_2 < 3000 Then
        ReDim Preserve L(a)
        ReDim Preserve S(a)
    End If
    Else
        Exit Do
End If

Counter_3 = Counter_3 + 1
Loop
Slope_Stiffness = WorksheetFunction.Slope(L, S)
Initial_Stiffness = Slope_Stiffness * 10 ^ 6 / Specimen_Area
Norm_Stiffness_Reduction = Initial_Stiffness / Initial_Stiffness

Cells(Counter_1, 16) = Initial_Stiffness
Cells(Counter_1, 17) = Norm_Stiffness_Reduction
Stiffness_Array(j) = Norm_Stiffness_Reduction
Cycle_Array(j) = Cycle
j = j + 1

ReDim Preserve Stiffness_Array(j)
ReDim Preserve Cycle_Array(j)

Counter_1 = Counter_1 + 1

Do Until Cycle = 0
Do Until Cycle = 0

If Cycle = 1 Or Cycle = 2 Then
  Counter = Counter + 1
  Cycle = Cells(Counter, 6)
  Exit Do
End If

Do
  Load_1 = Cells(Counter, 8)
  Load_2 = Cells(Counter + 1, 8)
  Load_3 = Cells(Counter + 2, 8)
  Load_4 = Cells(Counter + 3, 8)
  Load_5 = Cells(Counter + 4, 8)
  Load_6 = Cells(Counter + 5, 8)
  Load_7 = Cells(Counter + 6, 8)
  Stress_1 = Load_1 / Specimen_Area
  Strain_1 = Cells(Counter, 9) * (10 ^ -6)
  Strain_2 = Cells(Counter + 1, 9) * (10 ^ -6)
  If Load_2 < Load_1 And Load_3 < Load_1 And Load_4 < Load_1 And Load_5 < Load_1
  And Load_6 < Load_1 And Load_7 < Load_1 Then
    Exit Do
  End If
  Counter = Counter + 1
Loop

Stress_Array(i) = Stress_1
Strain_Array(i) = Strain_1
i = i + 1
ReDim Preserve Stress_Array(i)
ReDim Preserve Strain_Array(i)
Counter = Counter + 1

Do
  Load_1 = Cells(Counter, 8)
  Load_2 = Cells(Counter + 1, 8)
Load_3 = Cells(Counter + 2, 8)
Load_4 = Cells(Counter + 3, 8)
Load_5 = Cells(Counter + 4, 8)
Load_6 = Cells(Counter + 5, 8)
Load_7 = Cells(Counter + 6, 8)

Stress_1 = Load_1 / Specimen_Area

Strain_1 = Cells(Counter, 9) * (10^-6)
Strain_2 = Cells(Counter + 1, 9) * (10^-6)

If Load_2 > Load_1 And Load_3 > Load_1 And Load_4 > Load_1 And Load_5 > Load_1 And Load_6 > Load_1 And Load_7 > Load_1 Then
    Cycle = Cells(Counter, 6)
    Exit Do
End If

Counter = Counter + 1
Loop

Stress_Array(i) = Stress_1
Strain_Array(i) = Strain_1

Stiffness = (Stress_Array(0) - Stress_Array(1)) / (Strain_Array(0) - Strain_Array(1))

Norm_Stiffness_Reduction = Stiffness / Initial_Stiffness

Cells(Counter_1, 16) = Stiffness
Cells(Counter_1, 17) = Norm_Stiffness_Reduction

Stiffness_Array(j) = Norm_Stiffness_Reduction
Cycle_Array(j) = Cycle

i = 0
j = j + 1

Counter = Counter + 1
Counter_1 = Counter_1 + 1

ReDim Stress_Array(i)
ReDim Strain_Array(i)

ReDim Preserve Stiffness_Array(j)
ReDim Preserve Cycle_Array(j)

Do Until Cycle_a > Cycle
    Cycle_a = Cells(Counter, 6)
Counter = Counter + 1
Loop
Cycle = Cells(Counter, 6)
Loop
Call Secant_Stiffness_Chart(Stiffness_Array, Cycle_Array)
End Sub